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KRAFT PULPING OF WHEAT STRAW

BY

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Kraft Pulping of Wheat Straw

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This is to certify that the thesis prepared by Gebeyaw Engida entitled: *Wheat Straw Kraft Pulping* and submitted in partial fulfillment of the requirements for the degree of master of sciences (Chemical and Bio Engineering) complies with the regulations of the university and meets the accepted standards with respect to originality and quality.

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Declaration

I declare that the thesis for the M.Sc. degree at the University of Addis Ababa, hereby submitted by me, is my original work and has not previously been submitted for degree at this or any other university, that all resources of materials used for this thesis have been duly acknowledged.

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ACRONYMS

AAgSS Annual Agricultural Sample Survey

AD Anno Domini, which is Latin for "year of our Lord," and it means the number of years since the birth of Jesus Christ.

AFPA American Forest & Paper Association

ANOVA Analysis of variance

ASTM American Society for Testing and Materials (ASTM)

ATA Agricultural Transformation Agency

BSEN British Standard European Norm

CD Cross Direction

CIMMYT Spanish acronym, Centro Internacional de Mejoramiento de Maíz y Trigo for International Maize and Wheat Improvement Center

CSA Central Statistical Agency

CSF Canadian standard of freeness

CTMP Chemical Thermo- Mechanical Pulping

C.V Coefficient of Variation

DIN Deutsches Institute fur Normung (German Institute for Standardization)

EA Effective Alkali

EPPSC Ethiopian pulp and papers Share Company

E.U European Union

FAO Food and Agricultural Organization

GE General Electric

GSM Grams per Square Meter

GTP Growth and Transformation Plan

H - Factor Factors that depends on both time and temperature AND the temperature dependence is very strong.

ILO International Labor Organization

ISO International Organization for Standardization

LSR Liquor to Straw Ratio

MD Machine Direction

MOA Ministry of Agriculture

MT million Tonnes

mN mili newton

NSSC	Neutral Sulphite Semi-Chemical
OBA	Optical Brightness Agent
PPPDS	Public Procurement & Property Disposal Service
PRESS	Predicted Residual Sum of Squares
SCAN	Shorthand representation for Scandinavian group of standards
SNNP	Southern Nations, Nationalities and Peoples
TAPPI	Technical Association of Pulp and Paper Industries
TMP	Thermo-Mechanical Pulping
UK	United Kingdom
USAID	United States Agency for International Development
USD	United States Dollar
WRI	World Resources Institute

ABSTRACT

In this paper, pulping of wheat straw with Kraft process was studied. Chemical and morphological properties of wheat straw fibers were also investigated. Chemical properties including cellulose (39.8%), hemicellulose (29.47%), lignin (20.98%), ash (6.27%) and extractives (4.1%) were determined. Fiber length, diameter, lumen width and cell wall thickness were 1210, 17.20, 8.40 and 3.80μm, respectively. Wheat straw has acceptable Runkel ratio (0.9) and flexibility coefficient (48.8) in the range of non-woods.

Various pulping conditions including composition of cooking liquor (10, 15 and 20%) and cooking temperature (130, 140 and 150^{o}C) at reaction times (30, 60 and 120 min) were studied systematically to determine the suitable pulping conditions. The influence of pulping conditions on the pulp obtained (Yield and Kappa number) and paper strength properties (tensile, tear and bursting strength) are analyzed by three-factor and orthogonal experiments, and optimum pulping conditions are obtained. The best properties of the pulp and hand sheets were obtained with 10% sodium hydroxide, in the cooking solvent, 136.9^{o}C cooking temperature and 30 min as cooking time. As a result of using the processing variables over the variation ranges considered, the following optimum values of the dependent variables were obtained: 33.91% (yield), 16.76 (kappa number), 4027m (breaking length), 8.96*10^{-3} Nm^{2}g-1 (tear index) and 2.66 KNg-1 (burst index) for pulps and hand sheets. In general, results based on chemical and morphological analysis indicated that wheat straw fibers are promising fibrous raw material for the paper production.

1. INTRODUCTION

1.1. Background of the study

Traditionally, wood is considered the primary raw material and the major source of pulp in paper production. The recent global production of paper and paper board is 406 million tons per year which is derived from 225 million tones recycled paper, 176 million tonnes wood pulp and 12 million tonnes other fibres pulp (FAO UN, 2015). The paper and paperboard product group comprises graphic papers (newsprint, printing and writing paper) and other paper and paperboard. The latter is further subdivided into wrapping and packaging paper, household and sanitary paper, and other paper and paperboard not elsewhere specified. Under the category of 12 million tonnes other fibres pulp agricultural residues comprises the major parts and are increasing production every year. Agricultural fibres constitute an alternative to wood as raw material for making pulp on account of their high growth rate and adaptability to various soil types (FAO , 2016).

There is a growing interest in using agricultural residues such as rice straw for pulping and paper making. As future worldwide fibre shortages are predicted, agricultural fibres are believed to be a potential substitution for wood fibres in certain paper applications. Rice straw as an agricultural residue has been used in pulp and paper production for a long time and remains one of the major raw materials in many countries (Usta et al., 1999). The European Union has supported research on several non-wood species with high biomass production, that can be planted in areas made available from agriculture and that are adequate for different industrial uses (Van Dam et al., 1994). The most frequent raw materials are straw, in particular wheat and rice straw and sugar cane bagasse (MacLeod, 1988). The E.U. consumption of pulps from annual plants is around 400,000 tonnes of which about one-quarter are imported (Van Dam et al., 1994). The pulping of annual plants differs substantially from wood pulping. In most cases, delignification proceeds very fast and the resulting pulps have low residual lignin content (Akpakpan, Akpabio, Ogunsile, & Eduok, 2011).

The use of agricultural residues as a source of cellulose in the paper industry is supported by many institutions, due in part to increasing environmental pressures (Camaro et al., 2004). The principal aim of these incentives is to minimize the environmental damage of the

cellulose industry, in addition to reducing raw material pressure placed on forests (Pan et al., 1999). Another reason that renders the use of agricultural residues in the cellulose industry attractive is the limited forest resources of certain countries (Jimenez, 1999). When considering countries with an agriculture dependent economy and limited forest sources, the use of agricultural residues and cultured or uncultured annual stalks for pulp manufacturing offers a significant solution to the raw materials problem (utilizing the current potential of agricultural residues in the cellulose industry).

In the summer of 2012, Kimberley Clark, the global leader in tissue paper products, announced plans to replace half of its timber consumption with alternative source of fiber by 2025 (International Finance, 2013). The quality of straw pulp produced depends on the processing technology used. The main advantages of straw (which apply whichever technology is used) are smoothness and good tensile strength vis-a-vis other hardwoods, as well as opacity and good beatability, the ease with which pulp can be beaten to achieve the desired properties (International Finance, 2013).

Numerous researches conducted on a global scale are focused on identifying alternative non-wood raw materials as a source of cellulose fibers. Some types of non-wood fibres have been are already used in some paper grade productions, although the paper quality varies based on the source of the fibres.

Wheat straw is considered to be one of the most important agricultural residues based on the reported results. Namely, agro-based fibres are mostly used in the packaging and corrugated cardboard (liner and fluting papers) production (Akbari et al., 2012).

In Ethiopia wheat straw is currently one of the most abundant and cheap agricultural wastes. The crop is grown mostly during the main (meher) rainy season from June to September and harvested from October through January. As the data shown in Ethiopian central statistics agency wheat production for the year 2015/16 was 4.2 million tons, which covers a harvested area of 4.78 million hectares. Wheat yield with the specified year was 0.87 tons/hectare, due to favorable rainfall throughout the growing season in the wheat highland regions and its yield has been increasing since 2002 due to government initiatives that encouraged farmers to adopt improved seed varieties, increase fertilizer distribution, and expanded agriculture extension in rural area (CSA E. , 2016).

The wheat straw yield or the ratio of wheat straw to grain production has been estimated by a number of investigators. Such estimates approximate 1.3 tons of wheat straw per tons of

grain, 1.0 tons of barley straw per tons of grain, and 1.2 tons of Oats straw per tons of grain (Bowyer , Howe, Pepke, Bratkovich, Frank, & Fernhonz, 2014). So in the year 2015/16, wheat straw production in Ethiopia was estimated to 5.46 million tonnes which would result 3.64 million tonnes of pulp as the study by Michael Pilarski showed.

In Ethiopia there are about 22 companies that involved in paper making and trading businesses, of which only 'Ethiopia pulp and paper share company' and 'Barguba' plc. uses imported pulp for their paper mills while others import and produce paper rolls for further processing (Amsalu , 2015). Ethiopia has been imported 7.8 million tonnes of pulp at a cost of 6.3 million USD and 151 million tonnes of paper at a cost of 167 million USD in 2016 (ECCIDI, 2016). Only about 5% of the country's paper demand is produced in the country and the rest will be covered by importing finished paper from abroad.

In the future the excess availability of agricultural residues particularly wheat straw, the increasing demand in paper products, the limited resource of wood fiber, the inauguration of new pulp mill projects and the intentions of government towards wood product industries in Ethiopia would lead pulp from cereal grain straw may partially substitute for wood fiber in a range of paper and paperboard products. In addition to this using pulp from agricultural residues in combination with wood pulps can provide useful paper properties; however, better understanding of straw properties will be the basis for future developments using significant amounts of this raw material in Ethiopian paper mills.

For the production of pulp from straw and other non woody raw materials, several chemical pulping processes have been developed, but Soda, Kraft and neutral sulphite semi-chemical (NSSC) pulping are suitable. Lime process is also still utilized for pulping. Among all pulping methods, soda and modified soda processes are superior to others. However, one of the most serious problems associated with straw chemical pulping is high silica content of fibres, which makes conventional recovery difficult (Akpakpan, Akpabio, Ogunsile, & Eduok, 2011).

1.2. Statements of the problem

Paper is becoming an important commodity of today's society. The pulp and paper industries have been rising due to the increased demand of paper-based products and it is a must to find raw material sources for these industries. The consumption of paper has been also steadily increased over the world.

Although wood is renewable, the rate at which wood has been used is not commensurate with the rate it is being replaced. The rate at which forests are declining has been estimated to be 13.0 million hectares per year in developing countries (FAO, 2005). In many developing countries of the world today, wood is becoming scarce and expensive because of the heavy dependence on wood for construction and building purposes, grazing, and conversion of forests to agricultural land to grow crops and felling of tree for firewood.

As some study estimates about 5 tons of wood (10-17 trees) will be needed to produce 1 ton of pulp for paper and only 1.5 tons of straw will needed to produce the same amount. This shows that too much wood from virgin forests will be consumed by the paper industry. In addition to this heavy-duty industrial processes are required to turn wood into paper compared to straw sources and also wood pulp mill costs 5 times as much as a straw pulp plant, and it uses 10 times the energy (Michael , 1994).

As the data obtained from Ethiopian construction material inputs and chemical development institute presents, the amounts of pulp and papers imported from the year 2009-2015, was 895,000 tonnes at a cost of 989 million USD. This practice puts an additional foreign exchange burden to the government of Ethiopia which can be alleviated partially by using local raw material sources such as agricultural residues for the paper mill industries.

Ethiopia as a developing country has significantly very low paper and paper board consumption per capita of 0.43 kilogram /person /year when compared with the world average of 54.48 kilogram/person/year in 2005 (WRI, 2005) will be expected to grow significantly (Amsalu , 2015).

The ongoing economic development and livelihood improvement in Ethiopia inevitably would create huge demand and consumption of pulp and paper products due to increasing economic welfare household consumption, expansions of schools and universities, and increasing of manufacturing sectors that needs packaging and wrapping.

1.3. Significance of the study

Paper production that is based on agricultural crop waste offers numerous important environmental and economic advantages over tree based paper production. Advantages of making paper from agricultural waste particularly wheat straw instead of trees includes: protect forests, released fewer toxic chemicals, it is renewable and sustainable, requires less energy, and promotes local paper manufacturing. In addition to this a shift from wood pulp to

straw pulp in making paper would take pressure off wild forests and the species that live in them.

Agricultural crops are easier to turn into pulp than wood is, and consequently, they require smaller quantities of toxic solvents to turn them into pulp for paper-making. Unlike virgin forests, which are endangered by human, harvesting agricultural waste will be readily available as long as humanity engages in agriculture.

Directing agricultural waste into paper manufacturing provides an additional income stream for farmers, without impacting food production or increasing energy inputs, and without putting new land into production. Consequently, increasing reliance on agricultural inputs allows for greater decentralization in the paper manufacturing industry, which allows in turn for reductions in transportation costs and energy consumption. Local economic independence is also improved with greater local control over manufacturing processes that impact local economies.

In Ethiopia's Growth and Transformation Plan-2 (GTP II), the GoE has prioritized wheat as a major implementation strategy, with targets to increase bread and durum wheat production volumes from a collective 42.3 million quintals in 2015 to 62.08 million quintals by the year 2020 (USAID, 2011), with the corresponding 5.5 and 8.0 million tonnes wheat straw respectively . The GoE has explicitly prioritized private sector investment in the wheat sector, with investors in prioritized regions eligible for an additional one year income tax exemption and import of duty-free capital goods and materials to establish or expand existing businesses. A variety of improved seeds are also capable of growing in Ethiopian soil and the ATA is currently working with farmers to increase yield and train on efficient handling processes (USAID, 2011).

So, the rationale of this research work was to investigate the possibility of substitution of wheat straw for imported wood pulp in paper production which could be a good source of raw material to help meeting the Growth and Transformation Plan of the country.

1.4. Objectives of the Research

1.4.1. General objective

The general objective of this study was to investigate the potential of wheat straw for pulp and paper production through Kraft pulping process.

1.4.2. Specific objectives

This study has also the following specific objectives:

- ✓ To characterize the chemical and morphology of wheat straw.
- ✓ To synthesize and investigate the effects of pulping parameters on the yields of wheat straw pulp.
- ✓ To determine the optimum pulping parameters (time, active alkali and temperature) that gives best quality pulp and yield.
- ✓ To characterize pulp chemically and paper mechanically, obtained from wheat straw

1.5. Scope of the study

The scope of this study was directing agricultural wastes specifically wheat straw to pulp and then to paper products. Though there were numerous techniques of pulping, chemical pulping which uses delignification chemicals and produces quality pulp methods are used. Among the many chemical pulping methods Kraft or sulphate pulping process was selected for the study which uses sodium hydroxide and sodium sulfide chemicals. The study covers process steps, wheat straw delignification, paper sheet forming and sheet characterization.

2. LITERATURE REVIEW

2.1. Global pulp and paper production

The manufacture of pulp, paper and paper products ranks among the world's largest industries. Mills are found in more than 100 countries in every region of the world, and directly employ more than 3.5 million people. The major pulp and paper producing nations include the United States, Canada, Japan, China, Finland, Sweden, Germany, Brazil and France (each produced more than 10 million tonnes in 1994; Table 2.1) (ILO 1992).

Globally the production of pulp is led by North America, which accounts for over one-third of the pulp production and generates an excess supply of 5%. Europe and Asia follow, each with close to one fourth of global pulp production. The overall production of pulp in 2014 is 178.5 million tonnes, with total consumption of 179.6 million tonnes. Whereas the global production of paper and paper board in 2014 was 406 million tonnes, with 407.6 million tonnes consumed. Asia is the leader in both production and consumption with about 45% in both categories (Susanna , Lars , William, Hannah, Karsten, & Vera , 2016).

In 2015, the regional distribution of pulp and paper production was as follows: Asia-Pacific, 195 million tonnes (48%); Europe, 104 million tonnes (26%); Northern America 83 million tonnes (21%); Latin America and the Caribbean, 21 million tonnes (5%); and Africa, 4 million tonnes (1%) (FAO UN, 2015).

The two largest paper and paperboard producers in 2015 were China (111 million tonnes) and the USA (72 million tonnes). Their combined production accounted for 45 percent of global production. The other three largest producers were Japan (26 million tonnes), Germany (23 million tonnes) and India (14 million tonnes), which accounted for another 16 percent of global production. In terms of future capacity, future trends in the paper and board industry show a stable production over the next five years (FAO UN, 2015).

The first paper was invented for the first time by ancient Egyptian where they used papyrus paper. Papyrus paper was made from papyrus tree which was common in the bank of Nile River but this paper is fragile, rough and had limited supply. After that, in 200 BC ancient Greek used a parchment which was made from stretched, dried calf, sheep, or goat skin; but this paper was expensive, sensitive to humidity, and also limited supply. Then, a Chinese court official, Cai Lun, inspired by bee and wasp nests to make paper from wood pulp (especially mulberry tree) in 105 AD and this paper was used for writing, toilet paper, bank

notes, etc. The first paper mill was founded in Baghdad, Iraq at 739 AD but this paper-making by hand is a slow and batch process. The modern paper making uses the fourdrinier machine (1801) allows high speed and continuous paper making.

Then, as we travelled through time we will have a look the market of the paper nowadays. The paper consumption worldwide amounted to roughly 371 million tonnes in 2009 (Cielo, 2011), where 40% of this paper consumption belonged to Asian region (Bajpai, 2012). In Asia region, Japan is the lead consumer of paper products per capita followed by Malaysia and Singapore (Aripin, 2014).

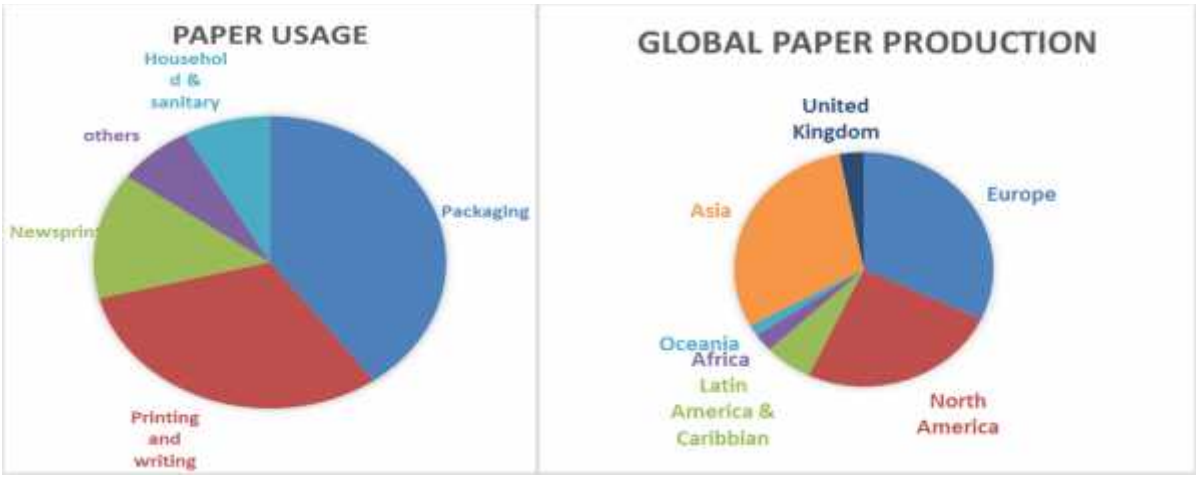


Figure 1 Global Paper Usage and Production (http://Paper Consumption)

2.2. Fiber sources for pulp and paper production

Normally, fiber resources for pulp and paper are obtained from trees or agricultural crops. These resources include plant materials harvested directly from the land (wood, straw and bamboo), plant material by products or residual from other manufacturing processes (wood chips from sawmills, bagasse and cotton linear) and fiber recovered from recycled paper or paperboard. Forest resources have important value in producing a range of different wood resources for pulp and paper-based industries (Aripin, 2014).

Wood resources are divided into two types which are softwood (such as spruce, pine, fir, larch and hemlock) and hardwood (such as eucalyptus and birch). Huge majority of wood resources (more than 90-92% of fibers) are used for pulp and paper production globally (Jiménez et al., 2009 and Sridach, 2010a). These wood resources are used in many kinds of paper grades due to its smooth surface area and strong strength (Dick et al., 2006). Wood

consists mainly of cellulose, hemicellulose, lignin, extractives and ash and the chemical composition of wood resources varies from species to species (Henricson, 2004).

Trees needed to meet virgin wood fiber demand of the forest product industry are already growing except for the new fast growing plantations. Therefore, in global term, there will not be a long-term fibre shortage. However, fibre supplies within and across particular regions will tighten. These regional imbalances are already significant and will continue to grow (Chandra, 1998). Nowadays, many countries are looking for non-wood plants fibre resources as alternative fibres in pulp and paper based industries. This is due to the depletion and rising prices of wood resources and readily available non-wood fibre resources in these countries (Atchison, 1992).

2.2.1. Non-wood fibre resources

Nowadays, in paper making industry, the environmental problems have brought forward the need for cleaner technology where the new non-wood resources have been introduced to replace traditional raw materials such as wood resources with non-wood resources. The cleaner technology or green technology is applied to achieve increased production with minimum effect especially on the environment and lessen the disposal cost, steadiness risks and resource cost resulting in a declined burden on the natural environment and also increase the profits in pulp and paper-based industries (Sridach, 2010b). The abundance of non-wood fibres in some countries, made them responsible for its use in pulp and paper based. This is considered as the best way and more profitable for non-wood fibre to be used as alternative fibres in paper based production.

There is a growing interest in the use of non-wood resources in pulp and paper-based industries. There are many studies about the potentiality of non-wood plant species which are tobacco (Shakhes et al., 2011a), wheat straw (Jiménez et al., 2002a), giant reed (Shatalov and Pereira, 2006), canola straw (Hosseinpour et al., 2010), Tunisian alfalfa (Marrakchi et al., 2011) and vine (Mansouri et al., 2012) stems as a good fibre resources to replace the wood fibre resources in pulp and paper-based industries.

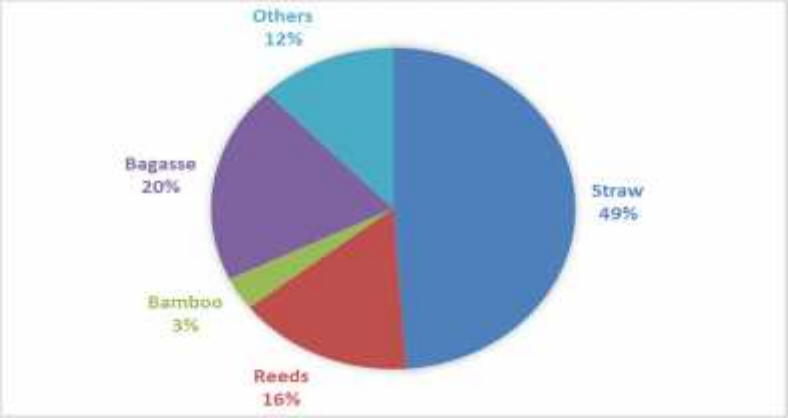


Figure 2 Consumptions of non-wood pulp in paper production by type (Sridach, 2010b)

The total of non-wood plants (8-10%) pulping capacity worldwide is increasing faster than the wood pulping capacity (González et al., 2008 and Rodríquez et al., 2008). In China, the consumption of non-wood resources in pulp and paper-based industries is higher than wood sources from the year 1995 to 2005 (Sbrilli, 2007). The development of these industries will need a continuous and sustainable forestry around the world. This is also due to the fact that non-wood plants sources have displayed different kinds of advantages in pulp and paper-based production compared to the wood resources.

High market demands as well as the environmental issues because of the large usage of wood supply in pulp and paper production have increased the interest to seek for non-wood plants as substitution fibre which is also environmentally friendly (González-García et al., 2010 and Jiménez et al., 2002a). Non-wood plants are the raw materials for production of high-quality specialty papers (Gomihno et al., 2001 and Wan Rosli et al., 2004). Non-woody plants have given many benefits such as it can give additional income to the farmers for food crop-waste such as straw, bagasse and grasses (Salmela et al., 2008). Apart from the above reasons, some non-wood plant fibres are in demand for pulp and paper-making due to the special properties that make them better than wood fibre. For example, abaca is an excellent raw material for manufacturing of specialty paper, for its long fibre length and high strength properties such as tear, burst and tensile indices (Peralta, 1996). In addition, sisal can be made into strong products whereas cotton linters are used for premium quality letterhead paper, currency paper, dissolving pulp and other specialty products (Chandra, 1998). Moreover, bagasse and straw are best at contributing excellent formation to papers and can replace hardwood chemical pulps for printing and writing paper (Sridach, 2010b). Generally, non-wood plant fibres that are used in pulp and paper industries can be broadly divided into three categories

based on their availability. These are agricultural residues, natural growing plants (annual plants) and non-wood crops grown primarily for their fibre (Sriadch, 2010b). First, agricultural residues are considered by low raw materials price, moderate quality and the abundance of raw materials after harvesting season such as rice (Navaee-Ardeh et al., 2004), wheat straw (Jiménez et al., 2002b), corn stalk (Flandez et al., 2010) and sugarcane bagasse (Hemmasi et al., 2011).

The naturally growing plants which are the type consist of bamboo (*dendrocalamus strictus*), reeds (*Phragmites communis Trinius*), sabai grass, (*Euaiopsis binata*), papyrus (*Cyperus* papyrus) and elephant or Napier grass (Madakadze et al., 2010). Fibre from baste fibre, Jute (*Corchorus capsularis*), ramie (*Boehmeria nivea*), leaf fibres, abaca (Musa textiles), seed hair fibre, cotton fibre, rags and linters and kenaf (*Hibiscus cannabinus*) are the third category of non-wood fibre that are the most important resources in pulp and paper making (Chandra, 1998). Agricultural residues have higher cellulose and lower lignin content than annual plants and non-wood crops. These contents generally provide the higher mechanical properties of hand sheet. The chemical and physical properties of non-wood fibres also affect their mechanical properties. For example, Elephant grass, in annual plants contains lower lignin and short fibre length that contribute to high strength property (Aripin, 2014).

Table 1 Summary of properties of non-wood plants

Categories	Raw materials	Chemical properties			Physical properties		Mechanical properties		
		Cellulose w/w %	Hemicellulose, w/w %	Lignin, w/w %	Fibre length, mm	Fibre diameter, µm	Tensile index, mN/g	Tear index, mN.m2/g	Burst index, kPa.m2/g
Agricultural residue	Banana stem (Musa paradisica)[a]	59.18	17.50	18.21	1.55	22.00	47.76	9.10	4.51
	Rice straw[b,c]	41.20	19.50	21.90	1.41	8.00	26.11	0.31	1.20
	Sugarcane bagasse [d]	42.34	28.60	21.70	1.51	21.40	58.00	5.80	4.20
	Wheat straw [b,e]	38.20	36.30	15.30	0.74	23.02	76.70	4.11	3.74
Annual plant	Bamboo [f]	43.00	39.00	31.00	2.70	14.00	n.a	18.10	4.90
	Elephant grass [g]	45.60	45.60	45.60	45.60	45.60	45.60	45.60	45.60
	Switch grass [g]	41.20	41.20	41.20	41.20	41.20	41.20	41.20	41.20
Non-wood crops	Kenafbast [h]	55.50	17.70	12.50	2.90	28.16	2.09	11.84	55.50
	Palmyra plant fruit[i]	37.01	31.51	18.54	1.07	n.a	13.80	1.12	37.01

(Aripin, 2014): n.a: non-available, a: Goswami et al. (2008), b: Enayati et al. (2009), c: Sridach, (2010b), d: Agnihotri et al. (2010), e: Berrocal et al. (2004), f: Sarwar et al. (2009), g: Madakadze et al. (2010), h: Udohitinah & Oluwadare, (2011), i: Sridach, (2010a), j: Khiari et al. (2010).

Therefore, with the best intention of solving disposable issues of agricultural waste in Ethiopia and finding the best alternative for waste non-wood pulp, this study is focused on wheat straw (non-wood crops grown primary) due to their abundance and less utilization of these waste materials. In addition, the concepts of "from waste to wealth" and "recyclable material" are now important in Ethiopia in order to build a sustainable and sound material-cycle society through the effective use of these waste resources.

There are four different categories of sources for the non-wood fibers (sometimes compacted to three), these includes: Agricultural residues (sometimes called Field or harvesting residues), Industrial residues (sometimes included with agricultural residues and called 'process residues') and naturally occurring uncultivated crops.

Agricultural residues

These are sometimes called Field or harvesting residues, materials left in an agricultural field or orchard after the crop has been harvested. These residues include straw, stalks (corn, sorghum, cotton). It is renewable in real time, while the fastest renewable time for commercial pulpwood is seven years. The cost of such plant fibre has already been "pre-paid" by the production of grain and oilseeds. Existing farm machinery can be used (Wong). Crop residues can be used as animal fodder, bedding, and soil amendment and as energy source. Agricultural by-products are characterized by a low raw material price and moderate quality (Finell).

Industrial residues

These are sometimes called 'process residues', those materials left after the processing of the crop into a usable resource. The wastes after agricultural products are processed are called Agricultural Processing Residues. They include bagasse, hemp residue, rice husk and peanut crust, cotton linters snipped from cottonseed after ginning for textiles but before pressing for oils; cotton or linen scraps from clothing production, flax residue from oilseed. It is easy to collect and transport this type of raw material because it is already mainly stocked in the factories. High quality pulp can be produced from industrial crops, but the raw material is

more expensive; however, the raw material costs of natural plants are competitive with wood (Finell).

Naturally occurring crops: These are the natural stands such as wild grasses, sisal, reeds, papyrus and bamboo (Kinsella).

Table 2 Availability of non-wood plant fiber worldwide

Raw material	**Potential world wide availability(BD* metric tons)**
Sugar cane bagasse	75,000,000
Wheat straw	570,000,000
Rice straw	320,000,000
Oat straw	60,000,000
Barley straw	150,000,000
Rye straw	40,000,000
Seed flax straw	2,000,000
Grass seed straw	3,000,000
Subtotal Straw	1,145,000,000

(Chandra, 1998)

Agricultural residues account for 73% of the world's non-wood pulp capacity, natural plants such as reed and bamboo account for 18% and the remainder consists mainly of industrial crops (Finell).

Cultivation and harvesting of non-wood crops is favorable compared to wood fiber in several ways:

- The payback time of non-wood plantations is much shorter than that of forest plantations, because harvesting of non-woods can start already after few months from sowing.
- Perennial crops: multiple harvests from one plantation.
- Non-wood plantations consume less water and fertilizers.
- Equipment for sowing, harvesting and baling is already available at farms.
- Ready infrastructure and no special vehicles required for transportation.
- Producing paper from non-wood fibres would help in reducing the need to procure pulpwood from natural forests, and for large-scale plantations (Pande).
- The use of non-wood materials can reduce deforestation in some countries and

- Reduce emissions of carbon monoxide and carbon dioxide that arise from the burning of waste agricultural residues.
- It can have a positive effect on employment and social structure in sparsely populated areas (Finell). Some non-wood fibres used as raw materials for papermaking have high annual yields per hectare.

2.2.2. Wheat straw

Wheat is an annual agricultural crop grown for the grain portion of the plant that is a valuable food product. The rest of the wheat plant is in the wheat straw consisting of stems and leaves, chaff that is a protective cover over the grain, and the underground root system (James & John , 2001). Wheat straw is one of the most abundant and cheap agricultural wastes, and the annual worldwide production of wheat straw was estimated to be approximately 540 million tons (Reddy & Yang, 2007).

After the grain is harvested the fields are prepared for the next crop. The straw is burned, removed, left on the field or plowed back into the soil. The choices made by the landowner depend on a variety of factors including the quantity of material, the next crop to be planted, the weather conditions, the soil erodibility and nutrient needs, the slope of the land, and any markets that may be available for the straw (James & John , 2001).

Small portion of has been used as animal feed-stock and bedding. Farmers also use wheat straw in construction of mud houses but still most of these agricultural byproducts are left on the ground to decompose. In some parts of the world, wheat straw is burnt in open fields, causing air pollution. Many possible composite products can also be made using wheat fibers including structural (using thermosetting resins) and nonstructural (using either a thermosetting or thermoplastic resin) materials, geotextiles and molded products (using fiber mats prepared by physical entanglement, nonwoven needling, or thermoplastic fiber-melt matrix technologies), sorbents and filters (for removing pollutants from contaminated air, water, and/or soil), packaging, and provide fiber for the paper industry. One of such applications is use of wheat straw in composites (Muhammad , Abdul , Aqeel , & Sadia, 2010).

Despite the large amount of straw grown annually, the first challenge to overcome in the chain of production is related to raw material availability, in that the low bulk density of straw makes its transportation expensive. In addition, large storage areas are required for this bulky seasonally produced material (Clark & Deswarthe 2009, Yuan & Sun 2010). It has

been shown, however, that straw can be stored without quality degradation (Leponiemi 2011) and the development of compacting devices, e.g. pelletizing, briquetting and baling equipment will ease the transportation and storage problems (Karjalainen, 2015).

The ratio of straw to grain production has been estimated by a number of investigators. Such estimates approximate 1.3 tons of wheat straw per tons of grain, 1.0 tons of barley straw per tons of grain, and 1.2 tons of Oats straw per tons of grain (Bowyer , Howe, Pepke, Bratkovich, Frank, & Fernhonz, 2014). When geographic differences are considered, and assuming that less than 100 percent recovery can be attained, estimates of straw yield are often adjusted to more conservative values than those cited above. For example, the Alberta Department of Agriculture (2000) estimates residue volumes more conservatively, reflecting geographic differences and an assumption of only 80 percent recovery combination of other resources (Bowyer , Howe, Pepke, Bratkovich, Frank, & Fernhonz, 2014).

2.2.2.1. Morphological Characteristics of Wheat Straw

Of all the Non wood fibers used for Papermaking, straw or grasses such as wheat straw, rice straw, reed and sugar cane bagasse account for most of the total non-wood pulp capacity. Wheat straw has better quality for papermaking compared to the other non-wood fibers such as sunflower stalks, vine shoots and cotton stalks due to its stronger breaking length of paper hand sheets. The physical content of wheat plant and its component percentage is described below in Table 3.

The morphological properties of wheat straw and its comparison with common papermaking fiber resources are summarized in Table 4. The result shows that the wheat straw contained short fibers with a mean length of 1.14mm (0.4-3.2 mm) and width of 0.0105 mm (0.008-0.034 mm). Typically wheat straw fibers are fairly narrow, thick-walled, and have a blunt or pointed ends and are as long as the short fibers of non-wood plant such as Bamboo, reed and corn stalk. However, they are longer than tobacco straw, cotton stalks and aspen and are similar to rye straw fibers. The fiber diameter and lumen width of wheat straw fibers are similar to those of cotton stalks and aspen fiber. On the other hand, cell wall thickness of wheat straw fibers is thicker than bamboo, reed, cotton stalk and aspen fibers (Jafar. & Ahmad., 2011). In blends of various proportions with other fiber sources, it is used to make printing and writing paper, glassine and greaseproof paper, duplex and triplex paper, corrugating medium, strawboard and “B” grade wrapping paper (Ilvessalo & Pfaffli, 1995).

Table 3 Table Physical Content of Wheat

Physical component	Mass Percent*
Internodes	68.5
Leaves-Sheaths	20.3
Leaves-Blades	5.5
Nodes and Fines	4.2
Grain and Debris	1.5

(Mckean. & Jacobs., June 1997)

Table 4 The mean values of wheat straw fiber dimensions and derived indexes, comparison with common papermaking fibers.

Fiber Properties	Wheat straw[a]	Bamboo[b]	Rye straw[c]	Corn stalk[d]	Tobacco straw[e]	Reed[f]	Cotton stalk[g]	Aspen[h]
Length, L (mm)	1.14	2.30	1.15	1.32	1.07	1.39	0.83	0.96
Diameter, D (μm)	19.32	15.1	14.7	24.3	26.8	13.5	19.60	20.80
Lumen Width, d (μm)	10.54	6.9	4.2	10.7	16.3	7.0	12.80	16.94
Wall thickness, W(μm)	4.39	4.17	4.6	6.8	5.3	3.2	3.40	1.93
Runkel ratio,(2w/d)	0.83	1.21	2.19	1.27	0.65	0.91	0.53	0.23
Slenderness ratio,(L/D)	59	152.3	78.23	54.32	39.93	102.96	42.35	46.15
Flexibility coefficient, (d/D * 100)	54.55	45.69	28.57	44.03	60.82	51.85	65.31	81.44

(a): (Jafar. & Ahmad., 2011) , (b): Deniz and Ates 2002, (c): Usta and Eroglu 1987, (d): Usta et al. 1990, (e): Eroglu et al. 1992, (f): Kirci et al. 1998, (g): Ververis et al. 2004, (h): Law and Jiang 2001.

Consequently, the calculated Runkel ratio for wheat straw fibers (83.3) is higher than that of tobacco straw, cotton stalk and aspen fibers. The slenderness ratio of wheat straw fibers is 59 and is comparable with that of corn stalk and aspen fibers and higher than that of tobacco straw and cotton stalk fibers. Generally, the acceptable values for slenderness ratio and Runkel ratio of Papermaking fibers are more 33 and less than 1, respectively. Referring to this and morphological properties of wheat straw fibers, it can be deduced that the wheat straw fibers can be collapsed to form ribbon like structures in the paper and that the overall morphological properties of wheat straw fibers are satisfactory for papermaking, although they would be classified as short fibers (Jafar. & Ahmad., 2011).

2.2.2.2. Chemical Constituents of wheat straw

The cellulose content of wheat straw was found to be 49.78 %, which is satisfactory for pulp production (close to or above 40 %) (Jafar. & Ahmad., 2011). According to Nieschlag et al. (1960) plant materials with cellulose of 34 % and above are characterized to be suitable for pulp and paper manufacture. The lignin content of wheat straw was found to be lower than that of rice straw (21.90 %), Egyptian cotton stalks (22.50 %) and bamboo (24.5 %). The organic solvent extractive of wheat straw was found to be higher than those of rice straw (0.56 %) and aspen (2.50 %). The organic solvent extractive was lower than that of wheat straw (7.80 %) and the ash content of wheat straw was also high.

Table 5 Chemical Composition of Wheat straw (% on OD basis)

Component	Value %
Cellulose	49.78
Hemicellulose	20.37
Lignin	19.64
Extractives soluble in alcohol - acetone	4.93
Ash content	5.28

Cuticle wax only accounts for a small fraction of the weight of the straw. Generally the major constituents of straw consist of cellulose, hemicelluloses and lignin. Few pectic compounds and few mannans are also present. Ethanol extracts from wheat straw contains the low molecular weight sugars, fructose, glucose, sucrose, arabinitol and mannitol (Jafar. & Ahmad., 2011).

Cellulose

Cellulose is a high molecular weight (106 or more) linear polymer of β-(1-4)-D-glucopyranose units in the 4C1 conformation (Figure 3). The fully equatorial conformation of α-linked glucopyranose residues stabilizes the chair structure with minimized flexibility. After the removal of water from glucose, the formed glucose anhydride is polymerized into long cellulose chains that contain 5000-10000 glucose units. Two glucose anhydride units

constitute the cellobiose unit, which is the basic repeating unit of the cellulose polymer.

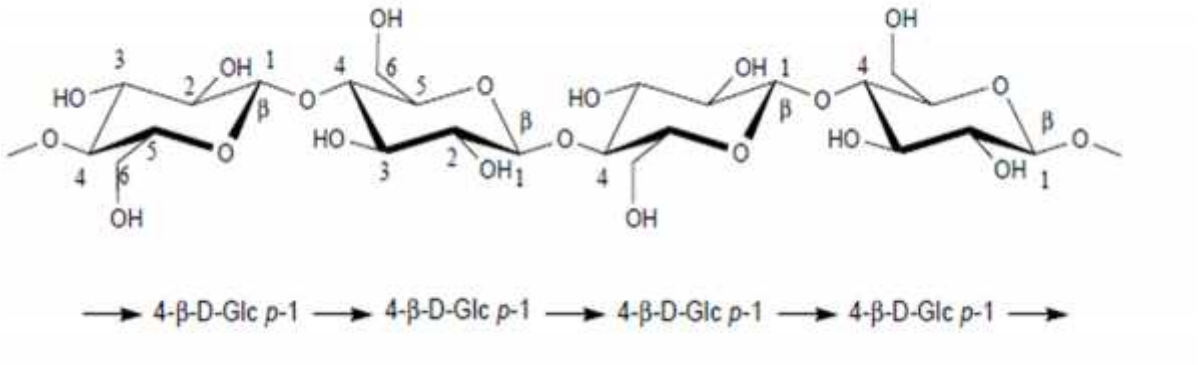


Figure 3 Structure of cellulose *(Christiane , 2005).*

The neighboring glucose units are joined through carbon atoms 1 and 4 and all glucose unit in cellulose chain rotated 1800 to each other which make to form a straight chain (Sjöström, 1993). Cellulose molecules are relatively linear and have strong tendency to form intra and intermolecular hydrogen bonds. Bundles of cellulose molecules are thus aggregated together in the form of micro-fibrils, in which highly ordered (crystalline) regions alternate with less ordered (amorphous) regions. The crystalline regions in which the linear molecules of cellulose are bounded laterally by hydrogen bonds are characterized by the cellulose lattice which extends over the entire cross-section of the micro-fibrils. This crystalline region is bounded by a layer of cellulose molecules that exhibit various degrees of parallelism. The less ordered region is called the Para crystalline or amorphous region. The disordered region allows disintegration of the cellulose by hydrolysis into rod-like particles with aqueous, non-swelling, strong acid. Micro-fibrils build up fibrils and finally cellulose fiber bundles. As a consequence of its fibrous structure and strong hydrogen bonds cellulose has a high tensile strength and is insoluble in most solvents. Orientation of the linkages and additional hydrogen bonding makes the polymer rigid and difficult to break (Hamelinck et al, 2005). The basic repeating unit of cellulose is cellobiose. The fibrils are embedded in hemicelluloses and lignin. Under normal conditions, cellulose is extremely insoluble in water, which is of course necessary for it to function properly as the structural framework in plant cell walls (O'Sullivan, 1997).

Cellulose being relatively resistant to oxidation, lignin and other coloring matters can be removed with bleaching agents without appreciable damage to the strength of pulp. The alpha or true cellulose content of a fibrous material does not affect directly its palpability, but the

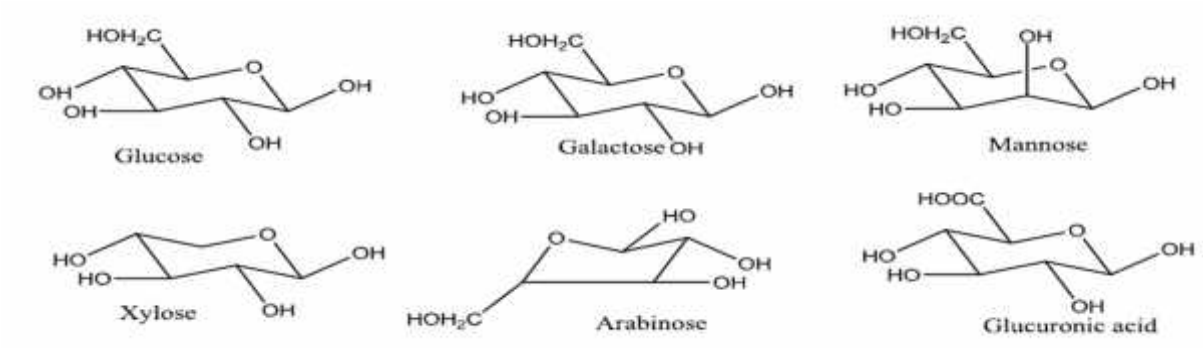
higher the alpha-cellulose content of a material, the higher the yield of fully delignified, bleached chemical and semi chemical pulps (Sjostrom, 1993).

Hemicelluloses

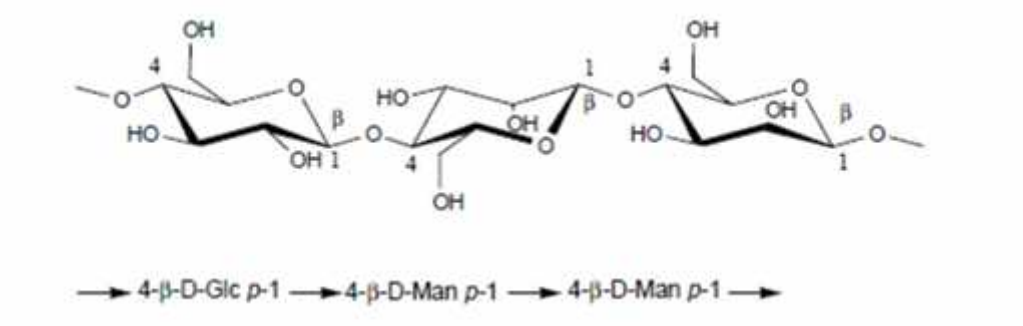
Hemicelluloses, the second most abundant natural polysaccharides after cellulose, comprise roughly one-fourth to one-third of most plant materials, and this amount will vary according to the particular plant species, such as for wheat straw is 33- 36 %. The hemicelluloses are usually defined as the polysaccharide part of plant tissue, which is accessible to the action of dilute acids and alkalis. They are chemically complex and comprise a mixture of sugar monomers. For instance, besides glucose, sugar monomers in hemicellulose also including arabinose, galactose, mannose, and xylose, etc. Hemicelluloses exhibit lower molecular weights with little strength than cellulose (Sjostrom, 1993).

When wood is freed from extractives (compounds which are soluble in cold water or in neutral organic solvents) and is then carefully freed from lignin, it yields a fibrous product termed holocellulose, which represents the sum total of cellulose and other polysaccharides; the latter are usually termed hemicelluloses (polyoses). Pulping processes remove not only lignin (imperfectly) but also some of the less resistant hemicelluloses; so, holocellulose cannot be obtained by ordinary pulping operation.

The hemicelluloses contain mainly sugar units other than glucose (such as Xylose, mannose, Arabinose, rhamnose, galactose, etc.). Usually the dominant unit in the hemicelluloses is Xylose, but frequently mannose units are present in appreciable amounts, especially in the case of the hemicelluloses of coniferous woods. The hemicellulose fractions which contain Xylose (and uronic acid) units are often termed ‘xylans’ or more loosely ‘pentosanes’. Those contain mannose units linked to each other and to glucose units have been referred as ‘mannans’.



(a)



(b)

Figure 4 Components (a) and Structures (b) of the major hardwood hemicelluloses *(Christiane , 2005).*

The hemicelluloses (when freed from lignin) swell more than does cellulose and are in part dispersible in water. They have adhesive properties not shared by cellulose. Whereas cellulose is fibrous, hemicelluloses are non-fibrous. Whereas cellulose is quite insoluble in cold alkali, hemicelluloses are quite soluble in dilute caustic soda. In any chemical pulping operations, some of the initial hemicelluloses are retained in the pulp. A portion of the less resistant hemicelluloses is removed during digestion, and their degradation products are then found in the spent liquors (Sjostrom, 1993).

In the case of pulps freed from lignin by adequate and controlled bleaching, the hemicelluloses have been shown repeatedly to contribute greatly to tensile and bursting strength and to folding endurance of the pulp sheet. Both the quantity and the type of hemicelluloses in a pulp influence the pulp properties and the type of paper that can be made from such a pulp. There are certain disadvantages also about their presence. These are undesirable for dissolving grade pulp. In the case of certain bleached pulps, these are responsible for a loss in brightness of the bleached pulp on storage or aging.

Lignin

Lignin is the third most abundant natural polymer present in nature after cellulose and hemicellulose. It is an amorphous cross-linked resin with no exact structure. It is the main binder for the agglomeration of fibrous cellulosic components while also providing a shield against the rapid microbial or fungal destruction of the cellulosic fibres. Lignin is a three-dimensional, highly branched, polyphenolic substance that consists of an irregular array of variously bonded “hydroxy-” and “methoxy-”substituted phenylpropane units. These three general monomeric phenyl propane units exhibit the p-coumaryl, coniferyl, and sinapyl

structures (Figure 5). In lignin biosynthesis, these units undergo radical dimerisation and further oligomerization, and they eventually polymerize and cross-link. The resonance hybrids of the radical formed on oxidation of coniferyl alcohol illustrates the positions where radicals' dimerizations occur during lignin formation (Sjostorm, 1993).

Pulping is basically and mainly a delignification process employing inorganic acids or alkalis and other compounds or organic solvents (organosolv method) and compounds or by employing biological agents such as certain fungi which will selectively attack on lignin, causing its degradation and consequent dissolution. The amount and reactivity of lignin have a marked effect on the palpability of the material which depends upon the type of raw material (softwoods, hardwoods, bamboos, etc.). During most pulping reactions, components other than lignin are simultaneously removed. The character of pulp depends upon the form and amount of energy supplied for accomplishing the separation such as chemical, mechanical or a combination of the two forms of energy are utilized. In general, when chemical energy alone is supplied, completely separated fibers are obtained; whereas in mechanical and semi chemically pulping (combination of mechanical and chemical processing) whole fibers, fiber bundles, damaged fibers, and fiber fragments are produced. Bleaching of pulp is also a process mainly employed for further purification of the pulp by removing the remaining portions of lignin and other color bodies in the pulp.

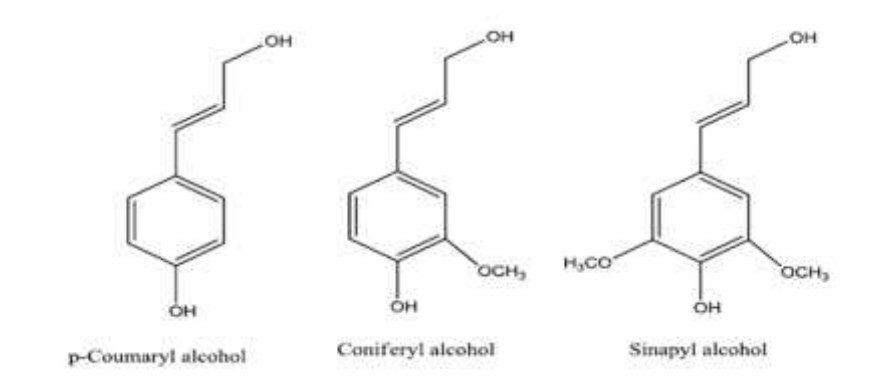


Figure 5 p-Coumaryl, coniferyl, and sinapyl structures *(Christiane , 2005)*

Extractives and ash

Extractive in straw includes cutin, suberin, tannins, waxes and minerals found in the cell walls. Cutin and waxes are attached to the epidermal walls on plants surface. Cutin is three-dimensional polyester composed of w-hydroxy and mid-chain hydroxy fatty acids. It is often esterified with phenolic acids, and maintains a close association with pectin in the epidermal cell walls. Cutin appears to be embedded in wax and pectin; these components serve as

diffusional barriers that impede ruminal digestion of the intact tissue. Suberin is a functional component of cell walls. The polyesters that appear in suberized tissue can be esterified with phenolic monomers, oligomers or lignin.

Ash is a term generally used to refer to inorganic substances such as silicates, sulfates, carbonates, or metal ions (Rydholm, 1965). The fraction of inorganic compounds also called ash constitutes of Ca, K, Mg, P, Mn, Fe, Si, Al and Na salts and the actual composition varies between wood species and is also influenced by the local compositions of the soil (Theliander et.al., 2002). Silicon is an important inorganic element in plant cell walls and mainly present in the form of silica in the walls of epidermal cells and leaf hairs. High amount of extractive substance in the raw material may result in poor quality of pulp and resulting in higher operating costs and an increased incidence of quality defects. Generally the distribution range of different cellulosic feedstock composition is presented in table 6.

Table 6 The distribution range of different cellulosic feedstock composition

Lignocellulosic materials	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Hardwood stem	40-45	24-40	18-25
Softwood stem	45-50	25-35	25-35
Bamboo	41-49	28-32	20-22
Corn cobs	45	35	15
Wheat straw	45	30	15
Sorted refuse	60	20	20
Leaves	15-20	80-85	0
Cotton seed hairs	80-95	5-20	0
Coastal Bermuda grass	25	35.7	6.4
Switch grass	45	31.4	12

(Sun & Cheng, 2003)

2.3. Wheat Production in Ethiopia

Wheat was one of the first domesticated food crops and for 8000 years has been the basic staple food of the major civilizations of Europe, West Asia and North Africa. Today, wheat is grown on more land area than any other commercial crop and continues to be the most important food grain source for humans. Its production leads all crops, including rice, maize and potatoes (Umar, 1999).

The total wheat output in 2015 was 734 million tonnes and in 2016 it is put at around 724 million tonnes, down 1.4 %, or 10 million tonnes. The decline would be mostly the result of expected lower year-on year outputs in the EU of 6.5 million tonnes, in Morocco of 5 million tonnes, in Ukraine of 4.5 million tonnes, and in the United States of 1.4 million tonnes (FAO , 2016). Total wheat production for 2017/18 is projected at 737.8 million tons, the second highest total on record and the global wheat consumption is projected down slightly from last year's record with reduced feed and residual usage partially offset by increased food use (USDA, 2017).

Ethiopia is the second largest wheat producer in Sub-Saharan Africa next to South Africa and Wheat is one of the major staple crops in the country in terms of both production and consumption. In terms of caloric intake, it is the second most important food in the country behind maize (FAO, 2014). Wheat is mainly grown in the highlands of Ethiopia, which lie between 6 and 16° N, and 35 and 42°E, altitudes ranging from1500 to 2800 meters above sea level and with mean minimum temperatures of 6^{o}C to 11^{o}C (Hailu, 1991; MOA, 2012). There are two varieties of wheat grown in Ethiopia: durum wheat, accounting for 60% of production, and bread wheat, accounting for the remaining 40% (Berghetal. 2012). Oromia accounts for over half of national wheat production (54%), followed by Amhara (32%); Southern Nations, Nationalities and Peoples (SNNP) (9%); and Tigray (7%) (CSA, 2013). Of the current total wheat production area, about 75% is located in the Arsi, Bale and Shewa wheat belts (MOA, 2012).

In Ethiopia wheat is a cool weather grain crop that is commonly grown at elevations ranging from 1,500 to 3,200 meters above sea level, with major wheat growing belts in the southern (i.e., Arsi and Bale) and central (i.e. Shewa and Gojam) highlands . The crop is grown mostly during the main (meher) rainy season from June to September and harvested from October through January. In the 2012/13 Meher season, about 4.8 million farmers grew wheat, and more than 1.6 million hectares of land were dedicated to wheat cultivation, constituting 13.5 % of the national grain area (CSA, 2013). Wheat production during 2012/13 Meher season was 3.4 million metric tons, accounting for 15% of the total grain output in the country (CSA, 2012, 2013). Official statistics indicate that wheat production in 2012/13 was 18% higher than in the previous year, and wheat production has steadily increased over the last decade.

Ethiopia is the 19th largest wheat (*Triticum Aestivum*) producer in the world and the second from Africa next to Egypt (USDA, 2016) and the production of wheat in Ethiopia for the year

2015/16 was 4.2 million tonnes (CSA, 2016). Wheat in Ethiopia is primarily grown in the Amhara, Oromia, Tigray and Southern Nations, Nationalities and Peoples (SNNP) regions. These regions account for more than 90% of national wheat production (sciencecodex.com).

Table 7 Wheat production by Country in 1000 MT 2016

Rank	Country	Production (1000 MT)
1	EU-27	145,270.00
2	China	128,000.00
3	India	90,000.00
4	Russian Federation	72,000.00
5	United States	63,156.00
6	Canada	30,500.00
7	Australia	27,500.00
8	Ukraine	27,000.00
9	Pakistan	25,300.00
10	Turkey	17,500.00
11	Kazakhstan	16,500.00
12	Iran, Islamic Republic Of	15,500.00
13	Argentina	14,400.00
14	Egypt	8,100.00
15	Uzbekistan	7,200.00
16	Brazil	6,000.00
17	Afghanistan	5,100.00
18	Mexico	3,900.00
19	Ethiopia	3,800.00
20	Iraq	3,400.00
21	Serbia	3,000.00
22	Morocco	2,800.00
23	Belarus	2,600.00
24	Syrian Arab Republic	2,400.00
25	Azerbaijan	2,000.00

(United States Department of Agriculture, 2016)

Wheat yield has been increasing since 2002 due to government initiatives that encouraged farmers to adopt improved seed varieties, increase fertilizer distribution, and expanded

agriculture extension in rural areas. As the data obtain from Central Statistical Agency the 2015/16 Annual Agricultural Sample Survey (AAgSS) indicates that both the largest grain cropped land area and the highest volume of production obtained in the current Meher season is reported for Oromia, Amhara, SNNP, Tigray and Benshangul Gumuz Regions.

From the literature, ratio of wheat straw to grain production was estimates approximate 1.3 tons of wheat straw per ton of grain (Bowyer , Howe, Pepke, Bratkovich, Frank, & Fernhonz, 2014). Therefore the wheat straw production for the year 2014/15 and 2015/16 in Ethiopia would be 5.499 and 5.473 million tons respectively.

Table 8 Estimate of area, production and yield of crops for 2014/15 and 2015/16, Meher Season

	Area In Hectares		Production In Quintals		Yield(Quintals/Hectare)	
	2014/15	2015/16	2014/15	2015/16	2014/15	2015/16
Grain Crops.	12,558,444	12,486,270	270,396,04	266,828,807		
Cereals.	10,144,252	9,974,316	236,076,624	231,287,970		
Teff.	3,016,053	2,866,052	47,506,572	44,713,786	15.75	15.60
Barley	993,918	944,401	19,533,847	18,567,042	19.65	19.66
Wheat.	1,663,837	1,664,564	42,315,887	42,192,572	25.43	25.35
Maize.	2,110,209	2,111,518	72,349,551	71,508,354	34.29	33.87
Sorghum	1,831,600	1,854,710	43,391,342	43,232,997	23.69	23.31
Finger millet	453,909	465,508	9,153,145	9,402,463	20.17	20.20
Oats/'Aja'	27,899	22,105	508,059	402,689	18.21	18.22
Rice.	46,823	45,454	1,318,218	1,268,064	28.15	27.90

(CSA E. , 2016)

(a) **(b)**

Figure 6 (a) shows the traditional harvesting of wheat in Ethiopia using sickle while (b) shows the pile of wheat straw harvested using modern machine.

2.4. Production and Consumption of Pulp and Paper in Ethiopia

Ethiopian pulp and papers Share Company was the first paper manufacturing industry, established and gets legal recognition in August 29, 1955 E.C. The shareholders of this company were ministry of finance, Ethiopian Development Bank, Ethiopian Investment Bank and Panser and Witmor Company with a beginning capital of 50,000 birr. When it started production in 1962 the total expenses were 22 million birr and at the same time its capital was pumped to 10 million birr.

During its establishment the company produces paper product of 25 ton daily and 8,500 ton annually. According to the study made in 2006, EPPSC has got a 38% market share in corrugated box and 25% market share in wrapping paper. The factory has opened job opportunity for 684 employees out of which 534 are permanent and 150 are contract. In addition to that EPPSC has organized more than 158 youths in association for the processing and supplying of recyclable paper.

Currently in Ethiopian there are about 25 companies that are involved in paper making, packaging and trading businesses i.e. two governments owned, four share company and the rests are owned privately. But only two factories namely the Ethiopian pulp and paper share company and Barguba private limited company, are manufacturing paper products and covering a limited amount of local demands for the product (ECCIDI, 2016).

Table 9 Pulp processes and paper converting industries in Ethiopia.

No.	Company Name	Designed Capacity (ton per yr.)	Attainable capacity (ton per yr.)	Product Type
Government owned				
1	Ethiopia Pulp & Paper S.C*	16000	6000	Printing and Writing Paper, Stationery Paper, Paper board, Corrugated Box,

				Wood free and manila papers Kraft liners, test liners, fluting papers etc.
2	Anmol Products Ethiopia PLC	14000	12000	Printing writing papers Wood free and manila papers Kraft liners, test liners, fluting papers etc
3	Addis Ababa Cement Bag Factory			
Share Company				
1	Yekatit Paper Converting Enterprise	1600	1400	paper products
2	Tana Pulp & Paper S. Co.*			
3	Ethio China Saudi International Co. Ltd (Ethiopia Branch)*			
Private Limited company				
1	DA packaging plc.	up to 10,000	2000	Kraft liners, test liners, fluting papers etc.
2	Barguba Trading P.L.C	up to 10,000		paper products
3	Three sisters pulp and paper manufacturing enterprise	10000	1500	Kraft liners, test liners, fluting papers etc.
4	Suzo plc.	2000	2000	Roll papers for tissue manufacturers
5	Mamco Paper Products Factory Plc.			Paper products

6	Moab Paper P.L.C			Paper products
7	Nice Paper			Paper products
8	Mohammed Mohasin Ahmed paper products			Paper products
9	Burayu Development PLC (Burayu Paper Industry Industry)*			
10	JMD Overseas*			
11	DA Packaging			
12	Fetlework file folder & packaging plant			
13	Adapty Packaging PLC			
14	Shiv Pack PLC			
15	Alem Kiros Carton Industry PLC			
16	Minaye Packaging PLC			
17	Balaje Packaging PLC			
18	East Africa Tiger Brands Industry PLC			
19	Seven Hills Packaging Solutions PLC			
20	Unlimited Packaging PLC			

*Companies that are established and undergo expansion projects

**Companies under project and will expected to start in the next five years (ECCIDI, 2016)

Ethiopia as a developing country has significantly very low paper and paper board consumption per capita of 0.43 kilogram /person /year when compared with the world average of 54.48 kilogram/person/year in 2005 (WRI, 2005).

The overall amounts of pulp and paper imported and its associated costs in USD for the last eight years, 2009-2016 was presented in table 10, which is obtained from Ethiopian construction inputs and chemicals development institute. Ethiopia has imported 51,388 tons of pulp and 844,446 tons of paper with 40.8 and 948.3 million USD for pulp and paper

respectively from year, 2009-2016. The average cost in USD per kg was estimated 0.8 for pulp and 1.12 for paper products (ECCIDI, 2016).

Table 10 The quantities imported and associated costs of pulp and paper in Ethiopia from 1909-2016.

Year	Pulp		Paper	
	Quantity (kg)	Cost (USD)	Quantity (kg)	Cost (USD)
2009	4,013,981	2,200,849.781	79,880,956.90	84,651,044.37
2010	1,759,353.89	1,632,848.177	74,798,037.69	82,450,928.73
2011	6,646,105	5,668,217.546	84,172,647.48	101,833,766.21
2012	8,617,781.00	6,759,688.00	101,756,192.18	116,359,837.88
2013	10,216,188.00	8,059,794.46	107,519,199.07	132,301,772.92
2014	5,101,365.00	4,214,596.83	136,955,682.27	127,237,688.80
2015	7,181,482.36	6,025,699.99	108,632,555.78	135,953,748.31
2016	7,852,516.85	6,310,023.24	150,731,302.60	167,529,679.09

2.5. Techniques of pulping

The pulping techniques of the non-wood plant materials are the modified methods of those which have been used in wood pulping. The pulping techniques can be categorized as mechanical, thermal, chemical and semi-chemical methods. To date a number of related hybrids of pulping methods that use a combination of chemical, thermal and mechanical treatments are employed in the separation of the fibres. Some common hybrid methods that have been used in pulping both wood and non-wood plant materials include thermo-mechanical pulping (TMP) and chemical thermo- mechanical pulping (CTMP) (Europian Commission, 2001).

Table 11 General Classification of Wood Pulping Techniques *(Amsalu , 2015)*

Process category	Fiber separation method	Fiber quality	Example
Mechanical	Mechanical energy	Short, weak, unstable, impure fibers	Stone ground wood, refiner mechanical pulp

Semi–mechanical or semi-chemical	Combination of chemical and mechanical treatments	"Intermediate" pulp properties(some unique properties	High yield Kraft ,high yield sulphite
Chemical	Chemicals and heat	Long, strong, stable fibers	Kraft, sulphite, soda

2.6. Chemical Pulping

Chemical pulping employs chemical reagents to effect a separation of the cellulose fibres from other wood components. Wood chips are cooked with suitable chemicals in aqueous solution, usually at elevated temperatures and pressures with the objective of dissolving the lignin and other extraneous compounds, leaving the cellulose intact and in fibrous form.

Chemical (i.e. Kraft, soda, and sulfite) pulping involves "cooking" of raw materials (e.g. wood chips) using aqueous chemical solutions and elevated temperature and pressure to extract pulp fibers. In Kraft pulping, white liquor, a water solution of sodium sulphide (Na_2S) and sodium hydroxide (NaOH), is used under high temperature and pressure to chemically dissolve the lignin that binds the cellulose fibres of the wood together while the similar soda process uses only NaOH. This cooking liquor is mixed with the wood chips in a reaction vessel (digester). After the wood chips have been "cooked," the contents of the digester are discharged under pressure into a blow tank. As the mass of softened, cooked chips impacts on the tangential entry of the blow tank, the chips disintegrate into fibers or "pulp." The pulp and spent cooking liquor are subsequently separated in a series of brown stock washers. Kraft pulping is by far the most common pulping process used by all over the world for virgin fiber, accounting more than 80 percent of total pulp production.

The cooking liquor in the sulfite pulping process is an acidic mixture of sulfurous acid (H_2SO_3) and bisulfite ion (HSO_3^-). In preparing sulfite cooking liquors, cooled sulfur dioxide (SO_2) gas is absorbed in water containing one of four chemical bases - magnesium (Mg), ammonia (NH_3), sodium (Na), or calcium (Ca). The sulfite pulping process uses the acid solution in the cooking liquor to degrade the lignin bonds between wood fibers. Sulfite pulps have less color than Kraft pulps and can be bleached more easily, but are not as strong. The efficiency and effectiveness of the sulfite process is also dependent on the type of wood furnish and the absence of bark.

2.6.1. Alkaline Methods of Pulping

The soda process and the Sulphate (Kraft) process are the two principal alkaline pulping techniques and the basis for several modified alkaline processes, including Kraft pulping after a pre hydrolysis step for the production of dissolving pulp. Sodium hydroxide is the principal cooking chemical in both processes, while in Sulphate pulping sodium sulphide is an additional pulping component. Both processes received their names from the regeneration chemicals used to compensate for the loss of sodium hydroxide, namely sodium carbonate (soda) and sodium Sulphate, respectively.

Kraft (Sulphate) Process

The Kraft process uses sodium hydroxide (NaOH) and sodium sulfide (Na_2S) to pulp wood. It is the dominant pulping process in the pulp and paper industry. About 130 million tonnes / year of Kraft pulp are produced globally accounting for two-thirds of the world's virgin pulp production and for over 90% of chemical pulp. The high strength of Kraft pulp, the ability of the process to handle almost all species of soft wood and hard wood, and the favorable economics due to high chemical recovery (about 97%) gives the Kraft process an advantage over other pulping processes (Honghi & Esa, 2002).

Soda process

Soda process, the first process to manufacture chemical pulp, was invented by Hugh Burgess in 1851, employed caustic soda (sodium hydroxide) solution for cooking. For papermaking soda-AQ pulping, compared to Kraft pulping is more environmental friendly pulping process. A higher pulp yield with soda-AQ pulps, compared to soda method, was attributed to higher hemicellulose retention (Atik, 2002).

Previous studies have shown that the modified pulping process by AQ addition in laboratory and industrial scales resulted in more uniform pulps with lower rejects, higher pulp yield at constant kappa number, and lower consumption of alkali (Akgul, M., and Tozluo, A., 2009). Similar results by Kamthai (2007) and Miller and Gounder (1986), reported that the addition of AQ to soda pulping can raise the delignification rate and protect cellulose degradation, which was demonstrated by the quantity of screened pulp yield.

2.6.2. Acidic Method of Pulping

The 'sulfite process' which is the so called acidic method of pulping was invented during 1857 by an American chemist, B.C. Tilaghman who observed the effect of sulphurous acid in softening wood. He obtained cellulose fibers by treating wood with bisulphate-sulphurous

acid solutions under high temperature and pressure. The base used was calcium and later magnesium. Sodium and ammonium are also used as base, which are having advantages in liquor-recover operations. Sulphite pulps are relatively light in color, are easily bleached to a high-white color, have moderately good strength properties. Sulfite pulp is almost pure cellulose and is used in fine papers.

In general the characteristic properties of the isolated pulp depend on the techniques of processing most especially the chemical method. The different chemical pulping methods that are commonly employed in pulping both wood and non-wood materials are given in table 12.

Table 12 Summary of commonly used chemical pulping methods

Chemical pulping Methods	Chemicals used	Properties of pulp isolated	Common uses of isolated pulp
Sulphite	Sulphurous acid/ sodium sulphite	High flexibility and requires very little bleaching	Used in making paper for special purposes
Kraft/ Sulphate	Combination sodium hydroxide and sodium sulphide	Strong, low brightness(dark brown)	Making boxes, paper bags and wrapping paper. Can also be used for writing paper and paperboard when bleached
Soda	Sodium hydroxide and anthraquinone	Have properties similar to those of sulphite.	Ideal for all paper uses

(Kamoga, Byaruhanga, & Kirabra, 2013)

2.7. Process Descriptions of Chemical Pulping

In chemical pulping, chips and chemicals in aqueous solution are cooked together in a pressure vessel which can be operated on a batch or continuous basis. In batch cooking, the digester is filled with chips through a top opening, the digestion chemicals are added, and the contents cooked at elevated temperature and pressure. Once the cook is complete, the pressure is released, “blowing” the delignified pulp out of the digester and into a holding tank. The sequence is then repeated. In continuous digesting, pre-steamed chips are fed into the digester at a continuous rate. Chips and chemicals are mixed together in the impregnation zone at the top of the digester and then proceed through the upper cooking zone, the lower cooking zone, and the washing zone before being blown into the blow tank (Herbert , 2006).

Kraft pulping process whereby timber is converted into paper involves six steps. The first four steps convert the logs into a mass of cellulose fibres with some residual lignin using a

mixture of physical and chemical processes. The fifth step is bleaching which removes the remaining lignin and the final step is sheet forming which spreads out the pulp into smooth, pressed sheets (often with chemicals added to provide particular properties such as color or water resistance). For some papers (e.g. cardboards and 'brown paper') the bleaching step is unnecessary, but all white and colored papers require bleaching. The manufacturing processes are outlined as follows.

2.7.1. Raw Material Preparation

Wood is delivered to the Kraft mill in one of two ways: whole logs and sawmill chips (residuals from sawmills). The logs have their bark removed, either by passing through a drum debarker or by being treated in a hydraulic debarker. The drum debarker, which consists of a slightly inclined, rotating drum is best suited to small diameter logs. The hydraulic debarker, which uses high pressure water jets, can handle large diameter logs. The removed bark is a good fuel, and is normally burnt in a boiler for generating steam.

After debarking, the logs are chipped by multi knife chippers into suitable sized pieces, and are then screened to remove overlarge chips. The thickness of the chips is the most important parameter, as this determines the speed and the thoroughness of the impregnation of the cooking chemicals into the wood chip.

2.7.2. Cooking

The "cooking process" is where the main part of the delignification takes place. Here wood chips are heated in a solution of NaOH and Na_2S in a pressure cooker, during which time a lot of the lignin (the reinforcing substance that make tree cells wood hard and 'woody' rather than soft like those of other plants) is removed from the wood. During the cooking process the sodium sulfide serves to buffer and to sustain the cooking reaction as the original sodium hydroxide content is consumed through reaction with the lignin and carbohydrates in the wood. Small amounts of sulfide react with lignin in the wood, giving rise to the odors characteristic of Kraft mills. These are due to a number of volatile compounds which include methyl mercaptan and dimethyl sulfide. The quality of pulp produced is closely related to the sodium sulfide-sodium hydroxide ratio (Sulphidity) in the cooking liquor as well as the cooking parameters. The formation of methyl mercaptan and dimethyl sulfide is directly proportional to the concentration of sodium sulfide in the cooking liquor, to the temperature employed, and to the duration of the cook. Hardwoods also produce more malodorous

compounds than softwoods. Depending on the pulping parameters the combined mercaptan and sulfide release may be 2-7 pounds per ton of pulp produced (Morton , Irwin , George , William , Navarre, & Yerger, 1969). At the completion of the cook, the residual internal pressure in the cooking vessel is used to discharge the pulp into a collecting vessel, referred to as the blow tank. Steam and gases released in the blow tank are usually vented through direct or indirect contact condensers, where heat is recovered. The pressure is then released suddenly, causing the chips to fly apart into fibres. This process is, like any chemical reaction, affected by time, temperature and concentration of chemical reactants. Time and temperature can be traded off against each other to a certain extent, but to achieve reasonable cooking and chips delignification a balance must be achieved.

2.7.3. Pulp washing

Because of the high amounts of chemicals used in the cooking wood in Kraft pulping, the recovery of the chemicals is of crucial importance. The process where the chemicals are separated from the cooked pulp is called pulp washing. A good removal and recovery of chemicals (inorganic and organic) is necessary for several reasons:

- The dissolved chemicals interfere with the downstream processing of the pulp
- The chemicals are expensive to replace
- The chemicals (especially the dissolved lignin) are detrimental to the environment

There are many types of machinery used for pulp washing. Most of them rely on displacing the dissolved solids (inorganic and organic) in a pulp mat by hot water, but some use pressing to squeeze out the chemicals with the liquid. An old, but still common method is to use a drum, covered by a wire mesh, which rotates in a diluted suspension of the fibers. The fibers form a mat on the drum and showers of hot water are then sprayed onto the fibre mat.

2.7.4. Pulp screening

Apart from fibres, the cooked pulp also contains partially uncooked fibre bundles and knots. Modern cooking processes (together with good chip screening to achieve consistent chip thickness) have good control over the delignification and produce less "rejects". Knots and shaves are removed by passing the pulp over pulp screens equipped with fine holes or slots.

2.7.5. Bleaching

Although pulp cooking can safely dissolve up to about 90% of the lignin without degrading the cellulose fiber, additional delignification can be done by bleaching. Bleaching is the treatment of cellulosic fiber with chemicals to increase brightness. Brightness may be achieved by either lignin removal (delignification) or lignin decolorization. Lignin remains a major constituent of pulp even after digestion by chemical pulping. For example, Kraft pulp may contain up to 6% lignin based on its dry weight. Unbleached ground wood spruce pulp may contain 27% lignin (Dauglas & Reeve, 1987).

The strength of paper is largely due to the chemical bonds (hydrogen bonds) formed between cellulose fibers. Although longer and more severe pulping might remove more of the lignin, thus reducing the amount of bleaching needed, the cellulose molecules might be degraded and their bonding power diminished. The removal of lignin by bleaching is regarded as a continuation of the pulping process, although somewhat gentler and less destructive, but bleaching too can degrade cellulose if done improperly.

Lignin imparts a color to the raw pulp (hence its name “brown stock”) and unless removed, will continue to darken with age (note the yellowing, darkening, and embrittlement of newspaper exposed to sunlight). Bleaching by removing the lignin gives higher brightness to the paper than is possible by leaving the lignin in the pulp and brightening by decolorization, and also leads to a more durable and stable paper. In addition to the removal and decolorization of lignin, bleaching serve to clean the pulp of dirt and foreign matter that escaped the digestion process. Bleaching also removes hemicelluloses and extractives (hemicelluloses are nearly completely removed for the production of dissolved pulps). Bleaching pulp adds significantly to its value as market pulp because the demand for bleached paper is increasing.

Pulp produced by the Kraft process is brown and this presents no problem for certain uses, e.g. for sack paper, most corrugated boxes, some bag paper etc. However, a major proportion of the Kraft pulp that made is used for white or colored papers such as writing and printing papers, and then the pulp needs to be bleached.

Bleaching of high-yield chemical pulps is achieved by decolorizing with either an oxidizing agent (combines oxygen) or a reducing agent (combines hydrogen). Chlorine gas, sodium hypochlorite, chlorine dioxide, oxygen gas, and hydrogen peroxide are oxidants. Sodium hydrosulfite is a reductant. Alkali is used to remove the solubilized lignin from the cellulose.

Since the 1930s, chlorine gas has been the predominant chemical used for the delignification of pulp. Chlorine dioxide can brighten pulp without damaging the cellulose. Oxygen is comparatively inexpensive and is now coming into its own both for delignification (immediately after digestion and before the bleach cycle) and as a supplement in the first extraction (alkali) stage of the bleach sequence. Hydrogen peroxide is expensive, so it is used much less than other bleaching agents. The effectiveness of a bleaching agent, although a major factor in determining its use in a pulp bleaching sequence, may be offset by the cost of the chemical or the equipment needed to handle it.

A critical determinant in choosing a bleaching chemical is the ‘‘selectivity” of the agent. Selectivity refers to the capacity of the chemical to attack lignin while doing minimal damage to the cellulose fibers. Unbleached pulp (brown stock) contains high levels of lignin; therefore less selective chemicals (e.g., oxygen and chlorine) can be used in the initial stages of the bleach cycle. With further delignification and lower residual lignin content of the pulp, more chemical is available to react with the cellulose and pulp strength may suffer.

Chlorine dioxide and hydrogen peroxide are highly selective, thus they react rapidly with lignin but affect cellulose very little. The highly selective chemicals are generally used in later bleach stages when the lignin content is low and the cellulose is susceptible to degradation. However, both chemicals are expensive and are therefore used sparingly. Sodium hydrosulfite, a reducing agent, and hydrogen peroxide are used for bleaching lignin-rich mechanical pulp.

The pulp and paper industry has developed a series of shorthand descriptors for the multistage bleaching sequences. The following abbreviations are used to designate the bleaching agents:

C Chlorination
E Extraction with sodium hydroxide
H Hypochlorite (sodium or calcium)
D Chlorine dioxide
P Hydrogen peroxide
O Oxygen
N Nitrogen dioxide
Z Ozone

Bleaching sequences are designated by listing each treatment serially. For example, "CEDED" represents a commonly used five-stage bleaching sequence consisting of a first-stage chlorine treatment, followed by a second-stage alkali extraction stage, followed by a third-stage chlorine dioxide treatment, followed by a fourth-stage alkali extraction treatment, and a final fifth-stage chlorine dioxide treatment. Washing is conducted between each chemical application.

Bleaching is achieved through chemical reactions. Operating conditions are related to temperature, time, chemical concentrations, and degree of acidity or alkalinity (pH). These factors must be kept in balance to achieve the desired degree of bleaching, while at the same time minimizing damage to the cellulose fiber. In addition, the "consistency" (amount of fiber being bleached in relation to the volume of liquid) of the fiber slurry being bleached affects chemical penetration and therefore must also be controlled.

Hydrogen peroxide is a very effective cellulose-preserving bleach agent and is well suited for improving the brightness of highly lignified pulps, such as mechanical ground wood or chime -mechanical pulps, without significantly reducing its yield.

$$H_2O_2 + HO^- \longrightarrow HOO^- + H_2O \quad \text{at pH 11.}$$

Hydrogen peroxide is an extremely versatile de lignifying chemical and has been proposed for use as a chip pretreatment before Kraft pulping and as a delignified in the pre bleach stage prior to, or as a substitute for, the C, CD, or Dc pre bleaching stages. It is also used in association with sodium hydroxide in alkaline extraction. Because of its high cost; hydrogen peroxide is used most often in the later stages of pulp bleaching. Peroxide is used in the intermediate stages of the bleaching sequence as a replacement for hypochlorite or chlorine dioxide. It is frequently used as the last stage in the bleach sequence where it can add a few points of brightness to the pulp and improve its brightness stability.

2.7.6. Paper making

Paper making is the process whereby pulp fibres are mechanically and chemically treated, formed into a dilute suspension, spread over a mesh surface, the water removed by suction, and the resulting pad of cellulose fibres pressed and dried to form paper.

The mechanical treatment of the fibre normally takes place by passing it between moving steel bars which are attached to revolving metal discs the so called refiners. This treatment has two effects: it shortens the fibre (fibre cutting) and it fibrillates the fibre. The latter action

increases the surface area, and as the fibres bond together in the paper sheet by hydrogen bonding, the increased surface area greatly increases the bonding and strength of the paper. Paper strength is dependent on the individual fibre strength and the strength of the bonds between the fibres. It is usually the latter, which is the limiting factor. Refining increases the inter fibre bonding at the expense of the individual fibre strength, but the net result will be an increase in paper strength. Pressing and calendaring (feeding through rollers) increase density and promote smoothness. Various chemicals are added, e.g. to give water resistance, to give increased strength, to produce colored paper, or to serve as inorganic fillers.

Henry and Sealy Fourdrinier have given their name to the Fourdrinier paper machine, the first of which was first used in 1804. The stock is diluted to 0.5-1.0% consistency, and then flows as a flat stream onto an endless travelling wire screen. Water (containing a considerable amount of filter and fine material) is extracted through the wire screen, and this is then circulated back to dilute the oncoming stock. The wire and the web pass over suction boxes, and the web is finally removed from the wire at about 20% solids. After being pressed to some 35-40% solids, the web passes round a large number of steam heated cylinders to be dried until it finally contains some 95% solids and 5% water.

Modern developments have tended towards so called twin-wire machines, which overcomes the difference in two-sidedness caused by water draining through the bottom side of the web only. The twin wires are nowadays usually synthetic fibre fabrics, between which the stock is introduced. The papermaking from non-wood materials was described and summarized as used by Anonymous (1998) and Douglas (1997) is shown in figure 9.

2.8. Ancillary Processes in Chemical Recovery

A variety of ancillary processes are used to recover the chemicals used at various stages of the process. The dilute liquor from the pulp washing (containing the dissolved inorganic and organic solids) is called "black liquor". The dissolved organics have to be removed for environmental reasons, and their burning also generates most of the heat energy required by the Kraft mill. The dissolved sodium hydroxide and sodium sulphide are regenerated so that they can be reused in the white liquor, and thus the escape of an environmental pollutant is prevented. The four steps involved in chemical recovery are outlined below.

Evaporation

The black liquor from the pulp washing contains 15-17% solids, and this needs to be concentrated to about 60-70% solids before it can be burnt in the recovery furnace. "Spill liquors" may also be recovered from various places in the mill, and these dilute liquors supply further amounts of water, which needs to be evaporated. Evaporation is carried out using multiple effects, normally 5 to 7 effects in series. In systems of this type, the vapour obtained in one evaporator effect becomes the heating steam for the next effect. Process steam enters the system at effect number one, while the vapour from the last effect is condensed, producing a vacuum in the latter effects.

Black liquor evaporation consumes a substantial part of the heat energy required in the Kraft process. However, it is much more efficient to carry out this evaporation in multiple effect evaporators than in the subsequent recovery boiler. Because of this, there has been a constant effort to try to reach higher liquor solids from the evaporators. The limit is set by the increase in viscosity and boiling point that takes place when the solids increase.

Combustion of the black liquor in the recovery boiler

The recovery boiler burns the organic material in the concentrated black liquor, at the same time reducing the oxidised sulphur compounds to sulphide. The burning of the organics generates high pressure steam in the boiler, which is normally passed through a turbine, generating electricity. The low pressure steam, which is exhausted from the turbine, is then used for process heat in the pulp mill and paper mill.

A modern recovery boiler is a complex and expensive chemical reactor, and the chemical reactions taking place inside it are many and complicated. The inorganic material is recovered as molten salts (a 'smelt'), which is then dissolved in dilute alkali. The resulting liquor is called 'green liquor' and consists mainly of sodium carbonate and sodium sulphide.

Causticising

As well as dissolved sodium based chemicals (Na_2CO_3, Na_2S and smaller amounts of NaOH, Na_2SO_4 and NaCl); the green liquor also contains small amounts of suspended solids, called “dregs”. This material mostly consists of unburned carbon and insoluble metal hydroxides. To avoid problems in the causticising and lime burning processes, these dregs must be removed as much as possible. This can be done either using a gravity clarifier (as described below) or using the more recently developed green liquor filter.

Clarified green liquor and burned lime (calcium oxide) are fed continuously in metered amounts into a reaction vessel, called a slaker. The calcium oxide reacts with the water in an exothermic reaction to form calcium hydroxide or milk-of lime.

$$Ca(OH)_2 + Na_2CO_3 \rightarrow CaCO_3 + 2NaOH$$

The overall reaction for the slaking and causticizing reactions are:

$$CaO + Na_2CO_3 + H_2O \rightarrow CaCO_3 + 2NaOH + heat$$

Conversion of $CaCO_3$ to CaO

The slurry from the causticisers is pumped to a clarifier which, as for the green liquor clarifier mentioned previously, is a gravity settling device. Generally the white liquor clarifier also serves as a white liquor storage tank.

The thickened “lime mud” underflow from the clarifier contains about 40% suspended solids. This slurry is washed by diluting it with water or dilute alkali and then allowing the suspended solids to settle in another clarifier (similar to the white liquor clarifier) called “mud washer”. The resulting dilute alkali solution (“weak wash”) is used for dissolving the smelt coming from the recovery boiler to make new green liquor. The thickened washed mud is stored before a final washing and dewatering stage on a rotary drum washer.

Just as for green liquor clarification, filters are nowadays becoming more common both in white liquor clarification and in mud washing.

The Ca_2CO_3 sludge (“mud”) coming from storage is washed and dewatered on a filter, which increases the solids content to 70-80%. Washing out the sodium compounds as much as possible is important for several reasons:

· The sodium sulphide can contribute to the emission of H_2S from kiln stack

· Sodium salts melt and function as glue for the lime particles, producing rings and balls inside the kiln

After washing and dewatering, the mud is then dried and re-burnt (calcined). Usually both operations are carried out in a slowly rotating lime kiln. In such a kiln, wet mud is fed into one end of the inclined kiln, and is slowly transported towards the other end. A burner, fed with natural gas or fuel oil, supplies hot gases which travel counter currently to the mud. The mud dries and is then calcined at a temperature of about 1000-1200^{o}C in a heat absorbing reaction as follows:

$$CaCO_3 + heat \rightarrow CaO + CO_2$$

The chart below shows production process of paper from non-wood raw materials (Ekhuemelo , Oluwalana , & Adetogun , 2006).

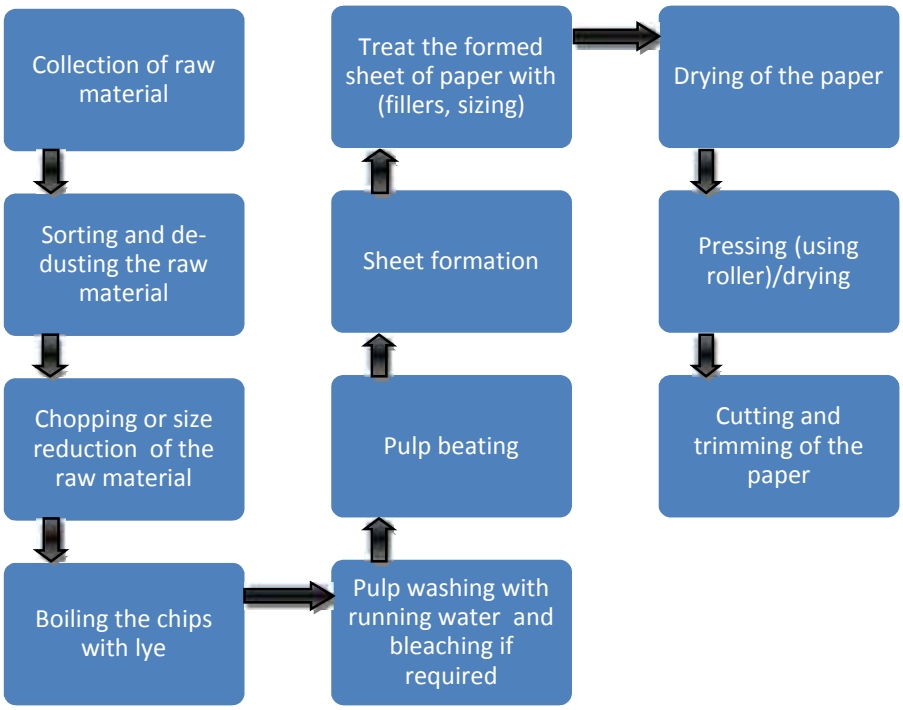


Figure 9 Chart showing the production process of paper from non-wood raw materials

2.9. Process Conditions that affect Kraft Pulping

The main variables dealt with in Kraft cooking are: the hydroxide ion concentration, the hydrogen sulphide ion concentration and the ionic strength. Temperature and liquor-to-wood ratio are also important parameters. The hydroxide ions are consumed during the cooking operation mainly when neutralizing acidic groups in carbohydrates. Hydrogen sulphide ions facilitate the lignin degradation. The ionic strength is detrimental to the pulping reaction rate when it reaches too high concentrations (Maria , April 2007).

2.9.1. Effects of Sodium Hydroxide Concentration

According to Lindgren and Lindström (1996) the bulk delignification rate is increased when the hydroxide ion concentration is increased but the rate in the residual phase is left unaffected. Although a higher concentration of hydroxide ions results in a pulp with less residual lignin. The dissolution rates of the carbohydrates also increases when the alkali

concentration increases, but the hemicelluloses are more sensitive towards alkaline degradation than the cellulose (Maria , April 2007).

The increase in concentration of caustic soda improves the delignification and provides better quality pulp with lower lignin content. Pulp yield decreases with increase of caustic soda concentration in liquor due to increase of delignification and solubilization of hemicelluloses in caustic soda. But the quality of pulp obtained at higher pulp yield is not suitable for further processing in paper industry due to high kappa number and residual lignin in pulp. A pulp kappa number equal to 26.2 was obtained at 85% Acetic acid concentration with 1% catalyst concentration at 90^{o}C temperature and 180 minutes of reaction time when liquor to straw ratio was maintained at 10 (Akhouri & Kumar , 2015).

2.9.2. Effects of Liquor to Straw Ratio

The amount of liquor to straw is an important parameter for uniform and efficient delignification reaction. According to Mortimer (1989) the delignification decreases when the L/S is increased without increasing the Effective Alkali or the H factor. In order to maintain a constant kappa number at an increased L/S ratio either the H factor or the EA has to be increased to counterbalance the lower pulping rate that is due to the lower alkali concentration. If the alkali concentration is increased rather than the H-factor, the total yield decreases at a constant kappa number (Maria , April 2007). In one of Mortimer's experiments a series were done at a constant H-factor (1600) and constant Sulphidity (30%) but with varied EA charge (16-41%) at L/S ratios of 4, 6 and 8. This showed that the selectivity was influenced by the interplay between alkali charge and liquor-to-wood ratio. The yield at a specific kappa number was reduced at higher liquor-to-wood ratios which were due to the observed increased alkali consumption (Maria , April 2007). The LSR was varied from 8 to 14 and best result was obtained at a LSR equal to 10 (Akhouri & Kumar , 2015).

When the cooking temperature and cooking time was set at 140 °C and 30min. while maintaining the liquor to straw ratio below 4:1, it is difficult for the ionic liquid, as cooking solvent, to fully infiltrate the straw and dissolve the lignin, resulting in a low pulp yield. When the ratio m (L): m(S) increases, the pulp yield first increases and then decreases. In particular, when the m (L): m(S) is 6: 1, the pulp yield reaches its maximal value. We think that the excessive ionic liquid might damage wheat straw fibres. Usually, more ionic liquid leads to more biomass dissolution due to reduction in viscosity and other factors, which

means less pulp yield. Therefore, a m(L) : m(S) ratio ranging from 9:1 to 6:1 is suitable for pulping (of wheat straw) in ionic liquid (Song, Deng, & Zhu, 2016).

2.9.3. Effects of Temperature

About 70% of the glucomannan content is lost during pulping but below 100°C the soluble glucomannan fraction is lost. Between 100°C and 130°C the peeling reactions dominate the dissolution of the remaining glucomannan content. Higher temperatures however, seems to have no or only a minor impact on the remaining content of glucomannan (Brännvall, 2004). Xylan is mostly dissolved by peeling reactions above 130°C. At lower temperatures it is only the soluble xylan that is dissolved (Maria , April 2007).

The increase of temperature of reaction from 145^{o}C to 190^{o}C has shown the decrease of Klason lignin percentage by slightly more than 50%. This is again supported by decrease of kappa number of pulp from nearly 65 to 26. The constituents of raw material get degraded in reaction; the overall yield decreases significantly (Akhouri & Kumar , 2015).

The range of cooking temperature for wheat straw in an ionic liquid with a specific liquid-solid m ((L): m(S) from 10: 1 to 4: 1) is 80 -150°C, and the cooking time varies from 10 to 40minutes. The pulp yield increases with the increase in cooking temperature, however, it begins to drop when the cooking temperature is higher than 140°C. Generally speaking, the dissolving capacity of various substances increases with the rise in temperature. Before 140°C, the amount of dissolved lignin increases, which makes beating easy, so pulp yield, is rising. After 140°C, the pulp yield begins to drop. This means that too high temperatures could damage cellulose in wheat straw pulping (Song, Deng, & Zhu, 2016).

During pulping both lignin and cellulose are dissolved at different rates. This rate is much accelerated by increasing the temperature (Iglesias et al., 1996). There was a general decrease in the pulp yield and Kappa number due to increases in the pulping time at constant temperature (Maria , April 2007).

2.9.4. Effects of Reaction Time

Reaction time increase leads to completion of reaction. Time increase up to 180 minutes results in lowering of lignin percentage in pulp to a level of 5.7% from 21% which shows a significant change. Further increase of time does not help in delignification. The change of kappa number also shows the similar trends (Akhouri & Kumar , 2015).

With the ratio m (L): m(S) 6: 1 and the cooking temperature 140°C, the pulp yield first increases with the increase in cooking time, and then levels off. The pulp yield reaches its highest value when the cooking time is 25min. Wheat straw pulping is in the quickly dissolving phase during 10-20min, and a number of lignin is dissolved out at this phase. The tendency levels off bit by bit after 25min. theoretically, with longer cooking time, the pulping reaction is more thorough and the delignification is more significant. The cooking time scope of the choice is also based on easy beating and makes pulp yield high. So, with the comprehensive consideration of the pulp yield and benefit, 20-30min is selected as the appropriate cooking time. Therefore, we draw a conclusion that the optimum conditions of the pulping process of wheat straw can be characterized by a m(L) : m(S) ratio of 8 : 1, a cooking temperature of 140°C, and a cooking time of 25min (Song, Deng, & Zhu, 2016).

2.10. Properties of Pulp and Paper

2.10.1. Basic Pulp Properties

Brightness of Pulp

Brightness of paper is discussed in Paper Properties. The paper brightness is mainly dictated by pulp brightness. There are some modifications in stock preparation which can alter paper brightness to some extent such as filler, sizing, whitening agent, dying etc.

Coarseness of Pulp

This is a measure of the average weight of fiber per unit length, often reported in units of mg/m. It is most conveniently measured using an optical analyzer. For fibers of a given average length, it is a measure of the cross sectional area of the fiber. For a given average diameter, it is measure of wall thickness. Coarse fibers are considered to be less conformable than fine fibers and do not bond as readily. Coarser fibers also result in fewer fibers per mass of pulp, which has a significant impact on sheet formation and light scattering potential.

Drainage Time of Pulp

Here the drainage time of pulp is discussed in reference to market pulp and/or unrefined pulp. The drainage time of pulp or freeness or slowness of pulp is modified to have some desired properties in the paper, here that is not discussed.

Drainage of unrefined pulp which is measured as freeness can give an indication on: 1) Fiber Length of pulp, as long fiber pulps have more freeness compared to short fiber pulps, 2)

Damage to fiber during pulping, bleaching or drying as short fibers or fines produced during pulping operation, reduces pulp freeness, 3) Refining energy required to achieve certain slowness during stock preparation. The standard procedure of measuring pulp drainage is laid out in TAPPI T221, T227, ISO 5267-1 and ISO 5267-2.

Consistency of Pulp

Consistency is the term used to describe solid content of pulp during pulp processing. For pulp and paper maker this is the most important process parameters. All equipment's are designed to handle pulp at and up to certain consistency. Pulp consistency is roughly divided in to three ranges: Low Consistency: <5%, Medium Consistency: 5-15% and High Consistency: >15%.

It is the desire of every pulp maker to keep pulp at the highest possible consistency to minimize dilution water usage and which ends up as effluent. Higher consistency also helps in reducing the bleaching chemical consumption. But there are practical limitations of handling pulp at higher consistency such as high viscosity which make pulp flow very difficult. The standard procedure of measuring pulp consistency (up to 25%) is laid out in TAPPI T240.

Moisture Content of Pulp

It is important from storage, transportation and handling point of view. Most of the market pulp are sold, stored, transported and used as air dry. The useable part of pulp is dry fiber only, so the tendency is to minimize the moisture content op pulp. Small quantity of pulp is sold as wet lap also. Wet lap pulp is not dried at source and transported at about 50% moisture content. It is feasible for short distance transportation and if pulp is to be used immediately at user end.

Extractives in Pulp

The low molecular carbohydrates indicate an extent of cellulose degradation during pulping and bleaching process, which may affect pulp strength and other properties. Pulp is treated with 1% hot NaOH solution for one hour to estimate loss of yield due to extractives. The standard procedure of measuring 1% Hot Alkali Solubility is laid out in TAPPI T212.

Fiber Diameter and Length of Pulp

The effect of fiber diameter, wall thickness and coarseness on sheet properties is rather complex and not clearly established. These qualities primarily affect fiber flexibility. Fiber

diameter may be expressed mean cross section or ratio of wall thickness to diameter, sometime termed as fiber density.

Length of fibers (arithmetic average, weighted average etc.) is one of the most important parameters of pulp. Pulp strength is directly proportional to fiber length and dictates its final use. A long fiber pulp is good to blend with short fiber pulp to optimize on fiber cost, strength and formation of paper. Softwood with pulps in general has longer fiber compared to hard wood pulp. Pulp made from woods grown in cold climate in general has longer fiber compared to wood grown in warmer climates.

Chemical pulps in general have higher fiber length compared to semi chemical pulp and mechanical pulp, when made from same wood. More fibers get damaged/shorten by mechanical action than chemical action.

There are several methods to measure /report fiber length of pulp. The 'fiber length of pulp by projection' is described in TAPPI T232. The 'fiber length of pulp by classification' is described in TAPPI T233. "Fiber length of pulp and paper by automated optical analyzer using polarized light' is described in TAPPI T271.

Hemicelluloses in Pulp

Pulps differ in their content of hemicelluloses and in the chemical composition of their hemicelluloses. Hemicellulose content of a pulp is an indicator of chemical differences originating with the tree and is affected by the pulping process used. Pulp with higher Hemicellulose content develops strength faster upon beating/refining. Hemicellulose helps cellulose fibers bonding. Softwood hemicellulose is much more effective in fiber bonding than hardwood hemicellulose.

Kappa Numbers of a Pulp

The Kappa number is an indication of the residual lignin content in a sample of pulp or it is the bleachability of a pulp by a standardized analysis method. Lignin is one of the three polymers in wood and the one that has to be removed in the chemical pulping process to liberate the fibers that are used in cellulosic products, such as paper and cardboard. Kappa number measures the volume (in milliliters) of 0.1N potassium permanganate solution consumed by one gram of moisture free pulp under the conditions specified in (TAPPI T236 cm-85) test method. The results were corrected to 50% consumption of the permanganate

added. Low Kappa numbers require less $KMnO_4$ for sample analysis, and the pulp is lighter in color.

Kappa number of the pulp is one of the important parameters in pulp manufacturing because it relates to the bleach ability or degree of delignification of the pulp. The pulp samples are analyzed at various points in the paper making process and different Kappa numbers are expected at each point in the process, which is dependent on the amount of lignin that has been removed from the sample during the pulping process. As lignin is removed from the sample, the Kappa number is reduced. Low Kappa pulps are easier to bleach but high kappa pulps usually require more energy in refining, and often produce stronger paper or board (particularly with regard to tear strength). A fully bleached pulp can have Kappa number as low as 1 and very high yield pulp may have kappa number as high as 100.

Pulp Yield

Pulp yield is the main concern of pulp milling industry and it is mainly govern by the pulping processes. The pulp ideally needs to be pure fiber. That is the reason to remove lignin and solvent extractives. When lignin was removed from cooked chips, fibers were obtained for paper making and called pulp.

Among all the pulping process, mechanical pulping provides high pulp yield, which retains almost all constituents of wood including lignin. Lignin which is second highest to cellulose, does not bond to itself or cellulose as fibers do. Therefore pulps with high yield results in weak pulp. In addition to this lignin is brown in color and to maintain high yield of bleached pulp, lignin is not removed during bleaching, but only chemically modified.

Viscosity of Pulp

Solution viscosity of a pulp gives an estimation of the average degree of polymerization of the cellulose fiber. So the viscosity indicates the relative degradation of cellulose fiber during pulping /bleaching process.

2.10.2. Physical Properties of Paper

Basis Weight or Grammage

The basis weight, substance or Grammage is obviously most fundamental property of paper and paperboard. The Basis weight of paper is the weight per unit area. This can be expressed as the weight in grams per square meter (GSM or g/m^2), pounds per 1000 sq. ft. or weight in

Kgs or pounds per ream (500 sheets) of a specific size. Paper is sold by weight but the buyer is interested in area of paper. The basis weight is what determines, how much area the buyer gets for a given weight. Paper maker always strive to get all desired properties of paper with minimum possible basis weight.

The area of several sheets of the paper or paperboard is determined from linear measurements and the mass (commonly called "weight") is determined by weighing. The Grammage is calculated from the ratio of the mass to the area after conversion to metric units when necessary. Grammage of up to 200gsm are considered to be papers and from 200gsm upwards they are referred to as paperboard or low quality board. Paper that is used in offices is usually between 70gsm and 80gsm, with 80gsm being the most commonly found weight. Some accountants and solicitors use heavier weight paper ranging from 90gsm to 120gsm for formal correspondence. Above 120gsm come various thicknesses of card with 160gsm and 200gsm being most commonly used for file dividers. Newspaper sheet ranges between 45gsm and 50gsm (ASTM, 2001).

All paper machines are designed to manufacture paper in a given basis weight range. Tighter the range, more efficient will be the machine operation. The standard procedure of measuring basis weight is laid out in TAPPI T 410, SCAN P6, and DIN53104 & ISO: BSENISO536.

Table 13 Shows the typical Grammage Values of standard paper sheet.

Grade	**g/m^2**
Cigarette Tissue	22 - 25
Newsprint	40 - 50
Bond	60 -90
Paperboard	120 - 300

Caliper or Thickness

For a given basis weight, thickness determines how bulky or dense paper is. A well beaten or refined pulp, short fiber pulp such as hard wood or straw pulp, highly filled or loaded paper will show lower thickness for given basis weight. Thickness or Caliper of paper is measured with a micrometer as the perpendicular distance between two circular plane parallel surfaces under a pressure of 1 kg / cm^2. Uniform caliper is good for good roll building and subsequent printing. Variations in caliper can affect several basic properties including strength, optical

and roll quality. Thickness is important in filling cards, printing papers, condenser paper, saturating papers etc.

Curl

Paper curl can be defined as a systematic deviation of a sheet from a flat form. It results from the release of stresses that are introduced into the sheet during manufacture and subsequent use. Paper curl has been a persistent quality issue and is increasingly important for paper grades being subjected to high speed printing, xerography and high precision converting processes.

There are three basic types of curl, mechanical curl, structural curl and moisture curl. Mechanical curl develops when one side of the paper is stretched beyond its elastic limits. One example of this is the curl in the sheet which forms near the center of a roll. Structural curl is caused by two sidedness in the sheet that is a difference in the level of fines, fillers, fiber area density or fiber orientation through the sheet thickness. Moisture curl can develop when the paper sheet is being offset printed. One side of the sheet may pick up more moisture than the other, the higher moisture side releases the built in drying strains and the paper will curl towards the drier side.

Dimensional Stability

Cellulose fibers (main constituent of paper) swell in diameter from 15 to 20% from dry condition to saturation point. Since most of the fiber in paper sheet are aligned in the machine run direction, absorption and de-absorption of moisture by paper causes the change in CD dimension. Such changes in dimension may seriously affect register in printing processes and interfere with the use of such items as tabulating cards. Uneven dimensional changes cause undesirable cockling and curling. Dimensional changes in paper originate in the swelling and contraction of the individual fibers. It is impossible to be precise about the degree of this swelling because paper making fibers differ considerably in this property, and because the irregular cross section of fibers creates difficulty in defining diameter. Change that occurs in the dimensions of paper with variation in the moisture content is an important consideration in the use of paper. All papers expand with increased moisture content and contract with decreased moisture content, but the rate and extent of changes vary with different papers. Dimensional stability of paper can be improved by avoiding fiber to absorb moisture. Well sized papers have better dimensional stability.

Formation

Formation is an indicator of how uniformly the fibers and fillers are distributed in the sheet. Formation plays an important role as most of the paper properties depend on it. A paper is as strong as its weakest point. A poorly formed sheet will have more weak and thin or thick spots. These will affect properties like caliper, opacity, strength etc. Paper formation also affects the coating capabilities and printing characteristics of the paper. A poorly formed sheet will exhibit more dot gain and a mottled appearance when printed.

There is no standard method or unit to express formation. It is a relative or subjective evaluation. However when holding paper up to a light source, a well formed sheet appears uniform while a poorly formed sheet has clumps of fibers giving a cloudy look.

Smoothness

It is most important parameter for printer. Smoothness is concerned with the surface contour of paper. It is the flatness of the surface under testing conditions which considers roughness, levelness, and compressibility. In most of the uses of paper, the character of the surface is of great importance. It is common to say that paper has a "smooth" or a "rough" texture. The terms "finish" and "pattern" are frequently used in describing the contour or appearance of paper surfaces. Smoothness is important for writing, where it affects the ease of travel of the pen over the paper surface. Finish is important in bag paper as it is related to the tendency of the bag to slide when stacked. Smoothness of the paper will often determine whether or not it can be successfully printed. Smoothness also gives eye appeal as a rough paper is unattractive.

Optical Properties (Brightness, Whiteness and Colour)

Brightness is defined as the percentage reflectance of blue light only at a wavelength of 457nm. Whiteness refers to the extent that paper diffusely reflects light of all wave lengths throughout the visible spectrum. Whiteness is an appearance term. Color is an aesthetic value and it may appear different when viewed under a different light source. Brightness is the most important measure of quality for many types of pulp. Bleaching is associated with high cost and control of brightness will reduce the cost of bleaching chemicals. The brightness parameter is used to monitor the bleaching process. Brightness together with fluorescence is used to supervise the dosage of OBA (optical brighteners) and it is the most commonly used

parameter in the sales specifications of paper and paperboard products (Lorentzen & Wettre, 2016).

Brightness is measured with two different standards TAPPI/GE and ISO. Though there is correlation, ISO brightness of a sample is usually lower by 1-1.5 units over GE brightness. The standards are as per TAPPI T 452. The procedural standards for the measurement of whiteness are explained in ISO 11475.

Bursting Strength

Bursting strength is the capacity of a material (such as a paper or textile) or object (such as a metal pipe) to maintain in continuity when subjected to pressure; broadly the pressure often expressed in pounds per square inch required to rupture such a material or object under rigidly controlled conditions. It is a pressure measured at which a pulp sheet will burst, used as a measure of resistance to rupture. TAPPI method T-403 is the official test used for measuring the bursting strength of papers pressure applied through a rubber diaphragm on a sample with thicknesses up to 0.6 mm and diameter 1.20 inch (30.5mm) (Anon, 1987). Burst strength depends largely on the tensile strength of extensibility of the material/pulp sheet/ and it is also commonly known as the Mullen test (http:/papersizes.org). The standards procedure is described in TAPPI T 403.

Table 14 Typical Brightness Values of ISO and TAPPI

Typical Brightness Values		Brightness Quality Levels of American Forest & Paper Association (AFPA)	
Grade	% ISO	Level	% TAPPI
Newsprint	62-65	Premium	88.0 & above
Fully Bleached Pulp	90	No. 1	85.0 - 87.9
Office/Business Paper	80-95	No. 2	83.0 - 84.9
Bond	70-92	No. 3	79.0 - 82.9
Coated Paper	85-90	No. 4	73.0 - 78.9
		No. 5	72.9 & Below

(Lorentzen & Wettre, 2016).

Tensile Strength

Tensile strength of paper is the maximum strength of randomly oriented pulp fiber when formed in a sheet. It is the force required to produce a rupture in a strip of paper or paperboard, measured in MD and CD, expressed in KN/m. This tensile strength gives an indication of the maximum possible strength of pulp beaten under ideal condition. In addition to this; tensile strength is an indicative of fiber strength, fiber bonding and fiber length. Tensile strength can be used as a potential indicator of resistance to web breaking during printing or converting.

Tensile strength can be described by stress-strain graphs and measured by TAPPI tests T-404 and T-494. Stress-strain curves provide a fundamental engineering description of the mechanical behavior of paper when subjected to tensile stress. TAPPI method T-404 measures tensile breaking strength and elongation of paper and paperboard using a pendulum type tester, and T-494 measures tensile breaking properties of paper and paperboard using constant rate of elongation apparatus (Caulfield. & Gunderson., 1988).

Tearing Resistance

Tearing resistance or strengths is a measure of how well a material can withstand the effects of tearing. More specifically it is how well a paper resists or withstands the growth of any cuts when under tension.

Tearing resistance indicates the behavior of paper in various end use situations; such as evaluating web run ability, controlling the quality of newsprint and characterizing the toughness of packaging papers where the ability to absorb shocks is essential. Fiber length and inter-fiber bonding are both important factors in tearing strength. It is measured in both MD and CD and expressed in mN (milinewton).

The most commonly used tearing test is T-414, which is often called the Elmendorf tear test, and measures the internal tearing resistance of paper rather than the edge tear strength of paper, which is described in T-470 (Anon, 1987). Internal tearing resistance is a measure of the force perpendicular to the plane of the paper necessary to tear a single sheet through a specified distance after the tear has already been started. Edge tearing strength (T-470) is a measure of the force needed to initiate a tear. The force needed to initiate a tear may be several times the force needed to propagate the tear once it is started. This is commonly known to anyone who has experienced the difficulty of opening a cellophane bag, which, once nicked, tears open easily. Those papers and other film materials that exhibit high tensile stretch or elongation to break also exhibit high edge tearing strength (Caulfield. &

Gunderson., 1988). High stretch makes it difficult to localize or concentrate stress in a sufficiently small area so that a tear can be initiated.

The table below contains list of various types of paper, based on their basis weight, based on their end use, process of manufacturing and raw materials used.

Table 15 The major classifications of paper sheets

Based on basis weight	**Based on Color**
Tissue: Low weight, <40 g/m^2 Paper: Medium weight, 40 - 120 g/m^2 Paperboard: Medium High weight, 120-200 g/m^2 Board: High weight, >200 g/m^2	Brown: Unbleached White: Bleached Colored: Bleached and dyed or pigmented
Based on Usage	**Based on Raw Material**
Industrial: Packaging, wrapping, filtering, electrical etc. Cultural: Writing, printing, Newspaper, currency etc. Food: Food wrapping, candy wrapping Coffee filter, tea bag etc.	Wood: Contain fibers from wood Agricultural residue: Fibers from straw, grass or other annual plants Recycled: Recycle or secondary Fiber

(Lorentzen & Wettre, 2016).

3. MATERIALS AND METHODS

3.1. Materials and Equipment

The main raw material that has been used during the experimental works was wheat straw which is one of the most important agricultural residues.

Equipment's used to conduct this study were: Razor blade, Autoclave for pulp cooking, washing Bucket and Vat, Poly Ethylene Bag, Strip Road, Silica Crucible, Muffled Furnace, Desiccators, Digital weighing Balance, weighing bottle, Stopper, Drying oven, Tong, special dryer Towel, Bunsen burner, Erlenmeyer flask, Water bath, Heating mantle, Vacuum Suction, Weighed Sintered Glass Crucible, Watt-man Filter paper, Measuring Cylinder, rounded Bottom Flask, Soxhlet apparatus, cellulose extraction Thimble, Cotton, Jar, Scissor, Glove, Mask and Goggles, volumetric flask, Stopwatch, 250µm-1mm size mesh sieve, Glass or porcelain, Pipettes, Burette, Reaction beaker, Spoon and magnetic stirrer, standard pulp beater machine equipped with motor, Disintegrator, standard 6 1/2 inch diameter sheet forming machine with stirrer, standard couch roll, couch plate, pump and press with pressure gage, suction template of sheet former, disks, drying rings, blotting paper, cup, freeness tester etc.

The chemicals and reagents used in this study were: Analytical grade caustic soda (NaOH), sodium sulfide (Na_2S), distilled water, Potassium permanganate solution standardized 0.1000±0.0005N $KMnO_4$, Sodium thiosulfate solution, approximately 0.2N $Na_2S_2O_3.5H_2O$, Potassium iodide solution 1.0N KI, Sulfuric acid 4.0N H_2SO4, Ethyl alcohol, Hexane, Toluene, Nitric acid, acetic acid, starch indicator solution 0.2%, hydrogen peroxide 3% (H_2O_2), potassium Hypochlorite 12.5% etc.

3.2. Raw Materials Collection and Preparation

The wheat straw sample used for this study was harvested from Amhara region, Eastern Gojjam zone, Gindewoin woreda, since wheat is the popular cereal crops in the specified area. The collected wheat straw was then air dried for a week and cut into small strips with scissor to a length of about 10-15cm. Then the resulting chips were placed in plastic bags for further processing.

(a) **(b)** **(c)**

Figure 7 Wheat straw while harvesting (a) after dried (b) and size reduced (c)

3.3. Wheat straw characterization

3.3.1. Morphological Characteristics

In order to get representative results of morphological studies three randomly selected samples of wheat straw were taken from a storage bag. For fiber length determination, small slivers were taken, macerated with 10ml of HNO_3, 67% and boiled in a water bath (100±2) ^oC for 10 min (Ogbonnaya et al.1997). The slivers were then washed, placed in small flasks, with 50ml of distilled water and the fibre bundles were separated into individual fibers using a small mixer with a plastic end to avoid fibre breaking. The macerated fibers suspension was finally placed on a slide (standard 7.5 * 2.5cm) by means of dropper (Hans et al.1999). For fiber diameter, lumen width and cell wall thickness determination, cross-sections were cut on Lietz base sladge microtome 1300. This cross-section were stained with 1:1 aniline sulphate-glycerin mixture to enhance cell wall visibility (cell walls retain a characteristic yellowish colour).

All fiber samples were viewed under a calibrated motic electron microscope; a total of 75 randomly chosen fibers were measured. Three derived values were also calculated using fiber dimensions: slenderness ratio as fiber length /fiber diameter, flexibility coefficient as fiber lumen width / fiber diameter) *100 and Runkel ratio as (2* cell wall thickness /lumen width).the results were compared with bamboo (Shalini, Dharm, & Tyagi, 2011) and Cotton stalk (Gedefaw, 2015) as per TAPPI standard test methods.

3.3.2. Proximate analysis

Moisture Content Determination

The chipped wheat straw was made free of moisture in an oven at 105±3^{o}C overnight prior to pulping since the moisture content largely affects the pulp yield. Then the moisture content was determined by using the following formula at every two hours interval until constant weight was obtained.

$$Moisture\ content\ \% = \frac{W_1 - W_2}{W_1} * 100 \quad (3.1)$$

Where,

W_1 = mass of chipped wheat straw before drying

W_2 = mass of chipped wheat straw after drying

Ash Content Determination

Silica crucible was dried over a burner for 10min, cooled in a desiccators containing silica gel up to room temperature. About 0.5g of moisture free sample was taken and added to the dried crucible and weighed as (W_1). Then the crucibles containing samples was ignited in a muffle furnace at 525±25^{o}C for 4hr. Ascertain all the carbon particles were removed by blocking air from the formation of black char particles, and then crucibles containing the sample was removed from furnace and cooled in the desiccators to room temperature and weighed as (W_2).

$$As\ Content\ (\%) = \frac{W_2}{W_1} * 100 \quad (3.2)$$

Where

W_1 – weight of moisture free sample + mass of dried crucible before ignition, g

W_2 – weight of sample + mass of dried crucible after ignition, g

Solvent extractives

Three grams oven dried sample was placed into a cellulose extraction thimble. The thimble was plugged with cotton and placed in a soxhlet extraction tube. The boiling flasks containing solvent mixture in the ratio 2:1 of 95% ethyl alcohol and distilled toluene respectively were placed on a heating mantle. The extraction was conducted for eight hours at the rate of approximately six siphoning per hour as shown in figure 8 below.

When the extraction was completed, all of the remaining solvents in the soxhlet were transferred to the boiling flask and evaporated in a rotary evaporator. The flasks were oven-dried at 105±3^{o}C, cooled in desiccators, and weighed until a constant weight was obtained.

Figure 8 Extractive components determination

The following formula was used to obtain the alcohol-toluene solubility content of wheat straw:

$$Extractives\ (\%) = \frac{(M_3 - M_2)}{M_1} * 100 \quad (3.3)$$

Where:

M_1 – oven dry weight of sample, g

M_2 – oven dry weight of flask, g

M_3 – oven dry weight of extract and flask, g

3.3.3. Chemical Compositions

Chemical composition of wheat straw was determined by applying standard isolation methods for major plant chemical components. Organic (cellulose, lignin, hemicelluloses and solvent extractives) and inorganic compounds (ash) were determined according to TAPPI (Technical Association of Pulp and Paper Industries) standards.

Klason lignin

One gram, oven dried sample of extractive free straw was placed in a 150ml beaker and 15ml of cold sulfuric acid (72%) was added slowly while stirring and mixing well. The reaction proceeded for two hours with frequent stirring in a water bath maintained at 20^{o}C. At the end of second hour, the specimen was transferred by washing it with 560ml of distilled water into a 1000ml flask, diluting the concentration of the sulfuric acid to 3%. Allihn condenser was attached to the flask and refluxed in a boiling water bath for four hours. The flasks were then removed from the water bath and the insoluble material was allowed for overnight to settle. The contents of the flasks were filtered by vacuum suction into a fritted-glass crucible of known weight.

The residue was washed free of acid with 500mL of hot tap water and then oven dried at 105±3^{o}C. Crucibles were then cooled in desiccators and weighed until a constant weight will be obtained. The following formula was used to obtain the lignin content of wheat straw:

$$Klason\ Lignin(\%) = \frac{(M_2 - M_1)}{M_s} * 100 \quad (3.4)$$

Where:

M_1 – oven dry weight of filter paper, g

M_2 – oven dry weight of filtrated lignin + weight of filter paper, g

M_s – oven dry weight of sample, g

Cellulose

One gram of extract free wheat straw sample was refluxed with three successive portions of a mixture of concentrated nitric acid (20% volume by volume) in ethyl alcohol for 1hr each, filtered, washed, dried, and weighed the white fiber residue and calculated as followed.

$$Cellulose(\%) = \frac{M_2 - M_1}{M_s} * 100 \quad (3.5)$$

Where:

M_1 – oven dry weight of filter paper funnel, g

M_2 – oven dry weight of funnel + extracted cellulose, g

M_u – oven dry weight of sample, g

Hemicelluloses

Two and half grams oven dried extractive free sample was weighted as W_1, and placed in a flask, the solution of one gram $NaClO_2$, 80ml distilled water and 0.5ml glacial acetic acid was added and well stirred for 1hr. Then the second solution of one gram $NaClO_2$, 80ml distilled water and 0.5ml glacial acetic acid was added. After heating for 3hr in the water bath at a temperature of 75^{o}C, the solution was transferred to filtering crucible and filtered, washed with 100ml of 1% aqueous glacial acetic acid and then washed with 10ml acetone. Filtering crucible containing the holocellulose was oven dried at 105±3^{o}C and placed in a desiccator to cool to room temperature. Weighed until the variation between two successive measurements were not greater than 0.005 and record as W_2. Finally, the holocellulose content was calculated as follow

$$Holocelulose\ \% = \frac{W_2}{W_1} * 100 \quad (3.6)$$

Where, W_1 is the oven dried extractive free sample and

W_2 is the weight of oven dried holocellulose

After getting the percentage holocellulose content, hemicellulose content was calculated by subtracting cellulose content from holocellulose content, because holocellulose contains both cellulose and hemicellulose.

$$Hemicellulose\ content, \% = Holocellulose, \% - Cellulose, \% \quad (3.7)$$

3.4. Kraft Pulping of wheat straw

3.4.1. Experimental set-up

Once the wheat straw material was prepared, the oven dried wheat straw then pulped using Kraft method, by varying the cooking parameters (active alkali charges, cooking time and cooking temperature) as per the design and the Liquor to straw ratio was kept to 9:1. Then after each cook, 30minutes gas down period was allowed before the black liquor ejection. Each batch of produced pulps was then disintegrated and washed with cold water on a standard size 250µm netted sieve. Then after washing and screening the produced pulp was dried in an oven. The pulping conditions of wheat straw used during the study are given in table 16.

Table 16 Pulping conditions of wheat straw

Parameters	**Specification**
Liquor to wheat straw ratio	9:1
Sulphidity	20%
Operating Pressure	2, 3 and 4 bar according to the temperature.
NaOH	10, 15 and 20%
Cooking Temperature	130, 140 and 150^{o}C
Cooking Time	30, 60 and 90 min.

(a) **(b)**

Figure 9 wheat straw before cooking (a) after cooking (b)

3.4.2. Pulp Yield Determination

One of the objectives of this study was to produce pulp and converting it to paper sheet. So the pulp ideally needs to be pure fiber. That is the reason to remove lignin and solvent extractives. When lignin was removed from cooked chips fibers were obtained for paper making and called pulp. Since wheat straws are made of many chemical components like other woody species, it is unthinkable to get 100% pulp yield. We expect the loss of pulp yield due to cellulose degradation and fiber loss with rejects.

Pulp yield which was expressed as the ratio of moisture free weight of screened pulp from digester with oven dry weight of the chips feed for pulping was determined by the method as per TAPPI (Technical Association of Pulp and Paper Industries) standards.

$$Pulp\ yield(\%) = \frac{\text{weight of oven dried pulp}}{\text{weight of oven dried wheat straw}} * 100 \qquad (3.8)$$

(a) **(b)** **(c)**

Figure 10 Pulp from digester (a), after washing (b), after drying in an oven(c)

3.4.2. Kappa Number Determination

In determining kappa numbers of the pulp three chemical reactions were expected to occur. The first reaction that takes place during the determination of Kappa number occurs between the lignin, the permanganate, and the acid. The lignin is oxidized and solubilized by permanganate in the presence of the acid and the excess permanganate is used in the second reaction. The second reaction occurs when the potassium iodide is added and reacts with the excess permanganate to produce iodine. The third reaction occurs when the free iodine is titrated with sodium theiosulphate to produce iodide and Sulphate anions.

$$\text{Lignin} + MnO_4^- + H^+ \longrightarrow \text{Oxidized Lignin} + H^+ + MnO_4^- \text{ (excess)}$$

$$2\,MnO_4^- + 10I^- + 16H^+ \longrightarrow 2Mn^{2+} + 5I_2 + 8H_2O$$

$$I_2 + 2S_2O_3^{2-} \longrightarrow 2I^- + S_4O_6^{2-}$$

The method calculates the volume of 0.02mol/L (0.1N) potassium permanganate consumed by one gram of moisture free pulp by the difference between the initial and the final excess volumes of potassium permanganate after a 10 minute reaction at 25^{o}C under weak acidic conditions. The final excess volume of potassium permanganate after the 10 minute oxidation reactions is determined by titrimetric using a standard theiosulphate solution after adding an excess of potassium iodide to the slurry to react with the excess permanganate to form iodine as indicated in annex part of PD 1(TAPPI T 236 cm-85).

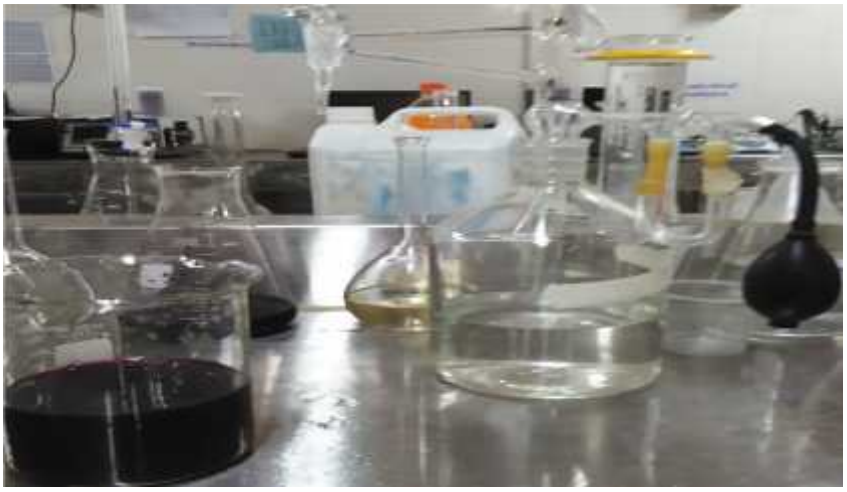


Figure 11 Kappa number determination of pulp using titration method

The Kappa number K, expressed as a numerical value only, is given by the formula:

$$V_a = \frac{V_1 - V_2\ C}{0.1} \quad (3.9)$$

And

$$K = \frac{V_a * d}{M} \quad (3.10)$$

Where:

V_a is the volume of the potassium permanganate solution consumed in the determination, in milliliters;

K = kappa number

V_1 is the volume of the sodium theiosulphate solution consumed in the blank test, in milliliters;

V_2 is the volume of the sodium theiosulphate solution consumed in the determination, in milliliters;

C is the concentration of the sodium theiosulphate solution, expressed in moles per liter;

0.1 is the numerical factor, which takes into account the molarities of potassium permanganate and the stoichiometry of the reaction during the titration (f = 0. 02* 5);

d is the correction factor to 50% (mass/mass) permanganate consumption; d is dependent on the value of Va, (Table 17);

M is the oven dried mass of the test specimen, in grams.

Table 17 Factors f to correct for different percentages of permanganate used

Va	**d**									
	0	1	2	3	4	5	6	7	8	9
10	0,938	0,942	0,946	0,950	0,954	0,958	0,962	0,966	0,970	0,975
20	0,979	0,983	0,987	0,991	0,996	1,000	1,004	1,009	1,013	1,017
30	1.022	1.024	1.026	1.028	1.030	1.033	1.035	1.037	1.039	1.042
40	1.044									

(Agency E. S., 2012)

The correction factor *d is* based on an experimental study which gave the following formula:

$$\log_{10} K = \log_{10} \frac{V_a}{M} + 0.00093\ 2V_a - 50 \quad (3.11)$$

Since the correction factor d, is not exist for some values of Va (the actual consumption of permanganate solution in the determination) equation (3.11) has been used to calculate the kappa numbers of samples.

The percentages of remaining lignin content in the pulp samples were determined by the equation (Agency E. S., 2012):

$$\text{Lignin } \% \cong \text{Kappa number} * 0.15 \quad (3.12)$$

3.4.3. Pulp Bleaching

The pulp should be bleached as lignin fragments contain conjugated chromophores that remain after chemical (Kraft) pulping. The conjugation needs to be destroyed by oxidation using bleach, so bleaching destroys residual lignin to reduce yellowing. Traditionally, Cl_2 has been used as bleaching agent but later replaced by ClO_2 for Elemental Chlorine Free process. Based on its reduction to Cl^-, ClO_2 is 2.5 times more oxidizing agent than Cl_2 per unit mass.

After the addition of bleaching agent, NaOH should be added to solubilize the oxidized lignin fragments and partially removed 'organic' chlorine and this process should be repeated for certain sequences. This process is 5-10 times less chlorinated by products than Cl_2, but this process is expensive and highly selective oxidant for lignin. Furthermore, there is another bleaching agent which is called 'totally Chlorine Free' process which uses $O_2/O_3/H_2O_2$.

But for this study a two stage bleaching was used: first the pulp was treated with 12.5% Hypochlorite solution with 1:10 ratio of dry weight pulp to bleaching solution for an hour at 70^{o}C and then treated with NaOH, 4% extraction by maintaining the PH 9 to 11, secondly the pulp was treated with 3% H_2O_2 at 70^{o}C for one hour and again treated with NaOH 4% to remove the residual lignin.

Table 18 The pulping and bleaching conditions after numerical optimizations has been performed.

Parameters	Pulping condition	Bleaching condition
NaOH	10%	4%
Na_2S	20%	-
H_2O_2	-	3%
$Ca(ClO)_2$	-	12.5%
Temperature	136.9^{o}C	70^{o}C
Time	30min	60min
Liquor to straw/ solvent to pulp ratio	9 :1	10 :1

(a) **(b)** **(c)**

Figure 12 Produced pulp before (a) while bleaching (b) and after bleaching(c)

3.5. Paper Hand Sheet Preparation and Characterization

3.5.1. Hand Sheet Preparation

Dry pulp produced from optimized pulping condition was used for sheet preparation. 400g of oven dry pulp was mixed with 23L water and pulp slurry with consistency of 1.7% was made. The prepared pulp slurry was then added to the beating machine. Freeness of slurry was checked out at each ten minutes beating interval. Freeness was checked by taking 130ml of slurry from the beater (this contains 2g of moisture free fiber) and diluted to 1000ml with distilled water and measured freeness value. When the freeness of pulp was 30CSF (Canadian standard of freeness), 1200ml of stock was taken from beater and diluted to 2000ml water (0.62% consistency) with 800ml wash. Then it was placed to the disintegrator with 500rpm for five minutes. Then the blade of the disintegrator was washed with 250ml water and transferred to plastic vat and diluted with 2000ml water to a total volume of 6250ml and agitated well by hand. Materials from the diluted suspension was then taken and spread over the required paper sheet forming machine. Once the sheets were prepared two stage pressing was followed by applying 0.4MPa pressure for two minutes by pressing machine. Then the stocks were removed from the press and attached to the drying plates in order to dry by oven at 130^{o}C for 45minutes.

(a) **(b)**

Figure 13 Pulp beating machine while beating (a), sheet forming and pressing machine (b)

3.5.2. Paper Hand Sheet Test

The prepared sheets from both bleached and unbleached wheat straw pulp were then tested for the following properties:

Figure 14 Paper sheet from bleached and unbleached pulp left to right

Grammage

Grammage is a French term used to express mass per unit area of a paper. The SI metric units, in which Grammage (mass per unit area) is expressed in grams per square meter (g/m^2).

After the test specimen has been prepared using cutting device of size 10cm by 10cm to accuracy of 0.2% and weighed. Then Grammage of paper sheet was calculated as

$$Grammage = \frac{\text{weight (g)}}{\text{Area } (m^2)} \qquad (3.13)$$

Tensile Properties

Tensile Strength

This tensile strength gives an indication of the maximum possible strength of pulp or paper beaten under an ideal condition.

After the sheet specimens were cut into 15±0.1mm wide by 230mm long test pieces, it was then placed in the clamps of L-W AB Lorentz &Wetter tensile strength tester by making sure that any slacks were so eliminated. Any touch of the test area between the clamps with the fingers was avoided. All readings were then recorded except for test pieces that broke with in 2mm of clamming line (T 494 om-01).

Tensile strength

$$X_1 = \frac{a}{b} \tag{3.14}$$

Where,

X_1 = tensile strength (KN/m)

a = maximum tensile force in (N) = instrument reading in kg to change in to (N) = kg * 9.807

b = initial width of the sample in (mm)

Tensile index

$$X_2 = 1000 * \frac{X_1}{W} \tag{3.15}$$

Where,

X_2 = tensile index (Nm/g)

X_1 = tensile strength (KN/m)

W = mean Grammage in (g/m^2)

Breaking Length

$$X_3 = \frac{a * 102{,}000}{b} \tag{3.16}$$

Where

a = mean tensile strength in, KN/m

b = Grammage in, g/m^2

X_3 = breaking length in meter, m

Burst Properties

Bursting strength

It is the capacity of a material (such as a paper or textile) or object (such as a metal pipe) to maintain in continuity when subjected to pressure; broadly the pressure often expressed in pounds per square inch required to rupture such a material or object under rigidly controlled conditions.

The test specimens was first prepared as 10 cm by 10 cm sheet and clamped in the tester tightly with Bursting strength tester of model PN-BSM600 and the maximum reading pointer was set to zero position. The pump motor was then started and the pumping system and the test piece then wait to burst. The maximum read pointer was then recorded and allowed to rest gently to zero position and the broken sample was removed. Instrument reading = Kg/cm^2

To change Kg/cm^2 in to Kpa (Kilo Pascal), Kpa = Kg/cm^2 * 98.07
Where, Kpa -SI unit of bursting strength
Kg/cm^2 - instrument reading.

Burst factor

This factor, which has been called the bursting area, is equivalent to the number of square meter of paper, the weight of which, if applied to each square centimeter of the test sheet camped in the instrument cause a burst.

$$\text{Burst factor} = \frac{\text{burst strength } \frac{kg}{cm^2} * 1000}{\text{basis weight}(\frac{g}{cm^2})} \quad (3.17)$$

Burst Index

Burst index is bursting strength divided by basis weight.

$$X = \frac{a}{W} \quad (3.18)$$

Where

x- Burst index Kpa m^2/kg a- Burst strength in Kpa w- Basis weight in g/m^2

Tearing Properties

Tearing resistance

Tear resistance is a measure of how well a material can withstand the effects of tearing. More specifically however it is how well a material resists the growth of any cuts when under tension using L-W AB Lorentz & Wetter tearing tester.

The pendulum was raised to its initial position and 4 pieces of specimens (65*7.5cm) was clamped. Silt in the specimens was then made by completely pressed down the knife rocker arm. The pendulum was then stopped by quickly pressing down the releaser and the pendulum then broken softly after oscillation to the right. The scare value then recorded. The average of the reading was then calculated and multiplied with the factor of the pendulum. So the product would be equal to the tearing strength in milinewton (mN).

Tearing Resistance

$$a = P * S \tag{3.19}$$

Where

a = tearing resistance in mN.

S= the average resistance reading from the instrument

P = 4, the factor of the pendulum i.e. the factor which is used for transferring the reading on the scale to the tearing resistance in mN.

N.B:- 1 sheet (specimen) = multiply 16(P=16)

2 sheet (specimen) = multiply 8(P=8)

4 sheet (specimen) = multiply 4(P=4)

$$Tear\ factor = \frac{Tear\ resistance}{Basis\ weig?t(\frac{g}{m^2})}\ mN \tag{3.20}$$

$$Tear\ index = \frac{Tear\ factor}{10.2} \tag{2.21}$$

3.6. Experimental Design

The first task before conducting experiments was the selection of potential parameters that needs to be varied. Therefore the three proposed main factors selected for this study were the digestion temperature, concentration of NaOH charge and digestion (reaction) time. The levels of the selected factors were determined from different previous researches as discussed above in the literature review part, under sub titles of factors affect the Kraft pulping processes. The experiment performed was completely randomized design with three main factors at three levels and two response variables. The two response variables were the pulp yield and kappa number.

Data analysis was done by DESIGN EXPERT® 6.0.8 software using Response Surface Design, 3-level factorial method and randomizes the runs. Randomization ensures that the

conditions in one run neither depend on the conditions of the previous runs nor predict the conditions in the subsequent runs.

Table 19 Level and code of variables used for Response Surface Design

Factors	Unit	Code	Levels		
			-1	0	+1
Temperature	^{o}C	A	130	140	150
NaOH Concentration	%	B	10	15	20
Time	min	C	30	60	90

Table 20 The proposed response surface design with 3-level factorial method results 32 runs

Std.	Run	Block	Factor 1 A: temp. ^{o}C	Factor 2 B: Conc. NaOH %	Factor 3 C: Time min	Response 1: Yield %	Response 2: Kappa num.
1	7	Block 1	130.00	10.00	30.00		
2	4	Block 1	140.00	10.00	30.00		
3	1	Block 1	150.00	10.00	30.00		
4	29	Block 1	130.00	15.00	30.00		
5	13	Block 1	140.00	15.00	30.00		
6	8	Block 1	150.00	15.00	30.00		
7	22	Block 1	130.00	20.00	30.00		
8	6	Block 1	140.00	20.00	30.00		
9	18	Block 1	150.00	20.00	30.00		
10	24	Block 1	130.00	10.00	60.00		
11	30	Block 1	140.00	10.00	60.00		
12	3	Block 1	150.00	10.00	60.00		
13	9	Block 1	130.00	15.00	60.00		
14	31	Block 1	140.00	15.00	60.00		
15	23	Block 1	150.00	15.00	60.00		
16	5	Block 1	130.00	20.00	60.00		
17	27	Block 1	140.00	20.00	60.00		
18	19	Block 1	150.00	20.00	60.00		
19	11	Block 1	130.00	10.00	90.00		
20	28	Block 1	140.00	10.00	90.00		

21	14	Block 1	150.00	10.00	90.00		
22	32	Block 1	130.00	15.00	90.00		
23	10	Block 1	140.00	15.00	90.00		
24	2	Block 1	150.00	15.00	90.00		
25	25	Block 1	130.00	20.00	90.00		
26	16	Block 1	140.00	20.00	90.00		
27	12	Block 1	150.00	20.00	90.00		
28	17	Block 1	140.00	15.00	60.00		
29	21	Block 1	140.00	15.00	60.00		
30	26	Block 1	140.00	15.00	60.00		
31	15	Block 1	140.00	15.00	60.00		
32	20	Block 1	140.00	15.00	60.00		

Table 21 Design Summary

Study Type	Response Surface						
Initial Design	3 - Level Factorial						
Design Model	Quadratic						
Experiments	32						
Blocks	No Blocks						
Response	Name	Units	Obs.	Minimum	Maximum	Trans	Model
Y1	pulp Yield	%	32			None	Quadratic
Y2	Kappa num.	-	32			None	Quadratic
Factor	Name	Units	Type	Low Actual	High Actual	Low Coded	High Coded
A	Temperat.	^{o}C	Numeric	130.00	150.00	-1.000	1.000
B	NaOH Conc.	%	Numeric	10.00	20.00	-1.000	1.000
C	Time	min	Numeric	30.00	90.00	-1.000	1.000

4. RESULTS AND DISCUSSIONS

4.1. Chemical Compositions

The chemical composition of the wheat straw may provide some insight into the ease of pulping and the source of troublesome components. Table 22 shows the result of chemical characterization of wheat straw, bamboo (Sun & Cheng, 2003), bagasse (Morsink, 2007), Eucalyptus (Sjostrom, 1993) and wheat straw (Luis, Alejandro, Antonio, Ana, & Luis, 2008).

Table 22 Chemical composition of wheat straw compared with other wood and non-wood raw materials

Biomass	Cellulose (%)	Holocellulose (%)	Hemicellulose (%)	Lignin (%)	Ash (%)	Extractives (%)
Wheat straw [a]	39.8	69.27	29.47	20.98	6.27	4.1
Bamboo [b]	41-49	69 - 82	28-32	20-22	1-3	-
Wheat Straw [c]	39.72	76.2	36.48	17.28	6.49	4.01
Bagasse [d]	55	82 - 87	27-32	18-24	1.5-5	6.3[x]
Eucalyptus [e] *(Eucalyptus Camaldulensis)*	45.0	64.2	19.2	31.3	1.15	2.8

a; Current study, b; (Sun & Cheng, 2003), c; (Luis, Alejandro, Antonio, Ana, & Luis, 2008) (Tehmina & Umarah, 2012), d; (Morsink, 2007), e; (Sjostorm, 1993), [x] (Shalini, Dharm, & Tyagi, 2011)

As can be seen from table 23, the cellulose content of wheat straw 39.8% is near to that of bamboo 41-49% which has been mostly used in pulping industries and has lower cellulose content than bagasse 55%. Wheat straw has a total carbohydrate fraction (holocellulose) approximately in the range of hardwood (64-85%). This is due to the high hemicellulose (mainly pentosanes), and low lignin content compared to wood which is a characteristic feature of agro-residues. This characteristic directly influences the fibrillation of fibers during refining operations. It has been shown that the higher is the hemicellulose contents, the better will be the swelling behavior of the pulp, which leads to an increase in mechanical strength properties including tensile, burst index and tearing resistance (Shalini, Dharm, & Tyagi, 2011). Holocellulose as a whole adds to the overall strength of paper. According to the rating

system designated by Nieschlag et al. (1960), plant materials with 34% and higher cellulose content are characterized as promising for pulp and paper manufacture from a chemical composition point of view.

The lignin content in whole straw 20.98% is lower than eucalyptus 31.3% and close to that of bagasse (18-24%) and bamboo (20-22%). The amount of lignin is directly related to the consumption of cooking liquor and the length of cooking cycle. The lower lignin content in wheat straw helps for the pulp mill industry minimizes both the delignification and chemical recovery costs. Also, the higher the lignin content, the greater will be the stiffness of the fibers (Shalini, Dharm, & Tyagi, 2011).

The ash content of wheat straw is 6.27%, which is much higher than that of bamboo 1-3%, bagasse 1.5-5% and eucalyptus 1.15%. The silica content in wheat straw is relatively high as compared to wood. Silica causes rather serious difficulties during recovery of pulping chemicals as well as poor drainage of straw pulp during paper making (Tandon et al. 1989). At the same time silica can play a role of inhibitor for O_2 delignification and bleaching with H_2O_2 thereby eliminating the need for additional inhibitors to mask transition metals ions during pulping /bleaching (Pytons and Canaries, 1997).

The solvent extractives (4.1%) in wheat straw are higher than in eucalyptus (2.8%) and lower than that of bagasse (6.3%). This is due to that wheat straw contains more of substances like waxes, fats, resins, phytosterols as well as nonvolatile hydrocarbons, low molecular weight carbohydrates, salts and other water soluble substances. A higher content of extractives in pulp can have an adverse effect on the ruunablity of process equipment, and paper made from such type of fibers shows reduced water absorbency (levitin, 1970).

The cellulose content of wheat straw is also comparable to the reported cellulose content of hardwoods (38-56%) and having cellulose contents in this range makes wheat straw a suitable raw material for the paper and pulp industry.

4.2. Morphological characteristics

Table 24 shows the morphological characteristics of wheat straw fibre and its comparison with those of Cotton stalk (Gedefaw, 2015) and Bamboo (Shalini, Dharm, & Tyagi, 2011). The total fibres in wheat straw are about 39.20%, compared to 45.5% in Cotton Stalk. Therefore wheat straw produces lesser pulp yield compared to Cotton Stalk, the fibres of wheat straw are slender with sharply pointed ends and with an average fibre length of

1.21mm compared to 0.83mm for Cotton Stalk and 1.70mm for bamboo. The average fibre diameter of wheat straw is 17.2µm compared to 24.38µm for Cotton Stalk and 15.1µm for bamboo. The lumen of wheat straw fibre is narrow having width of 8.40µm compared to 15.65µm for Cotton Stalk and 6.9µm for bamboo. The cell wall thickness of wheat straw fibre is 3.8µm which is less than that of both Cotton Stalk and bamboo fibres.

Table 23 Morphological characterization of wheat straw fibres

Parameters	Wheat straw [a]	Cotton Stalk [b]	Bamboo [c]
Length(L),mm	1.21	0.83	2.30
Diameter (D),µm	17.20	24.38	15.1
Lumen width (d),µm	8.40	15.65	6.9
Cell wall thickness (W),µm	3.80	4.37	4.17
Slenderness ratio(L/D)	70.34	34.04	152.3
Flexibility coefficient,(d/D) *100	48.8	64.20	45.69
Runkel ratio,(2w/d)	0.9	0.54	1.21
Fibre,%	39.80	45.5	50*

a: current study, b: (Gedefaw, 2015) , c: (Deniz & Ates, 2002), * (Amsalu , 2015)

The slenderness ratio of wheat straw fibre is 70.34 against slenderness ratio 152.3 for bamboo and 34.04 for Cotton Stalk. The slenderness ratio (L/D) which is also termed as the felting power is inversely proportional to the fibre diameter. Fibres having higher slenderness ratio have a low degree of collapsing and conformability, and such types of paper gives more tear porosity, bulk and opacity (Reedy and Young, 2005). A low slenderness ratio means a reduced tearing resistance, which is partly because the short and thick fibres do not produce good surface contact and fibre to fibre bonding (Ogbonaaya et.al. 2007). When used for applications such as paper, the slenderness ratio of individual cells in a fibre affects the flexibility and resistance to rupture of the fibres (Maiti 1997). The flexibility coefficients of wheat straw fibres 48.8 are close to that of bamboo fibres but much less than that of Cotton Stalk. Such types of fibre do not collapse readily to form double walled ribbons, and they tend to retain their tubular structure on pressing thus provide lesser surface contact area for bonding. The Runkel ratio of wheat straw is almost equal to that of bamboo. Rankle ratio is directly affected by cell wall thickness but not lumen diameter, and is related to fibre density (Pillow et al, 1941). The breaking length, bursting strength and double fold are determined by fibre density. However due to a lower fibre diameter, the flexibility and degree of collapse of

fibre, both of which control the degree of conformability, within the paper sheet and, as such the size and number of inter fibre bonds are improved in the case of wheat straw (Shalini, Dharm, & Tyagi, 2011).

4.3. Pulp yield and Kappa number

The experimental values of pulp yield and kappa number obtained under different pulping conditions are presented in table below. These results were inputs to the DESIGN EXPERT® software version 6.0.8 for further analysis and the statistical analysis of the pulping conditions is discussed in the following section.

Table 24 Shows the values of the two response variables in a response surface 3-level factors associated with their pulping conditions.

Std.	Run	Block	Factor 1 A: temp.^{o}C	Factor 2 B: Conc. NaOH%	Factor 3 C: Time min	Response 1: Yield%	Response 2: Kappa num.
1	7	Block 1	130.00	10.00	30.00	35.0	23.46
2	4	Block 1	140.00	10.00	30.00	32.9	10.36
3	1	Block 1	150.00	10.00	30.00	30.4	9.40
4	29	Block 1	130.00	15.00	30.00	33.9	21.72
5	13	Block 1	140.00	15.00	30.00	31.5	8.79
6	8	Block 1	150.00	15.00	30.00	29.1	8.40
7	22	Block 1	130.00	20.00	30.00	33.0	20.21
8	6	Block 1	140.00	20.00	30.00	30.8	7.82
9	18	Block 1	150.00	20.00	30.00	28.0	7.44
10	24	Block 1	130.00	10.00	60.00	32.9	13.56
11	30	Block 1	140.00	10.00	60.00	28.7	9.18
12	3	Block 1	150.00	10.00	60.00	25.2	7.24
13	9	Block 1	130.00	15.00	60.00	30.1	11.95
14	31	Block 1	140.00	15.00	60.00	27.6	7.44
15	23	Block 1	150.00	15.00	60.00	23.1	6.29
16	5	Block 1	130.00	20.00	60.00	28.0	7.05
17	27	Block 1	140.00	20.00	60.00	24.8	6.72
18	19	Block 1	150.00	20.00	60.00	22.0	4.03
19	11	Block 1	130.00	10.00	90.00	32.1	8.40

20	28	Block 1	140.00	10.00	90.00	28.0	5.91
21	14	Block 1	150.00	10.00	90.00	23.7	5.53
22	32	Block 1	130.00	15.00	90.00	29.7	7.63
23	10	Block 1	140.00	15.00	90.00	26.6	4.96
24	2	Block 1	150.00	15.00	90.00	21.3	4.40
25	25	Block 1	130.00	20.00	90.00	27.6	4.78
26	16	Block 1	140.00	20.00	90.00	22.7	4.60
27	12	Block 1	150.00	20.00	90.00	20.0	2.73
28	17	Block 1	140.00	15.00	60.00	29.4	6.86
29	21	Block 1	140.00	15.00	60.00	26.7	7.63
30	26	Block 1	140.00	15.00	60.00	28.8	7.24
31	15	Block 1	140.00	15.00	60.00	29.1	8.21
32	20	Block 1	140.00	15.00	60.00	24.6	7.82

4.4. Effects of pulping conditions

4.4.1. Effects of individual process variables

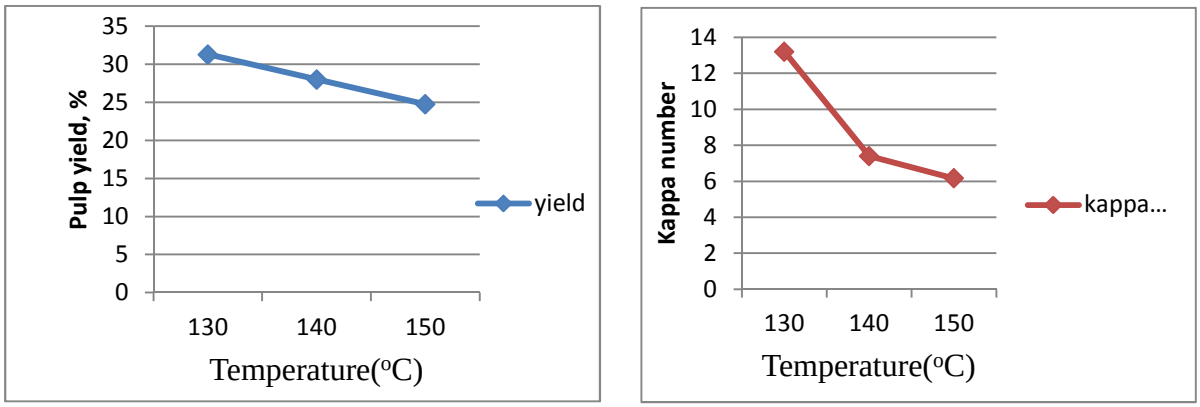
The effects of pulping time, temperature and active alkali on pulp yield and kappa number was studied and evaluated for best pulping conditions.

4.4.1.1. Effects of Temperature

The increase in temperature of reaction from 130^{o}C to 140^{o}C has shown the decrease of pulp yield percentage by slightly 10.4%. This was again supported by decrease of kappa number of pulp more than 44%. The influence of cooking temperature was much greater in kappa number than the yield i.e. an average of 1.1% decrease in pulp yield and 3% decrease in kappa number was observed for each 1^{o}C increase in cooking temperature. The study also showed that most of the lignin about 82.5% was removed during the cooking period increase from 130 to140^{o}C.

As presented in figure 15 a and b, higher pulp yield and higher Kappa number were obtained at the lower temperature of 130^{o}C, while lower pulp yield and lower Kappa number were obtained at the higher temperature of 150^{o}C. This implies that, high rate of degradation of cellulose and delignification occurs at elevated temperature. During pulping both lignin and cellulose are dissolved at different rates. This rate is much accelerated by increasing the temperature (Iglesias et al., 1996). There was a general decrease in the pulp yield and Kappa

number due to increases in the pulping temperature at constant time (Akpakpan, Akpabio, Ogunsile, & Eduok, 2011). The detailed effects are shown in graphs given below.

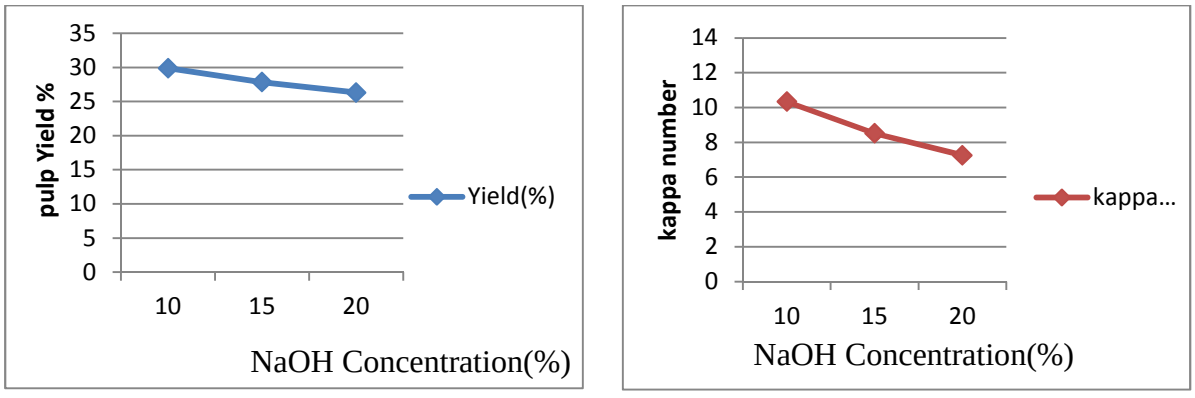


(a) **(b)**

Figure 15 Effect of cooking temperature on average pulp yield (a) and kappa number (b)

4.4.1.2. Effects of NaOH concentration

The increase in concentration of caustic soda improves the delignification and provides better quality pulp with lower lignin content. Pulp yield decreases slowly with increase of caustic soda concentration in liquor due to increase of delignification and solubilization of hemicelluloses in caustic soda. But the quality of pulp obtained at higher pulp yield is not suitable for further processing in paper industry due to high kappa number and residual lignin in pulp. In wheat straw chemical pulping, the increase in NaOH concentration affects the yield slightly and highly the kappa number i.e. the total pulp yield was decreased by 1.2% while the kappa number was decreased by about 3% for each increases in 1% NaOH concentration. The detailed effects are shown in graphs given below.

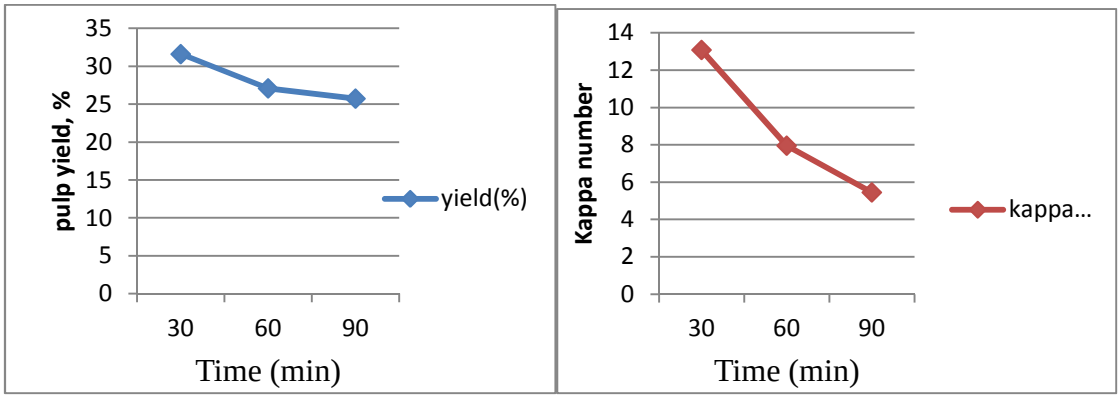


(a) **(b)**

Figure 16 Effect of NaOH concentration on average pulp yield (a) and average kappa number (b)

4.4.1.3. Effects of cooking time

Reaction time increase leads to completion of reaction. Time increase up to 60 minutes results in lowering of pulp yield percentage to a level of 27.08% from 31.62% which shows a slow change. But in the case of kappa number an increase of cooking time up to 60 minutes results a corresponding reduction of 39% which shows a significant effect. Detailed effects of time variations are shown in graphs given in Figure 17.



(a) **(b)**

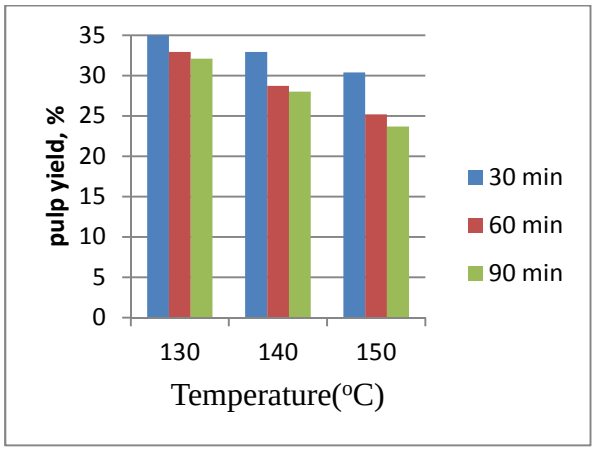
Figure 17 Effect of cooking time on average pulp yield (a) and average kappa number (b)

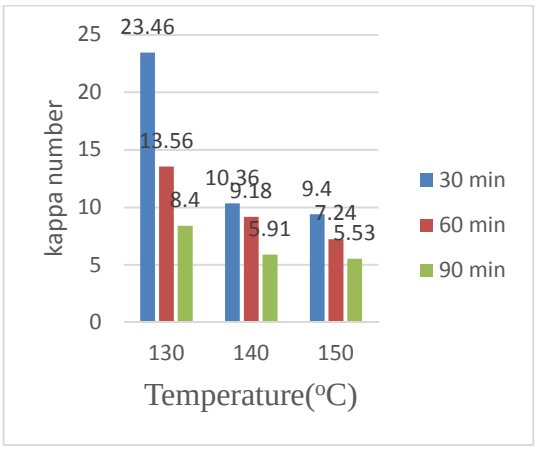
4.4.2. Interaction Effects of Process Variables

4.4.2.1. Effects of cooking time and temperature at constant concentration of NaOH

A pulp yield 35% and kappa number 23.46 were obtained at 10% NaOH concentration at 130^{o}C temperature and 30 minutes of reaction time when liquor to straw ratio was maintained at 9.

Increasing the cooking temperature by 10^{o}C and cooking time by 30minutes result a corresponding decrease in pulp yield about 6% and kappa number by 55.6%. Here also further increase the process variable by the same unit reduces the pulp yield and kappa number by 7.6% and 9.2% respectively. This showed that most of the lignin in wheat straw was solubilized and removed during the first increase of 30minutes cooking time and temperature from 130^{o}C to 140^{o}C and the yield was decreased little by little to a total of 13%.

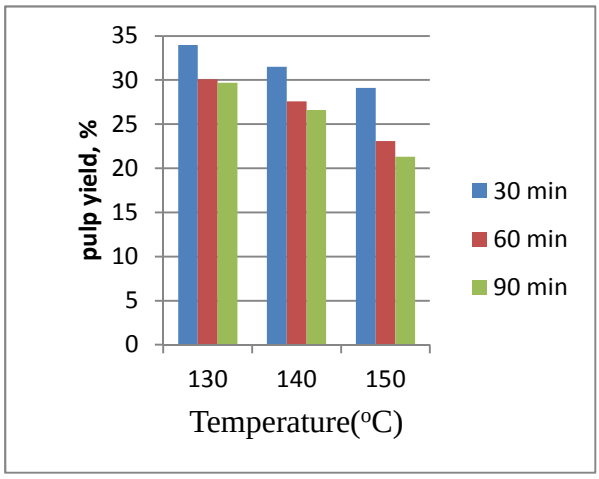


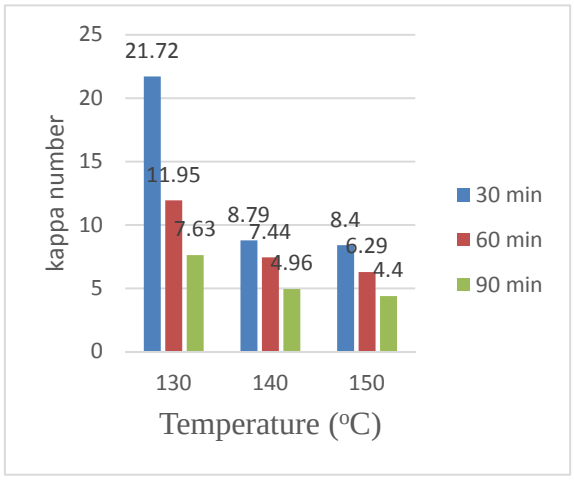


(a) **(b)**

Figure 18 Effect of cooking temperature on pulp yield (a) kappa number (b) at 10% NaOH concentration

When the NaOH concentration has changed to 15%, a total reduction of pulp yield 14.2% and kappa number 61.3% were observed as shown from figure 19.

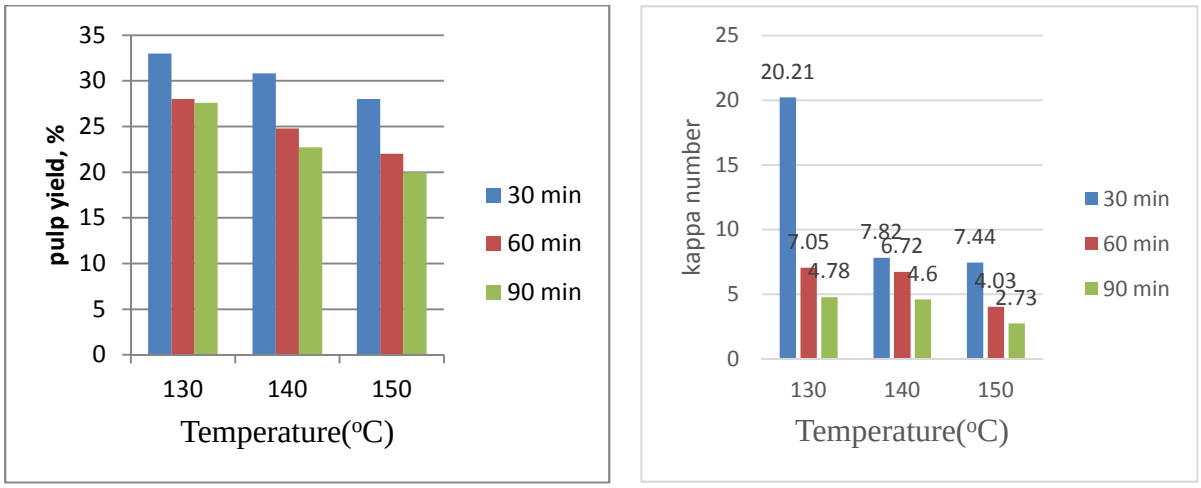




(a) **(b)**

Figure 19 Effect of cooking temperature on pulp yield (a) kappa number (b) at 15% NaOH concentration

Raising the concentration of NaOH to 20%, while maintaining other process variables the same as the previous, resulted a total pulp yield reduction about 15% and kappa number by 63%. From this study of wheat straw chemical pulping as the interaction effect of cooking time and temperature shows the reduction rate of response variables pulp yield and kappa number were increased along with NaOH concentration.



(a) **(b)**

Figure 20 Effect of cooking temperature on pulp yield (a), kappa number (b) at 20% NaOH concentration

4.4.3. Adequacy check for the developed response surface quadratic models

The adequacy of the model was checked by analysis of variance (ANOVA) and some diagnostic plots. Analysis of variance (ANOVA) is employed to test the significance of the developed models. Table 25 and, table 26 shows the summary of the analysis of variance (ANOVA) of the two response variables i.e. Pulp yield and kappa number.

Table 25 Analysis of variance [Partial sum of squares], pulp yield

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	440.40	9	48.93	50.01	< 0.0001	Significant
A	197.01	1	197.01	201.36	< 0.0001	
B	56.89	1	56.89	58.14	< 0.0001	
C	155.76	1	155.76	159.20	< 0.0001	
A^2	0.58	1	0.58	0.59	0.4492	
B^2	0.22	1	0.22	0.23	0.6377	
C^2	17.90	1	17.90	18.29	0.0003	
AB	0.37	1	0.37	0.38	0.5462	
AC	8.25	1	8.25	8.43	0.0082	
BC	4.08	1	4.08	4.17	0.0532	

Residual	21.53	22	0.98			
Cor Total	461.93	31				

Table 26 Analysis of variance [Partial sum of squares], kappa number

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	662.19	9	73.58	25.78	< 0.0001	significant
A	222.61	1	222.61	78.00	< 0.0001	
B	42.50	1	42.50	14.89	0.0008	
C	261.90	1	261.90	91.77	< 0.0001	
A^2	33.93	1	33.93	11.89	0.0023	
B^2	1.48	1	1.48	0.52	0.4784	
C^2	5.77	1	5.77	2.02	0.1692	
AB	2.44	1	2.44	0.85	0.3653	
AC	85.33	1	85.33	29.90	< 0.0001	
BC	3.333E-005	1	3.333E-005	1.168E-005	0.9973	
Residual	62.79	22	2.85			
Cor Total	724.98	31				

Based on a 95% confidence level, F-Value is a test for comparing model variance with residual (error) variance. If the variances are close to each other, the ratio will be close to one and it is less likely that any of the factors have a significant effect on the response with the P-value less than 0.05. It is calculated by Model Mean Square divided by Residual Mean Square. Also, the high F-value and a very low probability indicate that the present models are in a good prediction of the experimental results. The P- value serves as a tool for checking the significance of each of the coefficients. The variable with low probability levels contribute to the model, whereas the others can be neglected and eliminated from the model.

In this study for the response pulp yield and kappa number the Model F-value of 50.01 and 25.78 respectively implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case realized that for the response pulp yield the individual factors A, B, C, C^2 and the interaction effects AC are significant model terms. In the case of Kappa number the individual factors A, B, C, A^2 and the interaction effects AC are significant model terms. Values greater than 0.10 indicate the model terms are not significant.

If there are many insignificant model terms (not counting those required to support hierarchy), model reduction would improve the model.

The quality of the model developed could be evaluated from their coefficients of correlation, R^2 (correlation coefficient) was high for pulp yield; a value > 0.75 indicates the apropraitness of the model. For a good statistical mode, the R^2 value should be close to one.

Table 27 Model adequacy measures for pulp yield

Std. Dev.	0.99	R-Squared	0.9534
Mean	28.04	Adj R-Squared	0.9343
C.V.	3.53	Pred R-Squared	0.9294
PRESS	32.60	Adeq Precision	29.036

Table 28 Model adequacy measures for Kappa number

Std. Dev.	**1.69**	**R-Squared**	**0.9134**
Mean	8.68	Adj R-Squared	0.8780
C.V.	19.46	Pred R-Squared	0.7881
PRESS	153.59	Adeq Precision	20.663

The value of R^2 for pulp yield, 0.9534 which is very high and close to one which indicates a good agreement between experimental and predicted values. The "Pred R-Squared" of 0.9294 is in reasonable agreement with the "Adj R-Squared" of 0.9343.

In the case of Kappa Number the value of R^2 for pulp yield, 0.9134 which is very high and close to one which indicates a good agreement between experimental and predicted values. The "Pred R-Squared" of 0.7881 is in reasonable agreement with the "Adj R-Squared" of 0.8780.

Standard Deviation which is a square root of the residual mean square and it is the standard deviation associated with the experimental error; mean which is the dependent mean is the average of all the values for this particular response; C.V. % (Coefficient of Variation) is the error expressed as a percentage of the mean and computed as 100 x (Std Dev)/(Mean); PRESS is the Predicted Residual Sum of Squares for the model which is a measure of how well a particular model fits each point in the design; R^2 which measures the proportion of the total variability explained by the model. Whereas the adjusted R^2 statistics, defined as is a statistic that is adjusted for the “size” of the mode; that is the number of the factor. The adjusted R^2 can actually decrease if non-significant terms are added to a model. Pred R

Square is a measure of how good the model predicts a response value and the Adjusted R-Squared and Predicted R-Squared should be within approximately 0.20 of each other to be in "reasonable agreement." If they are not, there may be a problem with either the data or the model;

Adequate precision is a measure of the range in predicted response relative to its associated error, in other words a signal to noise ratio and its desired value is 4 or more. In this case the ratio of 29.036 and 20.663 for pulp yield and kappa number respectively indicates an adequate signal which could be used to navigate the design space or decide whether the model can be used or not.

Table 29 The details of model coefficients for pulp yield

Factor	Coefficient Estimate	DF	Standard Error	95% CI Low	95% CI High	VIF
Intercept	27.41	1	0.33	26.72	28.10	
A-Temperature	-3.31	1	0.23	-3.79	-2.82	1.00
B-NaOH Conc.	-1.78	1	0.23	-2.26	-1.29	1.00
C-Time	-2.94	1	0.23	-3.43	-2.46	1.00
A^2	-0.28	1	0.37	-1.05	0.48	1.10
B^2	-0.18	1	0.37	-0.94	0.59	1.10
C^2	1.58	1	0.37	0.81	2.35	1.10
AB	0.18	1	0.29	-0.42	0.77	1.00
AC	-0.83	1	0.29	-1.42	-0.24	1.00
BC	-0.58	1	0.29	-1.18	8.845E-003	1.00

Final Model Equation in Terms of Coded Factors:

Pulp Yield = +27.41

$-3.31 * A - 1.78 * B - 2.94* C$

$-0.28 * A^2 - 0.18 * B^2 + 1.58 * C^2$

$+0.18 * A * B - 0.83 * A * C- 0.58 * B * C$ (4.1)

Where the variables are:

A-Temperature,

B- NaOH Concentration and

C- Cooking time.

Final Model Equation in Terms of Actual Factors:

Pulp Yield = 14.46667

+0.58035 * A - 0.40029* B + 0.13633 * C

-2.84946E-003 * A^2 - 7.06452E-003 * B^2 + 1.75747E-003 * C^2

+3.50E-003* A * B -2.76E-003* A * C -3.89E-003* B * C (4.2)

Table 30 The details of coefficients estimates for kappa number

Factor	Coefficient Estimate	DF	Standard Error	95% CI Low	95% CI High	VIF
Intercept	7.21	1	0.57	6.03	8.38	
A-Temperature	-3.52	1	0.40	-4.34	-2.69	1.00
B-NaOH Conc.	-1.54	1	0.40	-2.36	-0.71	1.00
C-Time	-3.81	1	0.40	-4.64	-2.99	1.00
A^2	2.18	1	0.63	0.87	3.49	1.10
B^2	-0.46	1	0.63	-1.77	0.85	1.10
C^2	0.90	1	0.63	-0.41	2.21	1.10
AB	0.45	1	0.49	-0.56	1.46	1.00
AC	2.67	1	0.49	1.66	3.68	1.00
BC	1.667E-003	1	0.49	-1.01	1.01	1.00

Final Model Equation in Terms of Coded Factors:

Kappa num. Kappa num. = 7.21

-3.52 * A - 1.54 * B - 3.81 * C

+2.18 * A^2 - 0.46 * B^2 + 0.90 * C^2

+0.45 * A * B + 2.67 * A * C + 1.667E -003 * B * C (4.3)

Where: A -Temperature

B - Concentration of NaOH and

C - Cooking time

Final Model Equation in Terms of Actual Factors:

Kappa number = 588.64056

-7.11823 * A - 1.02375* B - 1.49147 * C

+ 0.021778 * A^2 - 0.018219 * B^2 + 9.976E-004 * C^2

+9.016E-003* A * B +8.89E-003* A * C +1.1E-005* B * C (4.4)

4.5. Process Factors and Response variables optimization

One of the objectives of this study was to find the optimal process parameters for better pulp yield at minimum kappa number. Optimization allows generating predicted response(s) for any set of factors. The process variables such as cooking temperature, concentration of active alkali and pulping time have been optimized.

Since the goals of optimization was to maximize economic benefit by minimizing process cost, the process variables need to set at their minimum value and the two response variables pulp yield and kappa number were set to maximum and minimum levels respectively. Once the optimum pulping condition was obtained, wheat straw pulp was produced and paper sheets formed. The produced paper sheet was tested for several strength and optical properties as shown from table 36.

The table below shows summary of factors, responses, goals and the corresponding set of specific objectives that will optimize the process condition to have the desired responses and Numerical optimization was used to optimize any combination of one or more factors.

Table 31 Summary of factors, responses and goals of optimization

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
Temperature	minimize	130	150	1	1	3
NaOH Conc.	minimize	10	20	1	1	3
Time	minimize	30	90	1	1	3
pulp Yield	maximize	20	35	1	1	3
Kappa num.	minimize	2.73	23.46	1	1	3

Table 32 The possible solutions obtained from design expert software of numerical optimization

Number	Temperature ^{o}C	NaOH Conc. %	Time	pulp Yield %	Kappa num.	Desirability	
1	137.05	10.00	30.00	33.7124	15.1506	0.750	
2	136.97	10.00	30.00	33.7311	15.2107	0.750	Selected
3	137.14	10.00	30.00	33.6891	15.0771	0.750	
4	137.05	10.00	30.53	33.6204	15.0394	0.750	

5	136.99	10.06	30.00	33.7159	15.1863	0.749	

As it has been shown above the table, five solutions was generated from design expert software as per the goals set in the criteria part, though the solutions have different values of process factors to achieve the corresponding response variables, the actual response out puts i.e. pulp yield and kappa number were the same since all the process variables in all possible solutions were very close to each other.

The desirability lies between 0 and 1, which represents the closeness of a response to its ideal value. If a response falls within the unacceptable intervals, the desirability is 0 and if a response falls within the ideal intervals or the response reaches its ideal value, the desirability is 1. Meanwhile, when a response falls within the tolerance intervals but not the ideal interval, or when it fails to reach its ideal value, the desirability lies between 0 and 1. The more closely the response approaches the ideal intervals or ideal values, the closer the desirability is to 1. Based on the above analysis best local maximum for pulp yield 33.73% with corresponding kappa number 15.21 was found at active alkali 10%, Sulphidity 20%, temperature 136.69^{o}C and 30 minutes cooking time and the value of desirability obtained was 0.750.

The model validations have been determined as optimum levels of the process parameters to achieve the pulp yield 33.73%. In order to verify this prediction, experiments were conducted and the results showed that 33.91% pulp yield was obtained with Kraft pulping table 34. Therefore, the numerical optimization can be taken as optimal value because the predicted value is close enough with experimental/actual value.

The model capable of predicting the maximum pulp yield and the minimum kappa number value showed from table 33, that the optimum values of the process variables were temperature of 136.97^{o}C, active alkali of 10% and cooking time of 30minute. Under these conditions, the predicted pulp yield and kappa number were 33.73% and 15.21 respectively. Desirability function was used to identify the optimum levels of factors and to get maximum desirable responses and the optimized combinations of process variables was selected among the solutions generated with maximum combined desirability value i.e. 0.75 from table 32.

Table 33 Model validation

Pulping Temperature	Active alkali charge	Cooking Time		Pulp yield	Kappa number
136.9^{o}C	10%	30 min	Predicted	33.73%	15.21
136.9^{o}C	10%	30 min	Experimental	33.91%	16.76

4.6. Materials consumption per tonnes of brown pulp production and bleaching

Once the optimum pulping condition has been established pulping, bleaching, sheet forming and sheet physical characteristics tests were proceed. Since the minimum amount of pulp required for paper mill machine and sheet forming process was 400 gram, the following input materials has been consumed for the production and bleaching process. In addition to this the consumptions of raw materials and chemicals per tonnes of brown pulp using Kraft pulping method based on the specified pulping and bleaching conditions were estimated from the experimental findings in table 34.

Table 34 Consumption of wheat straw and chemicals per tonnes of brown pulp production and bleaching

Inputs	Per 400 g brown pulp		Per ton of brown pulp	
	Pulping	Bleaching	Pulping	Bleaching
Wheat straw	1180g	-	2950 kg	-
NaOH	290 g	26 g	2170 kg	65 kg
Na_2S	145 g	-	1100 kg	-
H_2O_2	-	32 ml	-	80 lit
$Ca(ClO)_2$		28.6 g	-	71 kg
Water (washing)	121 lit	20 lit	911m^3	127.3 m^3

NB: washing in both cases were carried out until the washed effluent pH becomes neutral.

The brightness of pulp can be increased and the kappa number can be reduced by increasing the bleaching time, temperature and concentrations bleaching solvents. But further increase these bleaching conditions decreases the pulp yield due to the degradation of soluble carbohydrates mainly the hemicellulose.

Table 35 Losses in yield, kappa number and lignin content due to bleaching of pulp after optimization.

Parameters	Before bleaching	After bleaching	% loss
Pulp (g)	400	372	7.2
Kappa number (%)	16.76	9.85	41.2
Lignin content (%)	2.5	1.48	40.8

4.7. Paper Sheet Strength Properties

After optimization of the cooking conditions of wheat straw Kraft pulping, hand sheets were prepared from the pulp produced at optimized pulping condition and tested for different physical properties as per the method presented in section 3.5. The results obtained were presented in the following table and compared with eucalyptus derived paper widely applicable in the world.

Table 36 Strength properties of different Grammage paper sheet obtained experimentally from the bleached and unbleached wheat straw pulp.

Treatment	G (g/m^2)	Bu.S (kg/cm^2)	Bu.S (Kpa)	Bu.I (Kpa m^2/g)	Tn.S (KN/m)	Br.L (m)	Tn.I (Nm/g)	Te.R (mN)	Te.F (mN) m^2/g)	Te.I (mN) m^2/g)
Bleached pulp(WB)	60	1.5	147	2.45	2.24	3803	36	44	73	7.18
	80	2	196	2.45	2.89	3691	37	72	90	8.82
Unbleached pulp(BB)	60	1.9	186	2.66	2.76	4027	37	64	91	8.96
	80	2.8	274	3.43	2.96	3775	39	76	95	9.31

*G: Grammage, Bu.S: Bursting strength, Bu.I: Bursting index,Tn.S:Tensile strength, Br.L: Breaking length , Tn.I: Tensile index, Te.R: Tear resistance, Te.F: Tear factor, Te.I: Tear index, WB: white bond, BB: brown bond

Table 37 Comparision of wheat straw paper sheet with paper sheets obtained from other raw material sources

Category	G g/m^2	Bu.S kg/cm^2	Bu.I Kpa m^2/g	Tn.S KN/m	Tn.I Nm/g	Br.L m	Te.R 10^{-3}N	Te.F 10^{-3}N m^2/g	Te.I 10^{-3}N m^2/g
Wheat straw[a]	60	1.5	2.45	2.24	36	3803	44	73	7.18

ISO requiment[b]	60	na	1.45	na	40	na	na	na	5.4
Khar grass[c]	60	0.26	0.56	na	24.60	na	4.5	na	3.48
Wheat straw[d]	60	na	1.32	na	43.2	na	na	na	5.25
Eucalyptus[e]	60	na	1.35	na	28.0	na	na	na	3.90

a: current study ,b : (Caulfield. & Gunderson., 1988) , c: (Akhouri & Kumar , 2015), d: (Huang, Zhang, & Chen, 2006) , e: (Priti S. L., Vimlesh, Arvind, & Vinay , 2013). WB:white bond, BB: brown bond, ISO: international standards for organisations, na: not avialable

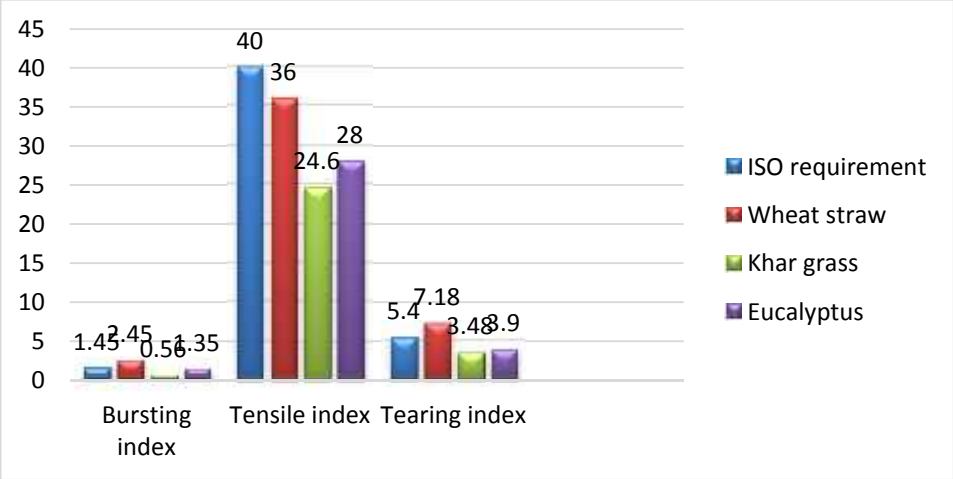


Figure 21 Shows the strength properties of paper sheet from different materials

The burst index of paper sheets 2.45 kPa m^2/g is above the ISO minimum requirements 1.45 Kpa m^2/g which is suitable for writing grade, book printing, computer printing and newsprint paper. Tear index of wheat straw paper sample $7.18*10^{-3}$ N m^2/g is greater than that of paper from eucalyptus and minimum ISO requiremnts $3.9*10^{-3}$ Nm^2/g and $5.4*10^{-3}$ Nm^2/g respectively and it is good for average grades of writing and printing paper. The tensile index of wheat straw paper sample has value 36 Nm /g which is below the ISO requirments 40 Nm /g and greater than that of eucalyptus 28 Nm /g . This is due to that wheat starw has short fiber length and the paper lacks bondig strength, but it is quite good considering such an indigenous pulp obtained from non-woody material. This tensile properties of paper sheet can be improved significantly by addition of some long fiber (as in usual practice in paper industries using short fiber pulp) in wheat straw based short fiber stock and can make it suitable for good quality writing and printing.

5. CONCLUSION AND RECOMMENDATION

5.1. Conclusions

The results of morphological study showed that wheat straw contained short fibers with similar morphological properties to the common non-wood fibers. Chemical composition analysis showed that the cellulose content of wheat straw was comparable to other Non-wood Papermaking fiber resources.

From the morphological characterization, properties of wheat straw fibers are satisfactory for papermaking, although they were classified as short fibers. Wheat straw fibers can be collapsed to form ribbon like structures in the paper.

In part of chemical compositions, the cellulose content of wheat straw was found to be 39.8%, which is satisfactory for pulp and paper production. In addition to this the cellulose content of wheat straw is comparable to the reported cellulose content of softwoods (40-52%) and hardwoods (38-56 %). The lignin content of wheat straw was comparable to other non-wood papermaking fiber resources and less than wood fiber sources. And it was also found that the wheat straw contained high amounts of extractives and ash due to the presence of high silica content.

It was found that the yield and kappa number of wheat straw pulp was influenced by pulping variables such as concentrations of active alkali, cooking temperature and time. Pulp yield was slightly influenced by these process variables and a slight decrease in pulp yield was observed up on increasing the levels from the bottom to the next. But a rapid falling was observed in the case of kappa numbers when increasing levels of process variables from bottom to the next and most of the delignification process was carried out during this period. Generally both the pulp yield and kappa numbers were inversely related to the processing variables.

The optimized wheat straw pulping conditions that has been considered high pulp yield, low kappa number, low chemical and energy consumption with short pulping time chosen using numerical optimization as a combinations of 10% active alkali, 136.9^{o}C temperature and 30 minutes time while keeping Sulphidity 20% and liquor to straw ratio 9:1 is practically feasible. Because the experimentally obtained results of pulp yield and kappa numbers 33.91% and 16.76, were very close to the software predicted values 33.73% and 15.21 respectively.

As the average tearing strength, bursting strength and their derived index of wheat straw showed it is comparable with the requirements used by ISO. But the tensile properties of wheat straw paper is a little lower than set by ISO due to the presence short fibres leading weak physical bonding among fibres. But the overall physical property results of wheat straw paper sheet lied in the acceptable ranges of international standards for organizations. And from the experimental results, the chosen optimized wheat straw pulping condition was fit and suitable both environmentally and economically.

Generally in addition to the suitable morphological and chemical properties of wheat straw, it can be an alternative source of non-wood fibers for pulp and paper production since it grows and widely available in most part of Ethiopia.

5.2. Recommendations

• Method of chemical pulping used for this study was Kraft or sulphate pulping process which provides a bleachable grade wheat straw pulp with yield 33.91%. But before doing any further study on it, investigation of another method of chemical pulping such as soda method is advisable if it results better pulp yield.

• It is also possible to study the effects on pulp yield and paper sheet physical characteristics by using wheat straw in combination with other widely available agricultural by products which might be feasible environmentally and economically.

• One can also investigate the effects of Sulphidity on the yields and kappa numbers of wheat straw pulp.

• For every processing industry, waste treatment and its characterization is one of the operational parts. So in pulp and pulp mills, black liquor is the major by product released from the process containing both organic materials and inorganic minerals which can be valuable inputs for petroleum industries and heat generation. Therefore investigations should be carried out on black liquor characterization and chemical recovery process.

• Wheat straw of *Triticum Astivum* species that has been used for the study was collected from specified part of our country, but the mineral content the soil and variety of the wheat will have an impact on the pulp yield and physical properties of the sheet, therefore one can do investigate the effects by collecting wheat straw from different parts of country and different species of wheat and even by using them in combinations.

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Appendix

Appendix A: Supporting pictures during the study

(a) (b)

Figure A_1. Wheat straw harvesting in the field (a) and size reduced and kept in poly bag (b)

(a) **(b)** **(c)**

Figure A_2. Cooking chemicals (a), straw socked in white liquor(b) cooking autoclave(c)

(a) **(b)** **(c)**

Figure A_3. Wheat straw after cooking (a) black liquor (b), after draining black liquor

(a) **(b)** **(c)**

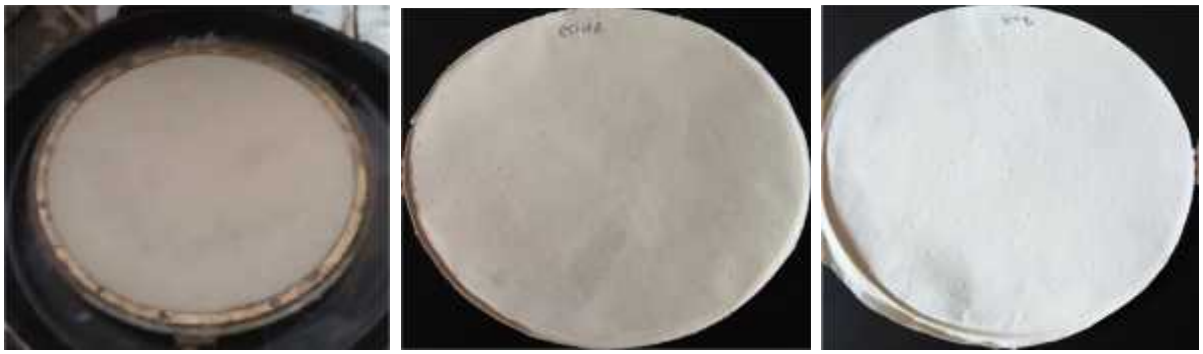
Figure A_4. Wheat straw pulp partially washed (a), fully washed (b) and after dried (c)

(a) **(b)** **(c)**

Figure A_5. Pulp beating machine (a), freeness tester(b), pulp disintegrator (c)

(a) **(b)** **(c)**

Figure A_6 Disintegrated pulp (a), sheet forming machine (b), sheet pressing machine (c)



(a) **(b)** **(c)**

(d) **(e)**

Figure A_7. Brown pulp on sheet forming mschine(a), sheet 80 g/cm^2 from unbleached pulp(b), bleached pulp(c), 60 and 70 g/cm^2 sheets(d) and a 10cm by 10cm sheet for grammage tests(e)

(i) **(ii)** **(iii)** **(iv)** **(v)**

Figure A_8. The process of wheat straw pulping from straw preparation to drying.

Appendix B: Tables contain different data inputs during the study

Table B_1 The quantity imported and associated costs of pulp and paper in Ethiopia from 1909-2016.

Year	Pulp		Paper	
	Quantity (kg)	Cost in (USD)	Quantity (kg)	Cost in (USD)
2009	4,013,981	2,200,849.781	79,880,956.90	84,651,044.37
2010	1,759,353.89	1,632,848.177	74,798,037.69	82,450,928.73
2011	6,646,105	5,668,217.546	84,172,647.48	101,833,766.21
2012	8,617,781.00	6,759,688.00	101,756,192.18	116,359,837.88
2013	10,216,188.00	8,059,794.46	107,519,199.07	132,301,772.92
2014	5,101,365.00	4,214,596.83	136,955,682.27	127,237,688.80
2015	7,181,482.36	6,025,699.99	108,632,555.78	135,953,748.31
2016	7,852,516.85	6,310,023.24	150,731,302.60	167,529,679.09

Table B_2 Raw data for pulp yield determination

Run	Weight of raw materials taken(g)	Weight of pulp obtained (g)	Yield (%)
7	30	10.50	35.0
4	30	9.87	32.9
1	30	9.12	30.4
29	30	10.18	33.9
13	30	9.45	31.5
8	30	8.73	29.1
22	30	9.90	33.0
6	30	9.24	30.8
18	30	8.40	28.0
24	30	9.87	32.9
30	30	8.61	28.7
3	30	7.56	25.2
9	30	9.03	30.1
31	30	8.28	27.6
23	30	6.93	23.1
5	30	8.40	28.0
27	30	7.44	24.8

19	30	6.60	22.0
11	30	9.63	32.1
28	30	8.40	28.0
14	30	7.11	23.7
32	30	8.91	29.7
10	30	7.98	26.6
2	30	6.39	21.3
25	30	8.28	27.6
16	30	6.81	22.7
12	30	6.00	20.0
17	30	8.82	29.4
21	30	8.01	26.7
26	30	8.64	28.8
15	30	8.73	29.1
20	30	7.38	24.6

Table B_3 Raw data for kappa number and lignin content determination

Run	Mass of pulp taken(g)	V1 (ml)	V2 (ml)	V a (ml)	Kappa number	Lignin content
7	1	25	13.2	23.6	23.46	5.42
4	1	25	19.5	11.0	10.36	5.10
1	1	25	20.0	10.0	9.40	4.71
29	1	25	14.0	22.0	21.72	5.26
13	1	25	20.3	9.4	8.79	4.88
8	1	25	20.5	9.0	8.40	4.51
22	1	25	14.7	20.6	20.21	5.11
6	1	25	20.8	8.4	7.82	4.77
18	1	25	21.0	8.0	7.44	4.34
24	1	25	17.9	14.2	13.56	4.97
30	1	25	20.1	9.8	9.18	4.45
3	1	25	21.1	7.8	7.24	3.67
9	1	25	18.7	12.6	11.95	4.66
31	1	25	21.0	8.0	7.44	4.27

23	1	25	21.6	6.8	6.29	3.30
5	1	25	21.2	7.6	7.05	4.27
27	1	25	21.9	6.2	6.72	3.84
19	1	25	22.8	4.4	4.03	3.41
11	1	25	20.5	9.0	8.40	5.10
28	1	25	21.8	6.4	5.91	3.90
14	1	25	22.0	6.0	5.53	4.34
32	1	25	20.9	8.2	7.63	4.60
10	1	25	22.3	5.4	4.96	3.58
2	1	25	22.6	4.8	4.40	4.12
25	1	25	22.4	5.2	4.78	4.34
16	1	25	22.5	5.0	4.60	3.52
12	1	25	23.5	3.0	2.73	3.10
17	1	25	21.3	7.4	6.86	4.56
21	1	25	20.9	8.2	7.63	4.14
26	1	25	21.1	7.8	7.24	4.46
15	1	25	20.6	8.8	8.21	4.51
20	1	25	20.8	8.4	7.82	3.81

Table B_4 Results of the response variables (pulp yield and kappa number) in wheat straw Kraft pulping.

Std.	Run	Block	Factor 1 A: temp. ^oC	Factor 2 B: Conc. NaOH %	Factor 3 C: Time min	Response 1: Yield %	Response 2: Kappa num.
1	7	Block 1	130.00	10.00	30.00	35.0	23.46
2	4	Block 1	140.00	10.00	30.00	32.9	10.36
3	1	Block 1	150.00	10.00	30.00	30.4	9.40
4	29	Block 1	130.00	15.00	30.00	33.95	21.72
5	13	Block 1	140.00	15.00	30.00	31.5	8.79
6	8	Block 1	150.00	15.00	30.00	29.1	8.40
7	22	Block 1	130.00	20.00	30.00	33.0	20.21
8	6	Block 1	140.00	20.00	30.00	30.8	7.82
9	18	Block 1	150.00	20.00	30.00	28.0	7.44
10	24	Block 1	130.00	10.00	60.00	32.9	13.56

11	30	Block 1	140.00	10.00	60.00	28.7	9.18
12	3	Block 1	150.00	10.00	60.00	25.2	7.24
13	9	Block 1	130.00	15.00	60.00	30.1	11.95
14	31	Block 1	140.00	15.00	60.00	27.6	7.44
15	23	Block 1	150.00	15.00	60.00	23.1	6.29
16	5	Block 1	130.00	20.00	60.00	28.0	7.05
17	27	Block 1	140.00	20.00	60.00	24.8	6.72
18	19	Block 1	150.00	20.00	60.00	22.0	4.03
19	11	Block 1	130.00	10.00	90.00	32.1	8.40
20	28	Block 1	140.00	10.00	90.00	28.0	5.91
21	14	Block 1	150.00	10.00	90.00	23.7	5.53
22	32	Block 1	130.00	15.00	90.00	29.7	7.63
23	10	Block 1	140.00	15.00	90.00	26.6	4.96
24	2	Block 1	150.00	15.00	90.00	21.3	4.40
25	25	Block 1	130.00	20.00	90.00	27.6	4.78
26	16	Block 1	140.00	20.00	90.00	22.7	4.60
27	12	Block 1	150.00	20.00	90.00	20.0	2.73
28	17	Block 1	140.00	15.00	60.00	29.4	6.86
29	21	Block 1	140.00	15.00	60.00	26.7	7.63
30	26	Block 1	140.00	15.00	60.00	28.8	7.24
31	15	Block 1	140.00	15.00	60.00	29.1	8.21
32	20	Block 1	140.00	15.00	60.00	24.6	7.82

Appendix C: Results obtained after analysis of response variables using DESIGN EXPERT 6.0.8 software.

Table C1 Diagnostics Case Statistics for pulp yield

Standard Order	Actual Value	Predicted Value	Residual	Leverage	Student Residual	Cook's Distance	Outlier t	Run Order
1	35.00	35.32	-0.32	0.497	-0.460	0.021	-0.452	7
2	32.90	32.95	-0.053	0.340	-0.066	0.000	-0.065	4
3	30.40	30.01	0.39	0.497	0.550	0.030	0.541	1
4	33.95	34.13	-0.18	0.340	-0.224	0.003	-0.219	29

5	31.50	31.94	-0.44	0.211	-0.496	0.007	-0.487	13
6	29.10	29.17	-0.071	0.340	-0.089	0.000	-0.087	8
7	33.00	32.58	0.42	0.497	0.594	0.035	0.585	22
8	30.80	30.56	0.24	0.340	0.293	0.004	0.287	6
9	28.00	27.98	0.025	0.497	0.035	0.000	0.034	18
10	32.90	32.21	0.69	0.340	0.856	0.038	0.851	24
11	28.70	29.01	-0.31	0.211	-0.357	0.003	-0.349	30
12	25.20	25.24	-0.045	0.340	-0.056	0.000	-0.055	3
13	30.10	30.44	-0.34	0.211	-0.382	0.004	-0.374	9
14	27.60	27.41	0.19	0.113	0.202	0.001	0.197	31
15	23.10	23.82	-0.72	0.211	-0.818	0.018	-0.812	23
16	28.00	28.31	-0.31	0.340	-0.381	0.007	-0.373	5
17	24.80	25.46	-0.66	0.211	-0.749	0.015	-0.741	27
18	22.00	22.04	-0.039	0.340	-0.049	0.000	-0.048	19
19	32.10	32.26	-0.16	0.497	-0.234	0.005	-0.229	11
20	28.00	28.24	-0.24	0.340	-0.294	0.004	-0.288	28
21	23.70	23.64	0.061	0.497	0.087	0.001	0.085	14
22	29.70	29.90	-0.20	0.340	-0.255	0.003	-0.249	32
23	26.60	26.05	0.55	0.211	0.624	0.010	0.615	10
24	21.30	21.63	-0.33	0.340	-0.410	0.009	-0.402	2
25	27.60	27.19	0.41	0.497	0.582	0.033	0.573	25
26	22.70	23.51	-0.81	0.340	-1.013	0.053	-1.014	16
27	20.00	19.27	0.73	0.497	1.045	0.108	1.048	12
28	29.40	27.41	1.99	0.113	2.134	0.058	2.341	17
29	26.70	27.41	-0.71	0.113	-0.764	0.007	-0.757	21
30	28.80	27.41	1.39	0.113	1.490	0.028	1.535	26
31	29.10	27.41	1.69	0.113	1.812	0.042	1.919	15
32	24.60	27.41	-2.81	0.113	-3.018	0.116	-3.853 *	20

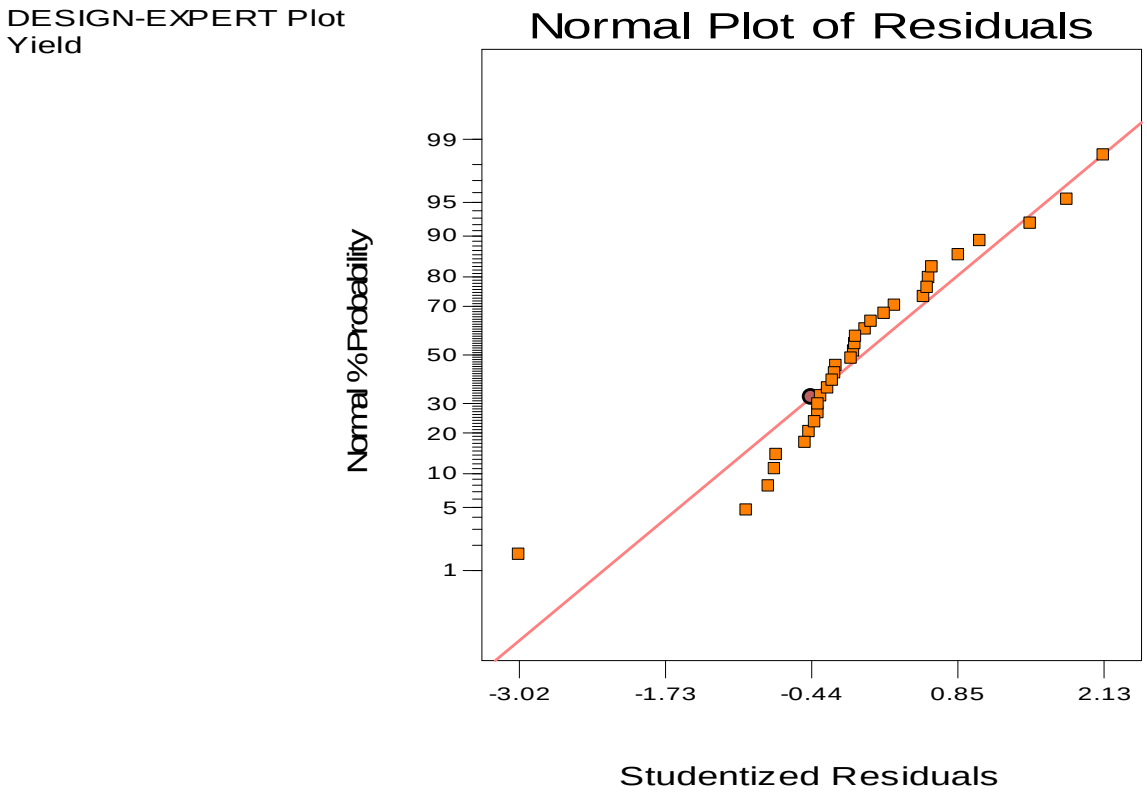


Figure C1. Pulp yield normal plots of residuals

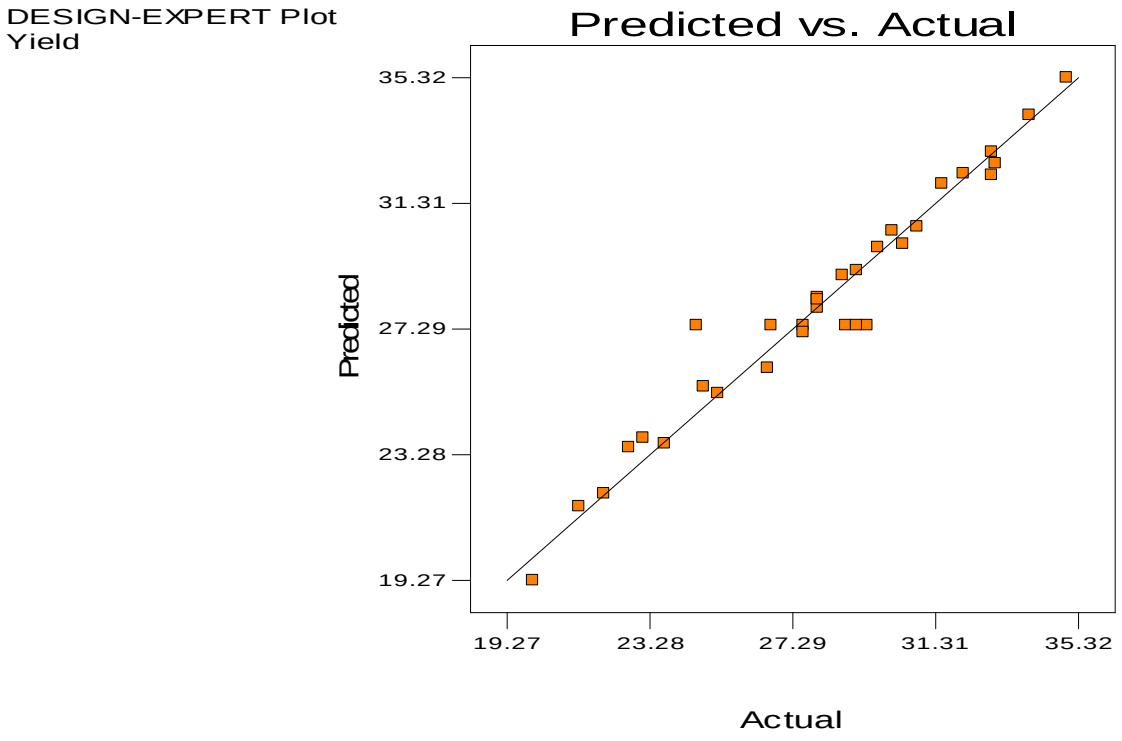
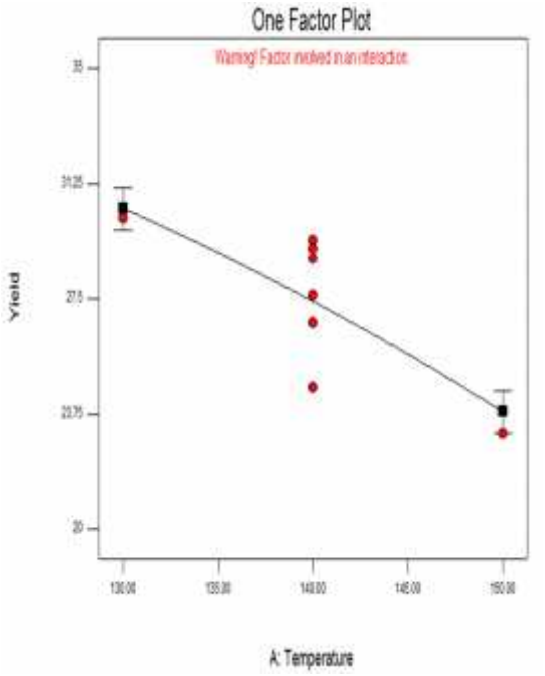
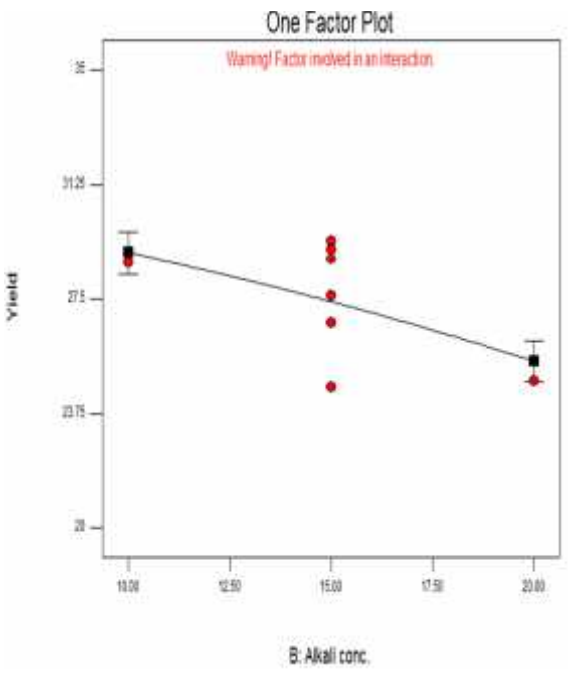


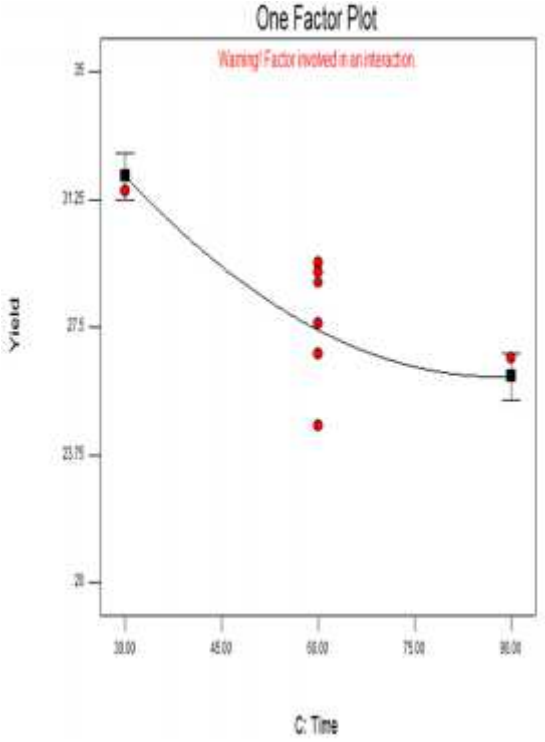
Figure C2. Pulp yield, predicted vs. actual plots


One Factor Plot
Warning! Factor involved in an interaction
Yield
A: Temperature

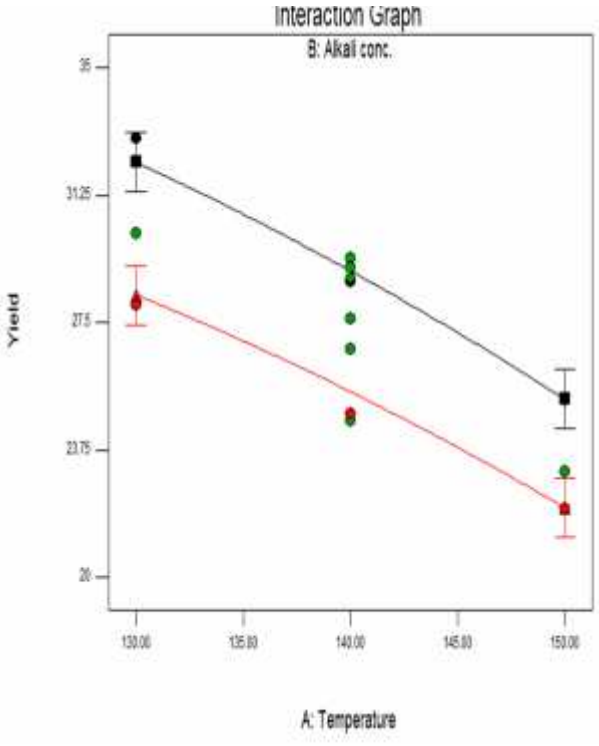
(a)


One Factor Plot
Warning! Factor involved in an interaction
Yield
B: Alkali conc.

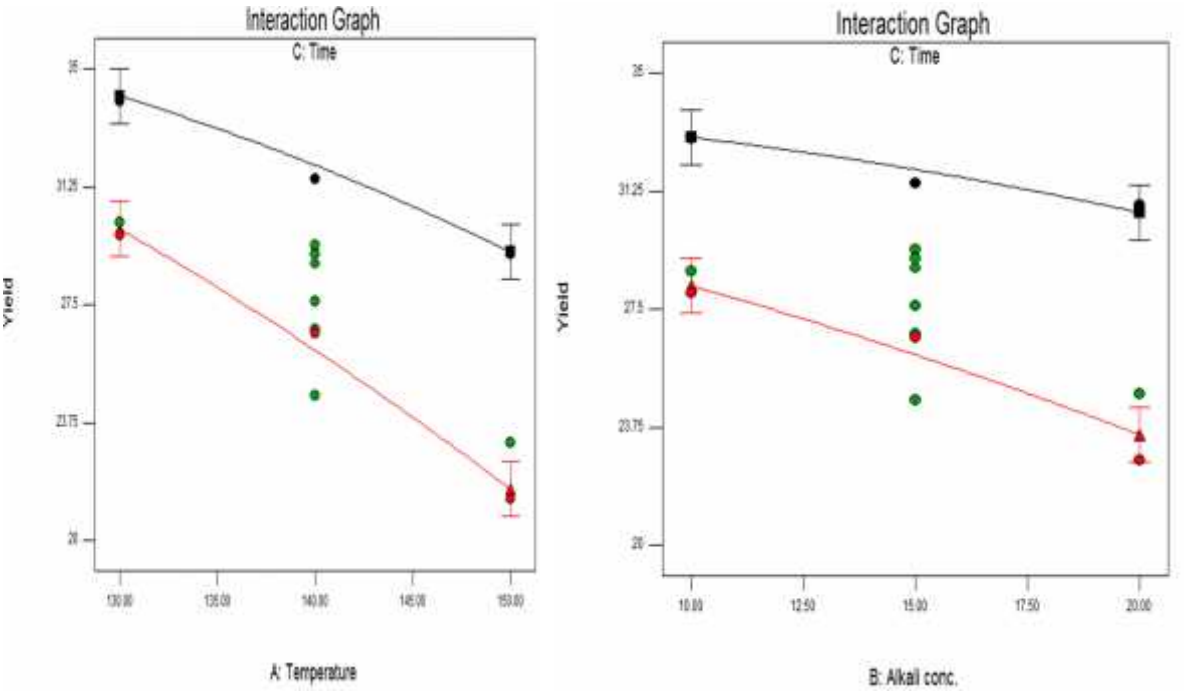
(b)


One Factor Plot
Warning! Factor involved in an interaction
Yield
C: Time

(c)

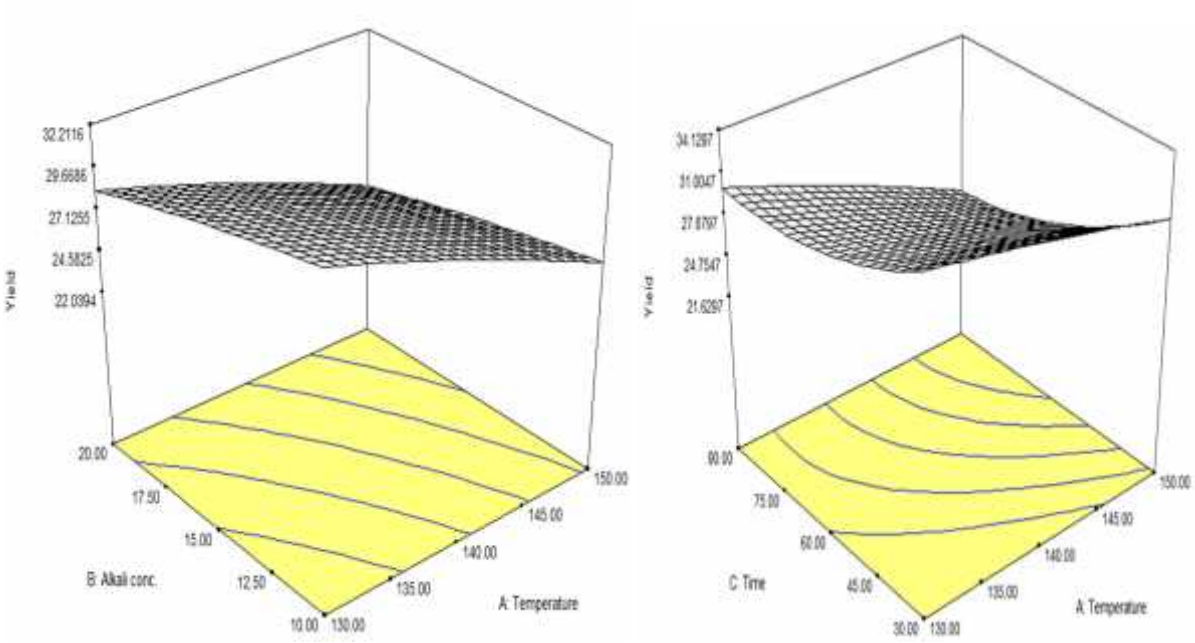

Interaction Graph
B: Alkali conc.
Yield
A: Temperature

(d)

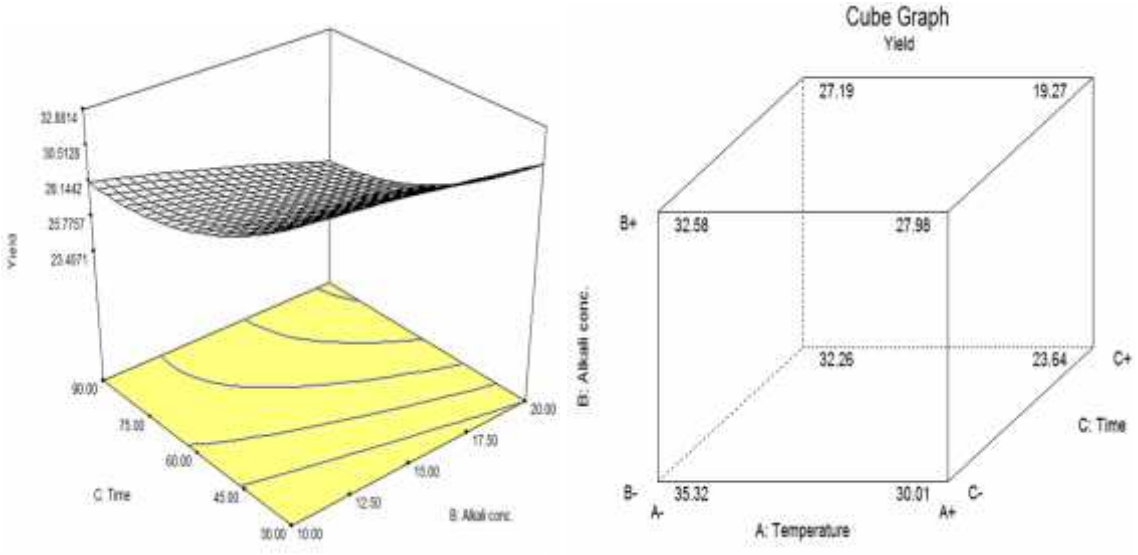


(e) **(f)**

Figure C3. The effects of process variables on pulp yield, ttemperature(a), alkali conc.(b), time(c) and interaction effects, temperature and alkali conc.(d),temperature and time(e), time and alkali conc.(f).



(a) **(b)**



(c) **(d)**

Figure C4. The 3D view of effects of temperature and alkali concentration (a), time and temperature (b), time and alkali concentration(c) on pulp yield and cubic view(d).

Table C2 Diagnostics Case Statistics for kappa number

Standard Order	Actual Value	Predicted Value	Residual	Leverage	Student Residual	Cook's Distance	Outlier t	Run Order
1	23.46	21.81	1.65	0.497	1.375	0.187	1.405	7
2	10.36	13.00	-2.64	0.340	-1.924	0.190	-2.061	4
3	9.40	8.54	0.86	0.497	0.714	0.050	0.706	1
4	21.72	20.28	1.44	0.340	1.049	0.057	1.052	29
5	8.79	11.92	-3.13	0.211	-2.085	0.117	-2.275	13
6	8.40	7.91	0.49	0.340	0.355	0.006	0.348	8
7	20.21	17.83	2.38	0.497	1.983	0.389	2.138	22
8	7.82	9.92	-2.10	0.340	-1.533	0.121	-1.585	6
9	7.44	6.37	1.07	0.497	0.893	0.079	0.889	18
10	13.56	14.43	-0.87	0.340	-0.636	0.021	-0.627	24
11	9.18	8.29	0.89	0.211	0.595	0.009	0.586	30
12	7.24	6.50	0.74	0.340	0.541	0.015	0.532	3
13	11.95	12.90	-0.95	0.211	-0.634	0.011	-0.625	9
14	7.44	7.21	0.23	0.113	0.147	0.000	0.144	31
15	6.29	5.87	0.42	0.211	0.282	0.002	0.276	23

16	7.05	10.46	-3.41	0.340	-2.482	0.317	-2.858	5
17	6.72	5.21	1.51	0.211	1.004	0.027	1.004	27
18	4.03	4.33	-0.30	0.340	-0.216	0.002	-0.211	19
19	8.40	8.85	-0.45	0.497	-0.374	0.014	-0.366	11
20	5.91	5.37	0.54	0.340	0.394	0.008	0.386	28
21	5.53	6.25	-0.72	0.497	-0.598	0.035	-0.589	14
22	7.63	7.32	0.31	0.340	0.228	0.003	0.223	32
23	4.96	4.29	0.67	0.211	0.447	0.005	0.439	10
24	4.40	5.62	-1.22	0.340	-0.887	0.040	-0.882	2
25	4.78	4.88	-0.096	0.497	-0.080	0.001	-0.078	25
26	4.60	2.30	2.30	0.340	1.676	0.144	1.753	16
27	2.73	4.08	-1.35	0.497	-1.125	0.125	-1.132	12
28	6.86	7.21	-0.35	0.113	-0.218	0.001	-0.213	17
29	7.63	7.21	0.42	0.113	0.266	0.001	0.261	21
30	7.24	7.21	0.034	0.113	0.021	0.000	0.021	26
31	8.21	7.21	1.00	0.113	0.631	0.005	0.622	15
32	7.82	7.21	0.61	0.113	0.386	0.002	0.378	20

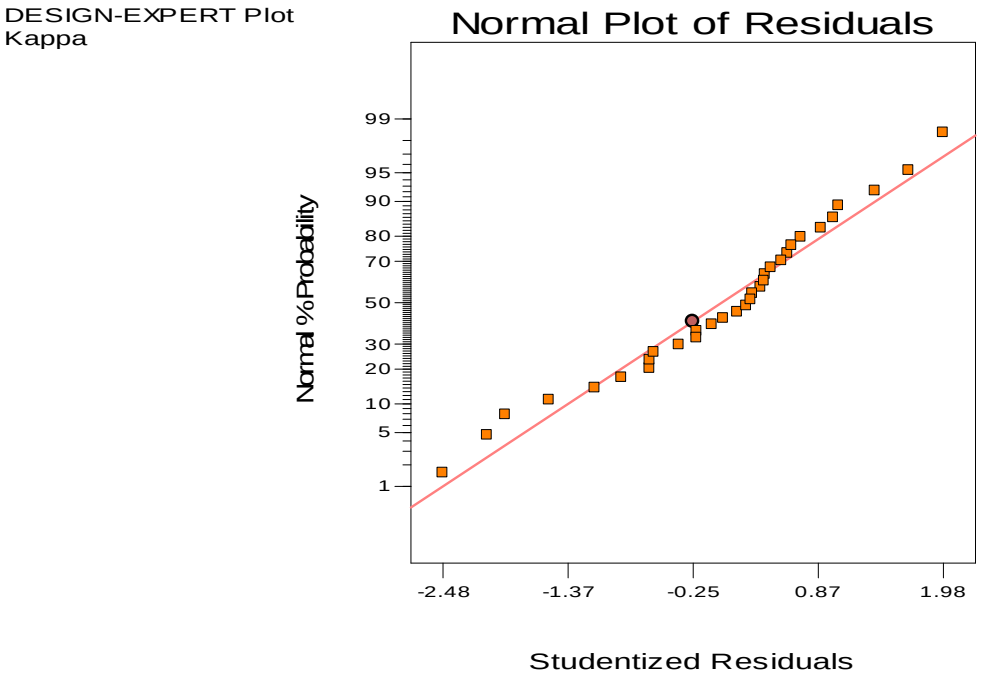


Figure C5: Kappa number normal plots of residuals

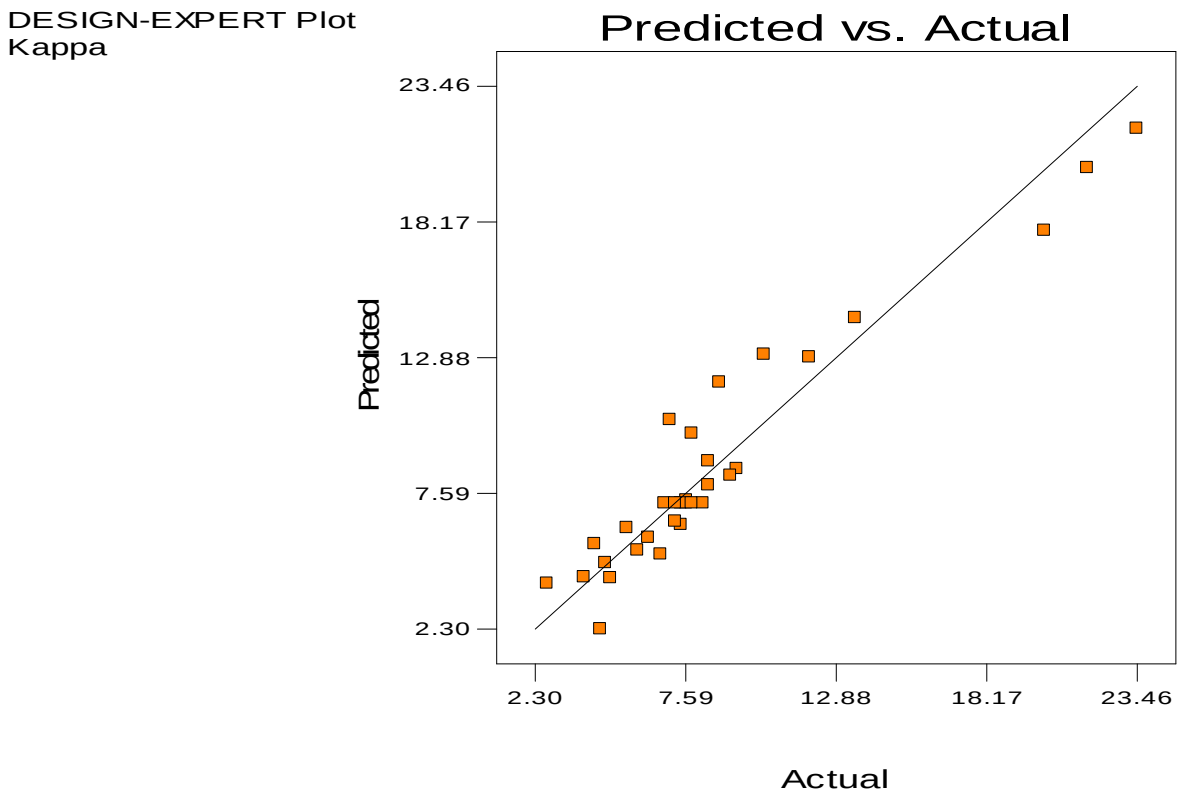
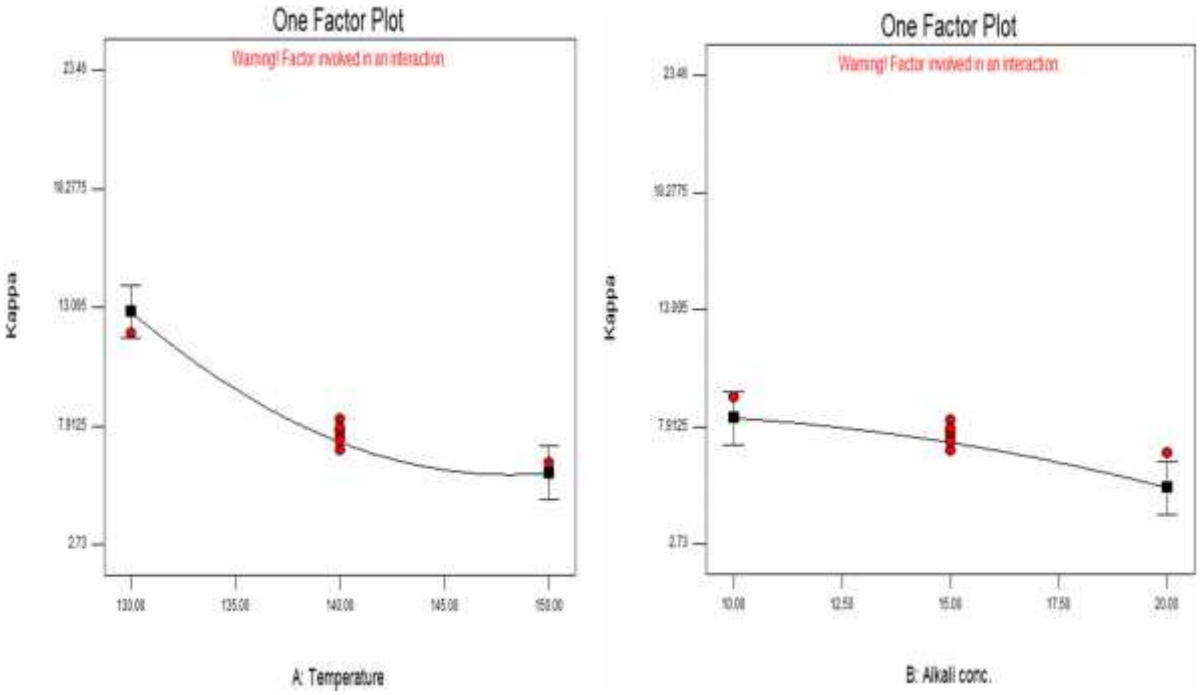
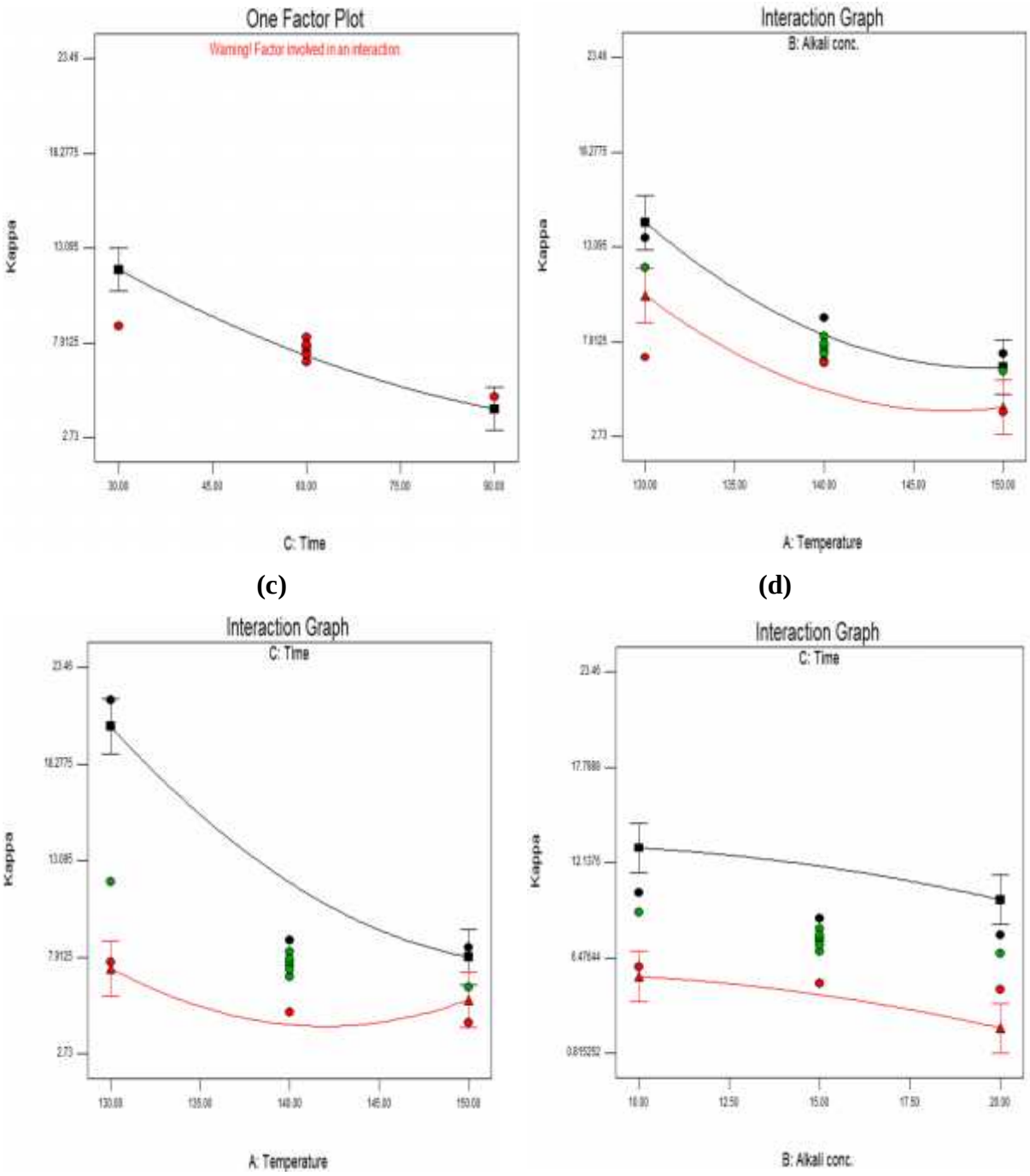


Figure C6: Kappa number, predicted vs. actual plots



(a) **(b)**



(c) **(d)**

(e) **(f)**

Figure C_7: The effects of process variables on kappa number; temperature (a), alkali conc. (b), time(c) and interaction effects; temperature and alkali conc. (d), temperature and time (e), time and alkali conc. (f).

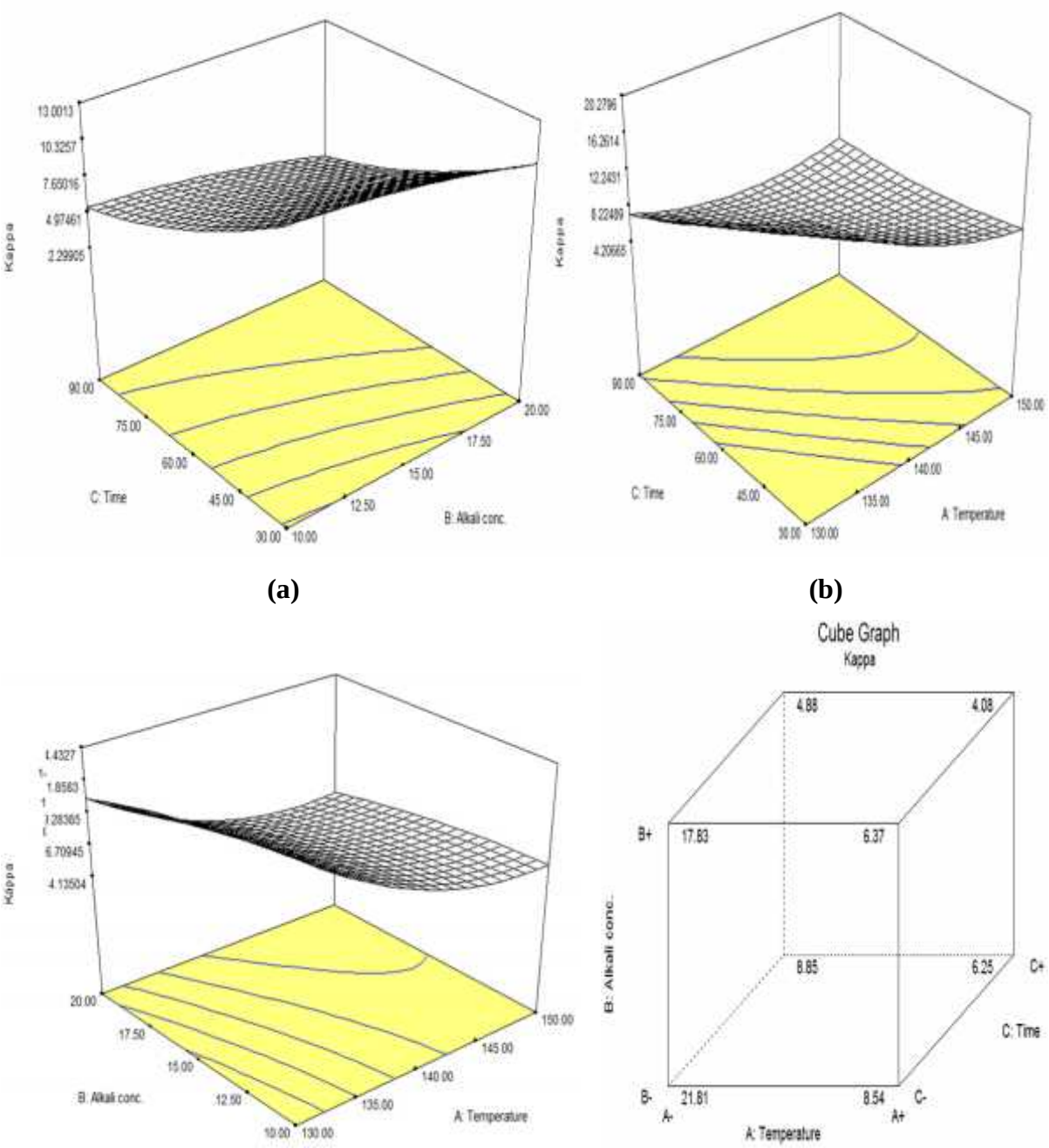


Figure C_8: The 3D view of effects of time and alkali concentration (a), time and temperature (b), temperature and alkali concentration (c) on kappa number and cubic view (d).

Appendix D: Procedures and commonly used terms

PD_1: Procedures for kappa number determination

- Prior to weighing the test samples were conditioned for 20min in the atmosphere near the balance.

- One gram of moisture free pulp specimens which will consume approximately 50% of the potassium permanganate solution were weighed out to the nearest 0.001g. The permanganate consumption should be between 30 and 70%.
- The test specimens were then disintegrated in 500mL distilled water until free of fiber clots and undispersed fiber bundles.
- The disintegrated test specimen was then transferred to a 2000mL reaction beaker and the apparatus was rinse out with enough distilled water to bring the total volume to 795mL. The distilled water was kept at 25.0±0.2°C.
- Then the beaker was placed in a constant temperature bath adjusted to temperature of 25.0±0.2°C during the entire reaction. The suspension was continuously stirred so as to produce a vortex about 25 mm deep but not fast enough to introduce air into the mixture.
- Potassium permanganate solution 50.0±0.1mL and 50mL sulfuric acid was pipette into a 250mL beaker. The mixture was brought to 25°C and quickly added to the disintegrated test specimen, simultaneously a stopwatch has started. The beaker, then rinsed out using not more than 5mL of distilled water, and the washings added to the reaction mixture. The beaker was filled with distilled water to the level of 500ml.
- At the end of exactly 10min, the reaction has stopped by adding 10mL of potassium iodide solution from graduated cylinder.
- Immediately after mixing, but without filtering out the fibers, the free iodine was titrated with sodium theiosulphate solution, and a few drops of starch indicator were added toward the end of the reaction.
- The blank determination was carried out using exactly the same method as above but the pulp was omitted.

PD_2: Reagents preparation for the determination of kappa number

For the determination of kappa number of our pulp the following chemicals of recognized analytical grade and distilled water of equivalent purity were used.

- Sulfuric acid (H_2SO_4) = 2.0 mol/l.

112 ml of sulfuric acid, H_2SO_4, of density 1.84 g/ml, was added with caution to about 600ml of water. Then the solution was allowed to cool and diluted with water to 1liter solution.

- Potassium iodide (KI) = 1 mol/l.

166 g of potassium iodide, KI, was dissolved in a 1000ml volumetric flask and filled up to the mark with water.

- Potassium permanganate ($KMnO_4$) = (0.020±0.001) mol/l.

3.161 g of potassium permanganate, $KMnO_4$, was dissolved in a 1000ml volumetric flask and filled up to the mark with water.

- Sodium theiosulphate ($Na_2S_2O_3$) = (0.200±0.0005) mol/l.

49.65 g of sodium theiosulphate, $Na_2S_2O_3.5H_2O$, was dissolved in a 1 000ml volumetric flask and filled up to the mark with water.

- Starch indicator solution of starch 2 g/l was used (Ethiopian, 2012).

Table D_1 Terms that are commonly used in chemical pulping process

Terms	Definition
Active alkali	NaOH +Na_2S
Effective alkali	NaOH+1/2 Na_2S
Total titratable alkali	NaOH +Na_2S+Na_2CO_3
Sulphidity	Na_2S/Titratable alkali
Causticity	NaOH/ NaOH +Na_2S
Causticizing Efficiency	[NaOH/ (NaOH +Na_2S)] x 100
Reduction efficiency	[Na_2S/ (Na_2SO_4 + Na_2S)] x 100
Over all recovery	Recovery ± black liquor stock/ froth liquor consumed
Green liquor	Liquor obtained after dissolving smelt from furnace in weak liquor from causticizing section
K.No.(Permanganate number)	Number of 0.1 N $KMnO_4$ consumed by 1gm of moisture free pulp
Dilution factor (D.F.)	DF = W-E. W- Water added per ton of mass, E -Water going in pulp per ton of pulp.
Bath Ratio	Liquor to wood ratio (the amount of total liquor per amount of dry wood in a digester)
Consistency	B.D. pulp/ total pulp weight (100 g) i.e. wt. of BD pulp in 100 g of pulp+ water mixture

Appendix E: Glossary

Black liquor

Liquor obtained after washing of cocked pulp which can be further concentrated for burning in furnace to recover chemicals.

Green liquor

The remaining smelt on the bottom of the boiler is again dissolved in water and normally stored in a tank. The now forming solution of sodium carbonate and sodium sulfide is known as green liquor.

White liquor

The main active chemical agents in the Kraft process or an aqueous solution of caustic sodium hydroxide and sodium sulfide, denoted as white liquor.

Kraft Pulp:

A wood pulp produced by chemical pulping of the wood, also called the sulphate process. This removes the lignin from the wood, leaving cellulose fiber used in high quality paper manufacture.

Kraft liner and Kraft Paper

Paper made from wood using the Kraft or sulphate chemical pulping process. It may be bleached or unbleached and produces a strong paper which is used for wrapping and packaging. Kraft liner is used specifically in the production of cardboard boxes (called Packaging Containers in this work), acting as the liners (outer layers). When recovered paper is used this paper is called test liner. The rippled material or corrugating acting as filler between the two liners is called fluting.

Specialty Papers

A diverse group of products that are sold on their performance, technical or appearance attributes. Examples include: filtration products; security and banknotes; decorative papers; abrasives (e.g. sand paper); letterhead; tracing and greaseproof papers.

Tall oil

Oil originates from the nonvolatile fraction of the wood extractives mainly lignin, is removed during evaporation of the black liquor by skimming and used for energy production.