ADDIS ABABA UNIVERSITY
SCHOOL OF GRADUATE STUDIES
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SCHOOL OF CHEMICAL AND BIO-ENGINEERING

PULP YIELD DETERMINATION OF ETHIOPIAN HIGHLAND BAMBOO (YUSHANIA ALPINA) BY SODA/AQ CATALYTIC PULPING

By
Andualem Legesse

June 2021

Addis Ababa, Ethiopia

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A Thesis Submitted to the School of Graduate Studies of Addis Ababa University, Addis Ababa Institute of Technology, in Partial Fulfillment of the Requirement for the Degree of Masters of Science in Chemical Engineering (Process Stream)

Advisor

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Approved by the Examining Board

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Dr. Shimelis Kebede Advisor		
Internal Examiner		
External Examiner		

DECLARATION

I, declare that this thesis entitled ***“Pulp yield determination of Ethiopian highland bamboo (Yushania Alpina) by soda/AQ catalytic pulping”*** conducted as part of Masters of Science degree at the University of Addis Ababa, Addis Ababa Institute of Technology School of Chemical and Bio-Engineering, is my original work and the research has not been submitted to this or any other university by any other person. All sources of material used for this research have been duly acknowledged.

Name of the Candidate	**Signature**	**Date**
Andualem Legesse		

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ACRONYMS

AHQ	Anthrahydroquionone
ANOVA	Analysis of Variance
AQ	Anthraquionone
AS	Alkaline Sulfite
AS-AQ	Alkaline Sulfite Anthraquionone
ASTM	American Society for Testing and Materials
CEH	Chlorination Alkaline Extraction & Hypochlorite
ECF	Elemental Chlorine Free
FAO	Food and Agriculture Organization
GSM	Gram per Square Meter
LCC	Lignin Carbohydrate Complex
NS	Neutral Sulfite
NS-AQ	Neutral Sulfite Anthraquionone
RSM	Response Surface Methodology
TAPPI	Technical Association of Pulp and Paper Industries
TCF	Total Chlorine Free

ABSTRACT

The ever growing demand for pulp, paper and paper products in Ethiopia on one hand and the continually increasing price of woody pulp on the other hand necessitates looking for an alternative source of pulp. The country is endowed with a huge potential of naturally growing bamboo and accessible and suitable land for its possible plantation. Bamboo grows rapidly, is easy to spread, and is highly productive and easy and suitable for harvest. Looking for an environmentally friendly pulping technique for appropriate utilization of such resource is essential. This research investigated the pulp yield of Ethiopian highland bamboo (Yushania alpina) using soda-Anthraquinone (AQ) catalytic pulping and characterized the paper made of such pulp. Samples of bamboo have been harvested from Western Ethiopia Jimma area, air and oven dried, milled, and the bamboo chips were cooked (pulped) using autoclave digester with 10%,15%, and 20% alkali charge; at cooking temperatures of 140, 160 and 180^{0}*C for 120, 150 and 180 minutes. The optimum operating condition that maximizes the pulp yield and minimizes the kappa number was obtained at active alkali concentration of 15.8%, cooking temperature of 158.6°C and cooking time of 156 minutes to be 50.7% and 13.4 respectively. This optimum condition was used to make laboratory scale paper and its mechanical properties were assessed. The tensile, bursting and tearing indexes were found to be 30.2Nm/g, 1.602kPam*^{2}*/g and 12.5mNm*^{2}*/g respectively. These values indicate that quality paper comparable to the one obtained from softwood pulp, with desirable properties can be obtained from Ethiopian highland bamboo with an environmentally friendly pulping procedure of soda – AQ pulping.*

Key Words: Highland Bamboo, Pulp, Pulping, Pulp Yield, Kappa Number, Soda/AQ

CHAPTER ONE

1. Introduction

1.1 Background

Pulp is wood or non-wood based clean and biodegradable wet mass of cellulosic fiber used to make paper. Cellulosic fiber is an elongated, tapering, thick walled cellular unit which is the main structural component of plants. Pulp is made from breaking down the fibrous part of plant materials: trees/woods, crops, grasses, straw or waste paper.

Wood and other plant materials used to make pulp contain three main components: cellulose, hemicellulose and lignin. Cellulose is the main fibrous material desired in paper making, hemicellulose is shorter branched carbohydrate polymer and lignin is a three-dimensional polymer that binds the cellulose fibers together.

Pulping is the process where this bulk structure is broken down mechanically or chemically and fibers are separated and treated to produce pulp. It refers to the process wherein wood or other fibrous raw materials are reduced to a fibrous mass, the purpose being to separate the cellulosic wood fibers by breaking down the wood's inter-cellular lignin glue, without damaging them so that they can then be reformed into a paper sheet in the papermaking process.

Contemporary world pulp largely comes from wood pulp. Deforestation, desertification, extinction of specific species, flooding, drought, climatic change, and disturbances of the water and carbon cycle are all consequences of the continued exploitation of wood for diverse applications, including pulp manufacture (Lobovikov et al., 2007). Therefore, the pulp and paper industry needs environmentally friendly pulping method and fast growing species that can replace itself easily.

Bamboo, a fast-growing biomass, has the potential to play a vital role in the pulp and paper industry in the future, by reducing resource costs while also enhancing product sustainability (Runge et al., 2013). FAO (2005) also reported that bamboo is gaining popularity as a viable alternative to wood in the production of pulp, paper, and paperboard.

Bamboo is an ancient grass that grows in tropical, subtropical, and mild temperate zones. It belongs to the Gramineae family and has about 90 genera with over 1200 species (FAO, 2005). Africa possesses about 40 species on over 1.5 million ha of land and bamboo forest in Ethiopia is the largest in Africa with two naturally occurring (indigenous) species: *Oxytenanthera abyssinica* (lowland bamboo) and *Yushania alpina* (highland bamboo) (Yirgadu et al., 2016). Lowland bamboo spans around 850,000 hectares of land, whereas highland bamboo covers more than 300,000 hectares (Getachew & Wubalem, 2014).

Bamboo, similar to woody plants, has high cellulose content of about 40-60% with fiber length ranging between 2-3mm making it a viable alternative to pulp and paper production (Hidayati et al., 2019). It has several advantages compared to other woody plants: its rapid growth, easy harvest, easy spread, its ease to pulping, its provision of good and high quality bleached pulp fiber for the special type of papers (Hidayati et al., 2019).

Import data obtained from Ethiopian Customs commission on the other hand show that the pulp demand of Ethiopia has been increasing reaching 221.88 million birr (USD 7.39 million) CIF value in 2019 and that of the downstream product (paper) has increased by 33% over the last five years (from 2015 to 2019) (Annex A).

Therefore, harnessing the vast potential of bamboo resource, determining the pulp yield of the bamboo species in the country in general and that of highland bamboo in particular and investigating the paper properties of such pulp is of a paramount importance.

Different pulping methods are applied to wood and non-wood materials worldwide. The pulping methods include mechanical, thermo-mechanical, chemical and semi-chemical. The pulping method employed depends on the type of raw material used and the end use of the pulp.

Chemical pulping that employs soda (NaOH) and sometimes Na_2CO_3 has been widely used in the pulping of bamboo world-wide. The application of anthraquinone (AQ) as a pulping catalyst has been extensively documented in scientific studies and mill applications, and it is known to speed up the delignification process, allowing for a reduction in pulping time, temperature, or chemical charge while increasing pulp yield (Hart & Rudie, 2014).

Soda/AQ catalytic pulping is a pulping method that uses soda and the catalyst AQ in pulp production. It is an environmentally friendly alternative pulping method (Sridach, 2010). This study therefore, has focused on determining pulp yield of *Yushania alpina* by an environmentally friendly pulping process and benefits Ethiopia to make use of its large bamboo resource to meet the country's growing demand of paper.

1.2 Statement of the Problem

The challenge of the world in general and that of Ethiopia in particular nowadays is the consumption of wood for purposes such as firewood, construction and building, grazing and other purposes and consequently following this is deforestation and global environmental change. On the other hand, Ethiopia is committed to developing a green economy which necessitates the use of renewable resources. Despite the fact that wood is renewable, the rate at which it is used does not match the rate at which it is replenished. According to FAO (2005) report, the rate of forest destruction in developing countries is projected to be 13.0 million hectares each year, making wood increasingly scarce and expensive.

Massive plantation and development of easily renewable non-wood species such as bamboo as a supplementary source of fibrous material for pulp and paper making should be the appropriate alternative developing countries like Ethiopia should turn to. The current utilization of bamboo in Ethiopia is limited to fencing, construction, furniture production and making of bamboo based panels. With the huge resource the country possesses (over 1 million hectare of land covered with bamboo corresponding to 7% of world bamboo resource – (FAO ,2005)) investigating the potential of pulp yield of the bamboo in the country and characterization of paper produced from such pulp shall be essential.

The environmentally friendly soda/AQ pulping method has not yet been investigated to examine the pulp and paper production and characterization of such bamboo resource of the country.

1.3 Objective of the Study

1.3.1 General Objective

The purpose of this study is to determine the pulp yield of Ethiopian highland bamboo by soda/AQ catalytic pulping process and characterization of its respective hand sheet paper.

1.3.2 Specific Objectives

- To determine effects of parameters on pulp yield and kappa number
- To determine the optimum operating parameters for maximization of the pulp yield and minimization of the kappa number
- To characterize the paper sheet obtained from the bamboo using soda/AQ pulping

1.4 Scope of the Study

This study has focused on pulp yield determination by soda/AQ catalytic pulping and characterization of pulp and paper made of naturally grown highland bamboo found in Ethiopia. Mature bamboo culms (age 3 to 5) are part of the bamboo plant used for pulping and the slight difference that may occur due to difference in age or silvicultural management of the bamboo plantation is assumed negligible. Among different pulping methods that can be applied on non-wood fibers, soda/AQ catalytic pulping is the method used in this research and other methods of pulping are not considered.

1.5 Significance of the Study

The necessity to lower the environmental effect of pulp mills and the need to improve the yield in wood-to-fiber conversion processes has encouraged innovation in advanced technology for pulp and paper production (Atalla et al., 2004). In this regard, because of the well-established pulping benefit of AQ-pulping catalyst, catalyzed soda pulping has been of attention for decades. Low-level AQ addition to a cook boosts pulping rates, increases pulp yields by protecting carbs against degradation, and lowers chemical recovery bottlenecks. Reducing mill odors, simplifying the recovery process, creating a brighter pulp, and enabling for simplified gasification technology to be used for recovery are all advantages of an AQ-catalyzed soda process. This study therefore contributes to the scientific community by investigating the benefit of AQ catalyzed pulping to pulp yield when applied to Ethiopian highland bamboo. The investigation further benefits the community in large by providing the potential use of such resource for the production of bamboo pulp and paper in an environmentally friendly (sulfur free) process.

CHAPTER TWO

2. Literature Review

2.1 Introduction

The term pulp is used to describe the raw material for the production of paper, paperboard, fiberboard and other cellulosic products. It is a material that has undergone chemical or mechanical action to free the fibers either individually or as fiber bundles from the matrix of cell wall components (cellulose, hemicellulose, lignin and others) of wood and non-wood materials.

Cellulose pulp is mainly produced from wood species and such materials have constituted 90-95% of all pulp produced in the world (Sugesty et al., 2015). The purpose of the pulping is to remove the lignin from the other components so that they can be utilized. The pulping process is the stage where fiber liberation takes place and is mainly done through delignification (removal of lignin).

The extent of delignification of a material varies depending on the pulping method employed. Based on the type or quality of the required pulp or paper to be produced, pulping can be done mechanically, thermo-mechanically, chemi thermo-mechanically, chemically or semi-chemically.

In chemical pulping, the delignification is achieved through addition of chemicals at elevated temperature, making the lignin water soluble. The pulping process is usually named cooking, even though it is unwanted for the cooking chemicals to actually boil. There are a number of chemical pulping processes that employ different chemicals depending on the desired pulp properties and level of delignification (Holm, 2018).

The three most established chemical pulping processes are kraft, sulfite and soda pulping. The active ions vary for the different pulping processes and they are hydroxide and hydrosulfide ions for kraft cooking, bisulfite/sulfite ions for sulfite cooking and sodium hydroxide for soda cooking (Holm, 2018).

2.2 Pulp and Paper Raw Materials

Worldwide cellulose pulp is manufactured from hardwood, softwood and agro residues. The consumption of paper has increased globally by 400 percent in the past 4 decades and about 4 billion trees are cut across the globe for pulp and paper mills on every continent.

This has resulted in global deforestation and forest degradation, causing an ecological and climatic imbalance as well as displacing 300 million people that made forests their home around the world. Because of the severe consequences, major pulp and paper companies around the world are considering not cutting down natural forests any longer, while pulp and paper researchers are investigating non-wood lignocellulosic materials and recyclable fibers to reduce or even disregard the use of wood fibers (Kamoga et al., 2016).

Non-wood fiber materials have a number of advantages as a pulp and papermaking raw material: (1) it is a fast-growing fiber resource with lower lignin content compared to that from wood; (2) low temperatures with lower chemical dosage can be used to produce non-wood pulp; (3) feasible smaller manufacturing factories are achievable processes, resulting in a simplified process; (4) the beating of non-wood pulp fibers is simple to implement (Liu et al., 2018).

China generates more than two-thirds of all non-wood pulp produced worldwide, while non-wood pulp output in Europe, America, and Africa is negligible. Straw, reed, bamboo, and bagasse are the most common non-woods used in papermaking. According to FAO statistics, total "other fiber pulp" production was 19.1 million tons in 2009, whereas total pulp production for paper was 178.1 million tons. Non-wood pulp is classified as "other fiber pulp," while certain data gathering systems may classify recycled pulp as "other fiber pulp" (Leponiemi, 2011).

In some emerging countries, like China and India, non-wood plants provide 70% of the raw materials needed in the pulp industry, and 80% of all non-wood pulp is produced by these two countries. In recent years as a result of increased production of wastepaper pulp and wood pulp, the structure of non-wood pulp in China has changed with the percentage of straw pulp decreasing from 77.2% in 2004 to 44.4% in 2015. Bamboo pulp, on the other hand, has increased dramatically from 2.7% in 2000 to 21.4% in 2015 (Liu et al., 2018).

2.3 World and Ethiopian Bamboo

There are around 1,200 species worldwide, divided into 90 genera. Bamboo occupies more than 36 million hectares (ha) of total land area on the planet. About 43 species and 11 genera bamboo is found in Africa, covering over 1.5 million ha of land. Bamboo species grow in the mountains and highlands of Eastern Africa, as well as the middle lowlands of other African nations. (Teshome K., 2015).

Ethiopia has two naturally growing bamboo species: the highland bamboo (*Yushania alpina*) and the lowland bamboo (*Oxytenanthra abyssinica*) which cover over one million ha of bamboo land (Amsalu & Sisay, 2017). Highland bamboo is estimated to cover over about 20% or 300,000 ha out of which 19,000 ha is planted by farmers. The lowland bamboo covers an area of 700,000-850,000 ha accounting for over 80% of the land covered with bamboo resources. These species are the highest (67%) bamboo resource in Africa (Getachew & Wubalem, 2014).

Highland bamboo is hollow and monopodial (leptomorph-spreading/running and single-stemmed form) while lowland bamboo, the monotypic genus confined to Africa, is solid and sympodial (pachymorph- clumping form) (Getachew & Wubalem, 2014).

Twenty three different bamboo species under seven genera were introduced to Ethiopia since 2007. The seven genera of introduced bamboo species include Bambusa(five species, two of them with two sub varieties each), Dendrocalamus (six species), Gigantochloa (three species), Guadua (two species), Phyllostachys (five species), Schizostachyum (one species) and Thyrsostachys (one species). Some have been tested for their adaptability and growth performance in different locations whereas others are under multiplication (Yirgadu et al., 2016).

2.4 Ethiopian Highland Bamboo Resource

Highland bamboo is named *Yushania alpina* and/or *Arundinaria alpina*. There is inconsistency in naming this species by a number of botanists working in different parts of the world (Yirgadu et al., 2016); because of such problems, the name *Yushania alpina* is used consistently in this paper. It grows in altitudes above sea level (a.s.l) of 2200-4000 m, 1500-2000 mm rain fall and 10-20ºC temperature. It reaches a mean height and base diameter of 12-20 m and 8-20 cm, respectively. It grows with a density of 6000 stems/ha under natural conditions (without management interventions) and having about 51 tone/ha above ground biomass with mean annual increment of about 8.6 tones/ha oven-dry matter and mass flowering occurs between 15-40 years (Getachew & Wubalem, 2014).

The major distribution of highland bamboo by Regional States, Zones and specific localities in Ethiopia is summarized in table below.

Table 1: Major highland bamboo distribution in Ethiopia

Regional State	Zone and Specific locality name
Oromia	Jima (Agaro, Gera)
	Bore/Gujji/HagereSelam
	Western Shewa (Ambo, Tikure-Inchini, Shenen, Jibat Mountain)
	Bale/Harena forest and Shedem Kebele
	Western Arsi (Degaga, Munesa, Shashemene)
Amhara	Awi-zone/Injibara, Gojam/Choke mountains, South Wello (Denkoro forest) and South Gondar (Debere Tabor), Debresina/Wofwasha
Southern Nations, Nationalities and Peoples	Sidama/Agere Selam
	Bonga (Ameya, Wushwush)
	Kefecho Shekicho (Ameya, Baha-Chapa, Gada, Gecha-Masha, Andracha, Chencha/Arbaminch, Mizan Teferi/Kulish, Dawaro)
	Gurage/Indibir-Jembero
Benishangul Gumuze	Assosa (Anbesa Chaka, Kurmuk)
	Kamashi, Mambuk, Mandura, Bambasi, Dibate, Guba, Begi, Demi, Pawe, Sherkole
Gambella	Gambella
Southern Nations, Nationalities and Peoples	Gamo, Gaelebena Hamer Bako,Benchi and Maji, Majina Goldiya
Oromia	West Walega (Begi, Nejo, Gimbi, Guten, Didessa Valley, Kelem) North of Nekemte/Hinde

Amhara North	Gondar (Metema, Metema/Dansha/Humera, Chilga, Wegera)
Tigray	Shire

*source: (Getachew & Wubalem, 2014)

2.5 Bamboo as a Raw Material for Pulping

Bamboo is a very adaptable plant that grows in a variety of climatic and soil conditions. Dwarf bamboo species are just a few centimeters (cm) tall, while medium-sized bamboo species can reach a few meters (m), and giant bamboo species can reach 30 meters (m) tall and have a diameter of up to 30 cm (Lobovikov et al., 2007).

The wide distribution, availability, rapid growth and renewability, easy handling and being source of flexible and useful goods and income for millions of people are only a few of bamboo's environmental and socio-economic benefits.

Bamboo’s growth is more rapid than any other plant on the planet (about 1.2 m height in 24 hours period in Japan), even faster than Eucalyptus species that can only be harvestable annually if managed properly (Getachew & Wubalem, 2014). One growing season of 3-6 months’ time is sufficient for growing to full height and diameter. It matures at about 3-7 years for construction and furniture purposes and it has rotation life that enables it to be harvested in 3-5 years versus 10-50 years rotations for most softwood and hardwood tree species. Bamboo has mean annual biomass increment of 10-30% versus 2-5% for other tree species (Getachew & Wubalem, 2014).

2.5.1 Chemical Constituents of Bamboo

Cellulose, hemicellulose and lignin constitute the main component of bamboo culms while minor constituents consist of resins, tannins, waxes and inorganic salts. The composition varies based on species type, the environment or conditions of growth, the age of the bamboo and the part of the culm. Cellulosic fibers generally occur in the internodes as caps of vascular bundles and in some species also as isolated strands. They constitute to 40-50% of the total culm tissue and 60-70% by weight (Liese, 1985).

Bamboo fibers are generally much longer than those from hardwoods. Across the culm wall, the fiber length often increases starting from the periphery, reaches maximum at about the middle and decreases towards the inner pan. Within one internode, longitudinal variation may exceed 100%: the shortest fibers are always at the nodes, while the longest are in the middle.

Nodes and internodes have different cell wall compositions. Bamboo internodes are made up of 40% to 45% cellulose, 25% lignin, and 25-30% hemicellulose or pentosan on average. In general, the percentage of hemicellulose in young stems and in the lower sections of the culm is higher (van Dam et al., 2018).

2.5.1.1 Cellulose

Cellulose is the main component of the fibrous part of plants. It is the most widely dispersed natural polymer, as well as the most common type of naturally occurring carbon molecules. All woods, straws, and grasses have this as the main component of their cell walls (bamboo) (Galal et al., 2004). Cellulose is a polymeric carbohydrate that is linear polymer of D-glucose units linked by β-1, 4-linked glucose or a polysaccharide containing repeating units of glucose and have strong tendency to form intra and intermolecular hydrogen bonds (Amsalu et al., 2017).

2.5.1.2 Hemicellulose

The main hemicellulose existing in bamboo is a 4-O-methyl-D-glucuronoarabinoxylan linked by a β-(1→4) glycosidic bond, which accounts for approximately 25% of the cell wall components. The acetyl group content covers 6-7% of the total xylan, compared to the 8-17% acetyl group content in hardwoods and 4-9% in softwoods (Vena et al., 2010).

2.5.1.3 Lignin

Lignin is the cementing substance found between fibers and tissues of wood and it is primarily concentrated in the middle lamella region and gives wood tissue its stiffness (Amsalu et al., 2017).

Bamboo lignin consists of higher phenolic hydroxyl group than wood lignin, resulting in an increased reactivity to pulping (Vena et al., 2010).

Extractives together with cellulose, hemicelluloses and lignin solid material of bamboo constitute less than 3%-5% which contribute to properties such as color, smell, decay resistance, density and flammability. Extractives can be either lipophilic or hydrophilic organic compounds, which can be extracted from the samples by using neutral (non-polar), organic solvents or water, respectively (Amsalu et al., 2017).

Silicates, carbonates, sulfates and other inorganic substances including metal ions constitute the ash component (Amsalu et al., 2017). The table below shows the compositions of bamboo as reported by a number of research papers.

Table 2 : Chemical Composition of Bamboo

Component (Property)	Deniz et al., 2017	Amsalu & Sisay, 2017	(Kaur et al., 2016)
Cellulose %	50.1	52.06	46.5-56.2
Alpha Cellulose %	43.3	-	-
Hemicellulose %	-	16.90	-
Holocelullose %	70.5	-	-
Lignin %	24.5	22.47	20-27
Ash %	1.35	5.30	-
Silica	1.03	-	-
Hot water solubility %	6.47	6.80	7.8-12.6
Alcohol-toluene solubility	-	5.60	3.2-5.2

2.5.2 Constituents of Ethiopian Highland Bamboo

A number of researches have been conducted and results on the composition (constituents) of Ethiopian highland bamboo have been reported.

Highland bamboo (*Yushania alpina*) is a hollow, huge and perennial grass. It is a thick walled woody grass with culms of diameter ranging between 12 cm at the base and rising to 15 m from a stout branching rhizome. It grows between 2200 and 4000 meter above sea level. This African alpine is a robust clumping/sympodial/pachymorphic species (Amsalu et al., 2019).

The constituents of a bamboo culm vary with age, position/height of the culm, silvicultural management and others. The significance of their impact on the main constituents (the cellulose, lignin and extractives) is reported by (Amsalu et al., 2019). The following standard conditions were employed to determine the compositions.

Table 3: Standard methods used for chemical analysis of bamboo culms

Composition/Parameter	Standard
Alcohol-benzene solubility	ASTM D 1106-56
Hot-water solubility	Tappi T 207/ASTM D 1106-56
Lignin	ASTM D 1106-56
Hemicellulose	ASTM D 1104-56
Cellulose	Kürschner-Hoffer method
Ash Content	ASTM D 1102-84

The following results have been reported (in % on dry basis) for the constituents of Ethiopian highland bamboos aged 3 to 5 years collected from Western part of Ethiopia particularly Jimma Zone as reported by (Ermias G., 2020) and from Amhara region Injibara Distirict as reported by (Amsalu et al., 2019).

Table 4: Chemical compositions/constituents of highland bamboo

Composition/Constituent	Amsalu et al., (2019)	Ermias G., (2020)
Alcohol-benzene solubility %	-	4.8
Hot-water solubility	-	5.9
Lignin %	24.53-28.28	24.2
Hemicellulose	-	18.1
Cellulose %	49.78 – 51.83	53.5
Ash Content	-	2.2
Extractives %	7.8 – 10.09	-
Cold water solubility	-	4.5

2.5.3 Suitability of Bamboo for Pulp and Paper Making

The high cellulose and hemicellulose constituents of bamboo in general and that of Ethiopian highland bamboo in particular and the morphological characteristics of bamboo make it favorable material for paper making.

Softwoods contain 37.7-42% cellulose, 24-30.9% hemicellulose and 26.4-29.3% lignin constituents which is generally lower than that of bamboo. The cellulose (39.4-56.6%), hemicellulose (27.1-36.7%) and lignin (16.3-25.4%) contents of hardwood are comparable to that of bamboo implying that higher chemical pulp yields can be obtained (Young et al., 2001).

Bamboo is endowed with a long (2.7-4mm) fiber length (Liu et al., 2018) which gives them comparable strength with softwoods and superior to hardwoods (Young et al., 2001) whereas bamboo paper has the same quality with paper made from wood. Its brightness and optical properties remain relatively stable while those papers made from wood may deteriorate over time. The morphological characteristics of bamboo fibers give paper made from bamboo a high tear index (Khalil, 2018). Bamboo fibers show superior characteristics as shown in table 5 when compared with other grass fiber materials making them more viable alternative for paper making.

Table 5: The fiber properties of grass fiber materials

Raw material	**Fiber length (mm)**	**Fiber diameter (µm)**	**Cellulose (%)**	**Hemicellulose (%)**	**Lignin (%)**
Bamboo	2.7-4	15	52-68	15-26	21-31
Reed	1.5-2.5	20	42-50	20-23	22-25
Giant reed	1.2	15	49.8	24-25	-
Bagasse	1.0-1.7	20	55	27-32	18-24
Wheat straw	1.0-1.5	13	29-35	26-32	16-21
Cornstalk	1.0-1.5	16-20	36-38	23-25	18-19

*Source (Liu et al., 2018)

In addition to the constituents of bamboo culms and its morphology, the difficulty in fiber dissociation and the degree of delignification during the pulping process determines its viability for pulping. Assessing the color, capacity of bleaching, drainability, beating performance as well as the adaptability of pulping methods, and the conveniences of stock preparation is important (Liu et al., 2018).

2.6 Chemical Pulping of Bamboo

In the alkaline pulping process, the delignification of gramineous fiber materials like bamboo is easier than that of softwood materials. The following are the key reasons: (1) Such fiber has a loose fibrous structure, low lignin content, and high hemicellulose content. (2) The structure of lignin which is lyophilic and highly soluble and containing a high percentage of phenolic hydroxyl and acid groups makes it easily ionized in alkaline medium. The lignin has a low molecular weight and a high dispersity, which may explain why it is so easy to remove. (3) The low molecular weight hemicellulose in gramineous straw material, which is mainly composed of alkali soluble xylan, degrades easily at cooking temperatures above 100ºC. Consequently following this is a decrease in the concentration of lignin-carbohydrate complex (LCC). This degrades the hemicellulose which leads to the penetration of the cooking liquid and digestion lignin, enhancing the elimination of lignin (Liu et al., 2018).

Alkaline pulping, sulfite pulping and organosolv pulping are the principal chemical pulping methods employed in non-wood pulping.

2.6.1 Alkaline Pulping

In this method, fiber materials are treated with an aqueous solution of an alkaline chemical agent to dissolve much of the lignin to make the pulp. The alkaline pulping process of non-wood can be classified into caustic soda method, oxygen alkali method, lime method, sulfate method and so on, depending on the cooking material employed.

Caustic soda pulping: NaOH and sometimes Na_2CO_3 are the mainly employed chemical agents (cooking liquors) in caustic soda pulping. It is the pulping method that has been widely used in the pulping of non-wood raw material. Active alkali concentration, cooking temperature and cooking time are operating parameters/variables considered. Cooking temperature range of 140–170ºC and alkali concentration of about 16% is generally used. Adding anthraquinone (AQ) gives the likely improvement of the effect of caustic soda pulping. Because AQ has the capacity to expedite the cooking rate while also protecting the carbohydrate, slurry with AQ has a lower kappa number and a higher yield than slurry without AQ under the same cooking conditions (Liu et al., 2018).

When anthraquinone is added to the digester, it oxidizes the carbohydrate's terminal group to form the carboxyl group, which prevents the peeling reaction while also reducing anthraquinone to anthrahydroquinone (AHQ). Following the ionization of Anthrahydroquinone to anthrahydroquinone ion in an alkaline solution, oxanthrone ion would be generated to react with lignin's methylene quinone structure. The oxanthrone ion can then be converted back into anthraquinone, which can then oxidize carbohydrates consecutively (Liu et al., 2018).

Kraft pulping: NaOH and Na_2S are the major ingredients in kraft cooking liquor. The HS^- and S_2^- produced by the hydrolysis of $S_2^-$and the ionization of Na_2S respectively, play a crucial role in the cooking process. The Kraft pulping method is not commonly used for pulping non-wood raw materials. This is mainly due to the fact that Kraft pulping produces pulp that is of inferior strength than that of other alkaline pulping (soda pulping) (Liu et al., 2018).

Alkaline-oxygen (Oxygen soda/AQ) pulping: - This pulping method is an environmentally friendly pulping, that is commonly employed in straw pulping process. During the cooking process, the method uses the synergistic action of oxygen and alkali to reduce polluted exhaust gases. Pulp of high brightness, excellent physical properties and higher yield is achieved through oxygen-alkaline pulping. As a result, oxygen-alkaline pulping is a promising clean pulping process (Liu et al., 2018). Oxygen alkali cooking uses mechanisms such as the breaking of chains and the dissolution of lignin. In an alkaline environment, free phenolic hydroxyl groups are ionized into negative ions that react with oxygen and produce cyclohexadienone peroxide intermediates. Following this is oxidative degradation that alters lignin structure and causes side chain cleavage. This cascade of structural changes in lignin can increase the hydrophilicity of lignin molecules, resulting in lignin breakdown and disintgration.

2.6.2 Sulfite Pulping

Sulfite is the cooking liquid employed in sulfite pulping process and much of the lignin dissolved, accompanied by the separation of fibers (Liu et al., 2018).

Sulfite (Sulphite) pulping methods can be neutral sulfite (NS) or alkaline sulfite pulping (AS) or acid (bi) sulphite pulping.

In neutral sulfite process (NS), Na_2SO_3 which is made by dissolving SO_2 in Na_2CO_3 solution is the active chemical used. Na_2CO_3 left in cooking liquor functions as buffer lets the pH value of the cooking liquor be kept at 7–8. The yield of neutral sulfite pulping is 8–10% higher than that of alkaline pulping, and the pulp is easier to bleach. NS-AQ pulping is neutral sulfite pulping process where anthraquinone (AQ) is used as additive. The NS-AQ method (neutral sulfite pulp) can produce yield comparable to kraft pulp. However, recycling waste liquid would diminish the rate of lignin removal, lowering the yield and quality of the slurry (Liu et al., 2018).

Alkaline sulfite pulping process employs cooking liquid containing NaOH and Na_2SO_3 at pH range of 10–13.5. One of the advantages of alkaline sulfite process when compared to kraft process is that it produces odorless gas. Adding AQ in alkaline sulfite process (AS-AQ) improves the pulp yield and viscosity (Liu et al., 2018).

Compared to kraft pulps, sulfite pulps have higher yield, lighter color and better bleachability. Sulfite pulp is easier to beat or refine for papermaking application and has a high water retention value (WRV). Use of limited variety of species, especially for acid sulfite pulping; its relatively low pulp strength and some technical and economic problems in chemical recovery are the disadvantages of sulfite pulping.

2.6.3 Organosolv Pulping

In organosolv pulping, organic solvents are employed as cooking liquor for dissolving much of the lignin and separating the fibers into pulp (Liu et al., 2018) .

Based on cooking agents used, the organosolv pulping process of non-wood can be divided into: organic acids solvent (formic acid, acetic acid, and formic acid + acetic acid, and so on), ester organic solvent (eg ethyl acetate),alcohols solvents (methanol, ethanol, n-butanol …), active organic solvents (dimethyl sulfoxide, dioxane, diethanol amine, and so on), compound organic solvent (eg methanol + acetic acid, ethyl acetate + ethanol + acetic acid, and so on) and phenol organic solvents (eg phenol, cresol and mixed cresol).

As in the sulfate and alkaline sulfite pulping, the β-ether bond fracture is more essential than that of α-ether bond fracture during the alkaline organic solvent cooking reactions (Liu et al., 2018).

2.7 Soda/AQ Pulping

The growing demand for paper has increased the demand for non-wood pulp as a low-cost papermaking raw material. Bamboo is one of the greatest choices for pulping because of its excellent qualities. The ease of pulping, fiber length and diameter comparable to softwood fiber, the yield of usable pulp, the availability of the bamboo resource in the country and thus lower collection costs, its composition (higher cellulose and hemicellulose content and lower lignin content), and so on are some of the suitable properties (Sridach, 2010).

On the other hand, growing environmental concerns have prompted the development of environmentally friendly alternative pulping processes (Sridach, 2010). Among these alternative pulping processes, Soda-AQ process offers the advantage of sulfur free pulping and elimination of the air pollution associated with Kraft process.

The addition of AQ accelerates the removal of lignin, increases the pulp yield and viscosity, lowers the consumption of the active alkali, decreases the kappa number, improves pulp whiteness showing that the slurry has a better performance (Liu et al., 2018).

A number of researches on pulping of bamboo have been conducted. Amsalu & Sisay, (2017) conducted a study on Soda-AQ pulping of Oxytenanthera abyssinica to determine chemical composition of the bamboo, optimum pulping conditions and characteristics of bamboo paper by Soda-AQ pulping. The cellulose content of the bamboo was determined to be 52.06%, the maximum pulp yield of 52.24% and minimum kappa number of 12.16 at alkali concentration of 20%, cooking temperature of 150 ºC and cooking time of 180 min. The mechanical properties of the paper made of such pulp shows that tensile index, bursting index, and tearing index were 33.58 Nm/g, 1.303kPa.m^2/g, and 11.5mN.m^2/g respectively.

Ermias G. (2020), in his research entitled “optimization and modeling of ethanol-alkali pulping process of bamboo (*Yushinia alpina*) by response surface methodology”, determined the optimum process variables at a cooking temperature of 162.30ºC, cooking time of 180 min, ethanol concentration of 60% and alkali concentration of 18.03% producing maximum pulp yield of 53.56% and brightness of 63.85%, and minimum kappa number of 11.65. The paper provided tensile index, burst index and tear index of 48.25 kN m/ kg, 2.95 kPa m^2 and 2.80 N m^2 /kg, respectively.

Yalewu et al., (2021) conducted a study to determine the Morphological, chemical, and physical characteristics of the Ethiopian highland (*Yushania alpina*) bamboo. Determining how much pulp may be obtained through soda-AQ catalyzed pulping of *Yushinia alpina* and characterization of paper made of such pulp is not yet investigated.

2.7.1 Bamboo Pulp and Paper Making Process

Industrially, bamboo pulp and paper processing can be summarized into materials preparation, pulp cooking, pulp washing and screening, pulp bleaching and paper sheet formation.

i. **Materials preparation:** involves bamboo cutting from plantation followed by storage as may be necessary due to its seasonality, cutting bamboo bundles into bamboo chips by bamboo cutter to (10-25mm) length, screening the chips – the long bamboo shall be returned to bamboo cutter and the sawdust shall be utilized as waste and cooked in the boiler. Material preparation also includes bamboo chips washing to reduce wearing, scaling and silicon interference.

ii. **Bamboo pulp cooking**: The qualified bamboo chips pass through the conveyor and enter into the pulper. Black liquor is poured from the bottom of the vertical pulper, the bamboo chips are presoak and air is removed from the bamboo. The pulper is heated with steam and the temperature is raised to cooking temperature (up to 145ºC) and maintained in a batch cooker (digester). The contemporary technology also employs horizontal pulper.

iii. **Pulp Washing and Screening**: Bamboo pulp washing uses equipment such as vacuum washer, horizontal belt filter and pressure pulp washer. Pulp cooking produces crude residues (knots that are not cooked sufficiently) which are removed by screening and cleaning. Multi-section countercurrent washing is utilized to extract black liquor efficiently and guarantee the alkali recovery.

iv. **Pulp Bleaching**: Bleaching is used to improve the pulp whiteness and keep the strength of bamboo pulp. ECF (Elemental Chlorine Free), TCF (Total Chlorine Free), CEH

(Chlorination, alkaline Extraction and Hypochlorite) are some of the bleaching methods employed.

v. **Paper Making or sheet formation**: sheet of paper is made of the bleached (or unbleached) pulp in paper mill.

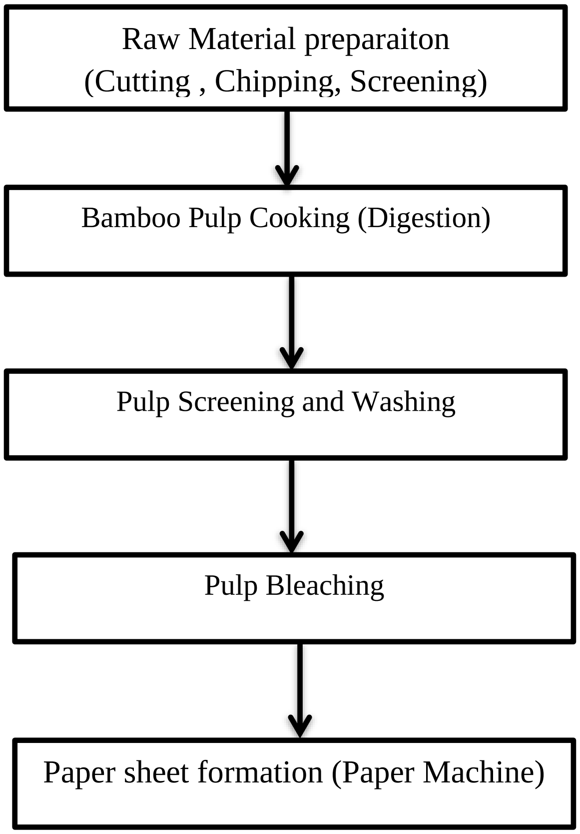


Figure 1: Bamboo pulp and paper making process

2.8 Factors Affecting Pulp Yield

Pulp yield is defined as the "weight of oven-dry wood fibers produced from unit weight of oven dry wood" (Liu et al., 2018). Making of pulp and pulp yield can be affected by a number of factors. These include: Cooking liquor concentration, ratio of cooking liquor to material, cooking time and cooking temperature (Liu et al., 2018).

Cooking liquor concentration:-The higher the concentration of the cooking liquor, the greater the amount of cooking liquor that reacts with lignin, but the use of excess cooking liquor is not good because it will cause the cellulose to degrade (Hasibuan et al., 2020). Cooking liquor concentration of 10 to 20% is employed in research undertaking to investigate the effect of soda-AQ pulping conditions on Semantan bamboo - Gigantochloa scortechinii (Pemulpaan et al., 2013). Amsalu & Sisay (2017) used cooking liquor concentrations of 15%, 20% and 25% in their research entitled Soda-AQ pulping of Oxytenanthera Abyssinica. Active alkali concentrations of 16%, 18% and 20% on oven dry (o.d) basis are used by Jahan et al., (2015).

Ratio of cooking liquor to material: The ratio of cooking liquor to material must be sufficient so that the lignin fragments are perfect in the degradation process and can dissolve completely in the cooking liquor. Ratios that are too small can cause re-deposition of lignin and hence increase the Kappa number (decreasing the quality of the pulp) (Hasibuan et al., 2020). A number of researches conducted on bamboo pulping used cooking liquor to material ratio of 1:4 (Md Sarwar Jahan, 2015; Osman et al., 2020; Pemulpaan et al., 2013; Vena et al., 2010).

Cooking Time: Longer cooking time shall increase the lignin hydrolysis reaction. However, cooking time that is too long will cause cellulose to hydrolyze, thus reducing the quality of the pulp. Cooking time of 60 and 120 to 220 minutes is commonly used in pulping of bamboo as in (Ermias G., 2020; Vena et al., 2010; Amsalu & Sisay, 2017)(Aklilu, 2020)(Aklilu, 2020)(Aklilu, 2020)(Aklilu, 2020)

Cooking Temperature: Cooking temperature is related to the reaction rate which increases the rate of delignification (removal of lignin), but high cooking temperature causes degradation of cellulose. Therefore optimum temperature range needs to be employed. Liu et al., (2018) described a temperature range of 140ºC to 170ºC for non-wood pulping. A number of researches have been conducted at temperature ranges very close to this. Cooking temperature of 150ºC to 170ºC is used on bamboo pulping by (Osman et al., 2020; Pemulpaan et al., 2013). Ermias G., (2020) used cooking temperatures of 140ºC, 160ºC, 180ºC and 220ºC on his study on highland bamboo.

2.9 Kappa Number

Kappa number is an index used by the pulp and paper industry to describe the residual lignin content in unbleached pulp. It gives information on the pulp bleaching process. A lower kappa number means the pulp requires fewer bleaching chemicals while preserving sufficient pulp strength throughout the delignification (lignin removal) process. A higher kappa number implies that the pulp requires more bleaching chemicals.

To predict the amount of bleaching chemicals to be used, determining the kappa number (lignin content) is essential. The lignin content of hard cooked pulp is high, whereas the lignin content of soft cooked pulp is low. Therefore, hard cooked pulp requires more bleaching chemicals to obtain the necessary brightness than soft cooked pulp.

CHAPTER THREE

3. Materials and Methods

3.1 Bamboo pulping, Yield and Kappa number determination

3.1.1 Raw Material Characterization

Materials

i) Apparatus

Oven, Wiley mill, sieve (40 and 60 mesh), digital weighing balance, Erlenmeyer flask, heating mantle, vacuum suction, measuring cylinder, desiccator, water bath, cellulose extraction thimble, furnace, silica crucible, tong, cotton, beaker, glove, plastic bags were used

ii) Reagents

Ethanol, toluene, nitric acid, sulfuric acid, distilled water were used as reagents

iii) Method

Highland bamboo culms (stems) of 3 and above years old were harvested from Western Ethiopia, Jimma area and air dried for two weeks. The culms were then cut into small strips with knife surgically and placed in a mill and were ground. Ground culms were then put in a shaker with sieves to pass through a No. 40 mesh (425-µm) sieve and be retained on a No. 60 mesh (250-µm) sieve. The resulting material is placed in plastic bags, labeled (coded) and made ready for chemical analysis.

Cellulose, Hemicellulose, Lignin and Ash contents determination were conducted following ethanol-toluene (because of health, safety and regulatory concerns associated with the use of benzene, toluene is used instead) extractive test as per American Society for Testing and Materials (ASTM) shown below.

Table 6: Standard test methods used for raw material characterization

Property	**Standard**
Ethanol-toluene extractive	ASTM D 1107-56
Cellulose	Kürschner-Hoffer
Hemicellulose	ASTM D 1104-56
Lignin	ASTM D 1106-56
Ash Content	ASTM D 1102-84

3.1.2 Pulping of Bamboo

Materials

i) Apparatus

Digital weighing balance, tong , measuring cylinder, volumetric flask, 500ml Erlenmeyer flask, aluminum foil, electrical autoclave, stopwatch, glove, 10 mesh(2mm) and 100 mesh (150µm) sieve, oven were employed.

ii) Reagents

Caustic soda (NaOH), anthraquionone (AQ), distilled water, tap water were reagents used

iii) Method

Bamboo chips milled with hammer mill was cooked in an electrical autoclave with the following pulping (cooking) conditions.

Table 7: Pulping conditions for soda-AQ pulping of Yushania alpina bamboo chips

Pulping Conditions	Values
Bamboo chips to cooking liquor ratio	1:4 (w/v) , 1gm oven dry (o.d) bamboo to 4 ml liquor
Alkali concentration	10%, 15%, 20%,
Cooking temperature	140ºC , 160ºC , 180ºC
Cooking time	120, 150 and 180 minutes
Anthraquionone (AQ)	0.1% on bamboo o.d basis

The active alkali (the soda cooking liquor) was prepared from a standard concentrated solution of sodium hydroxide by dilution with distilled water. Then at each run, 30 gram (gm) of oven dry (o.d) bamboo milled chips were cooked in a 500ml Erlenmeyer flask within an electrical autoclave at the cooking temperatures. At the end of each cooking, the resulting cook was thoroughly washed with tap water on 10 mesh (2mm) and 100 mesh (150µm) sieve as in (Vena et al., 2010). The pulp was then oven dried at 105ºC until constant weight is achieved and pulp yield and yield rejects were determined.

3.1.3 Design of Experiments

The potential independent variables (factors) affecting the pulp yield were identified to be the alkali concentration, the cooking temperature and the cooking time. It is assumed that all variables are significant.

As per the desired objective of finding the optimum operating conditions to maximize pulp yield and in parallel minimize the kappa number, Response Surface Methodology (RSM) is employed as it is mainly helpful in optimizing a process.

The data analysis in this experiment is performed using DESIGN EXPERT® 13 (file version 13.0.1.0). A randomized Box-Behnken design type with seventeen (17) runs is performed.

Table 8: Box-Behnken design of the experimental run

	Factor 1	Factor 2	Factor 3
Run	A:Alkili Concentration	B:Temperature	C:Time
	%	ºC	min
1	15	160	150
2	15	160	150
3	10	180	150
4	15	140	120
5	20	140	150
6	15	160	150
7	15	180	180
8	10	160	180
9	10	160	120
10	10	140	150
11	20	180	150
12	15	140	180
13	15	160	150
14	15	160	150
15	20	160	180
16	20	160	120
17	15	180	120

3.1.4 Pulp yield and Kappa number determination

Pulp yield is determined gravimetrically after each run using the formula:

$$Yield\ \% = \frac{Mass\ of\ pulp\ after\ cooking}{Mass\ of\ raw\ material\ before\ cooking} * 100 \qquad \text{i}$$

Kappa number is defined as the volume of 0.02 mole/L (0.1 Normality) potassium permanganate solution consumed by one gram of moisture free pulp in acidic medium. In this experiment, pulp kappa number was determined using spectrophotometric method by direct measurement of the permanganate concentrations in pulp-permanganate reaction solution as detailed in (Zhu, 2014). Strong acidification using sulfuric acid is employed to prevent the precipitation of MnO_2 and hence eliminating spectral interference.

Mathematically the pulp kappa number is defined as

$$K = {}^{p}/_{w} \qquad \text{ii}$$

Where K is kappa number, p is the amount of 0.02 mole/L (0.1N) permanganate solution actually consumed by the test sample in milliliter (ml), and w is the mass of the moisture-free sample in grams.

At a given wavelength, permanganate absorption spectral intensities measured at the beginning and end of the permanganate-pulp reaction can be used to calculate the Kappa number consequently eliminating the measurement of absolute values as in the following equation.

$$K = \frac{a}{w}\left(1 - \frac{A_e}{A_o}\right) \qquad \text{iii}$$

Where K is the kappa number, a is the initial volume of 0.02 mol/L permanganate in the blank solution, A_e and A_o are the permanganate absorbances (or spectral intensities) in the beginning blank solution and at the end of the oxidation reactions in the solutions respectively.

i) Apparatus

A spectrophotometer

ii) Reagents

5 mL standardized 0.02 ± 0.0001 mol/L (0.1 ± 0.0005 N) potassium permanganate solution, 20mL standard sulfuric acid of 2.0 mol/L concentration for each run.

iii) Method

The spectral intensity of a 5 mL standardized 0.02 ± 0.0001 mol/L (0.1 ± 0.0005 N) potassium permanganate blank solution is recorded. A weighted w amount of oven dried pulp of each run is thoroughly mixed with the blank solution for 3 to 5 minutes and the spectral intensity at the end of the reaction is recorded. Finally, the ratio of the absorption spectral intensities at 546nm wavelength measured at the beginning and end of the reaction as in **equation (iii)** is used to directly determine the kappa number without the need for titration. Because lignin and other organic molecules produced in the reaction do not absorb in the region of interest, the spectral intensity at 546nm is due purely to the absorption of potassium permanganate (Zhu, 2014).

3.2 Bamboo Paper Characterization

3.2.1 Materials

i) Apparatus

Beating machine, sheet former, sheet press, disintegrator, freeness tester, tensile, tear and burst strength measuring machines were employed in the analysis.

ii) Reagents

Tap water

iii) Method

Pulp beating: - Obtained pulp sample of 400gm was soaked in water (23 liter) for few hours, beaten in a beating machine for up to 15 minutes and during beating its freeness determined at Ethiopian Pulp and Paper Sh.C laboratory. 200ml of sample was taken from the beating machine and diluted to 1000mL in measuring cylinder. A disintegrator is used to homogenize the suspension, consistency of the stock after dilution was determined and a sheet of desired weight was made.

Sheet formation: - Paper sheet was formed using sheet former connected to vacuum system according to Tappi standards with a pulp suspension of 5% consistency drained through a wire. The sheet was then pressed in sheet presser machine and then dried in an oven at 125ºC for 10 to 15 minutes.

Characterization of paper sheets: - 60, 82 and 142 GSM (gram per square meter) paper were made and tested for strength properties such as tensile strength, tear strength, and burst strength.

Tensile strength: - Tensile strength is the maximum tensile force per unit width that a test piece of paper (paper strip) shall withstand before breaking when applying the load in a direction parallel to the strip. It is measured according to German Standard method by means of a Karl Frank 468 tester (Weinheim–Berkenau). Tensile index is the tensile strength divided by the grammage expressed in (Nm/g).

$$Tensile\ index\ =\ tensile\ strength/grammage \qquad \text{iv}$$

Burst strength: - Burst strength of paper is the maximum uniformly distributed pressure, applied at right angle to its surface that a test piece stands under standardized conditions. This test is extensively used in evaluation of wood pulps (Penttinen, 2012). Bursting strength tester (PN-BSM600) was used to determine the burst strength according to TAPPI Standard test method T 205M. A test piece was placed over a circular elastic diaphragm rigidly clamped at the periphery but free to bulge with diaphragm. The hydraulic fluid pressure increases by pumping at a constant rate to bulge the diaphragm until the test piece ruptures. The bursting strength of the test piece is the maximum value of the applied hydraulic pressure. Burst index is the burst strength divided by the basis weight expressed in kPa m^2/g.

$$Burst\ index\ = bursting\ strength/basis\ weight \qquad \text{v}$$

Tear strength: - Tear strength of paper is the resistance of a paper sheet to tearing force (measured in milli Newton- mN) that the paper is subjected to. The tearing resistance was measured according to Tappi, standard T22M-60 by means of an Elmendorf Tearing Tester. Tear index is the resistance of paper divided by its grammage expressed in (mNm^2/g).

$$Tear\ index = tearing\ strength/basis\ weight \qquad \text{vi}$$

CHAPTER FOUR

4. Results and Discussion

4.1 Bamboo pulping, Yield and Kappa number

4.1.1 Raw material Characterization

The composition (constituents) of the highland bamboo collected from south-western part of Ethiopia (Jimma) locality was determined using the standard methods as in Table 6 for ethanol-toluene extractives, cellulose, hemicellulose, lignin and ash content. The experiment was repeated in three replicates and average of the results was taken.

Table 9: Results on chemical constituents of highland bamboo

Composition/Constituent	Value
Ethanol-toluene extractive %	3.9
Cellulose %	52.2
Hemicellulose	17.3
Lignin %	26.9
Ash Content	2.6

i. Ethanol-toluene extractive

Ethanol-toluene extractives consists mainly of waxes, fats, resins as well as some water soluble substances that are not generally considered part of the cell wall structural component of bamboo. Ethanol-toluene extractive method provides the most complete removal of residual solvent extractable substances in bamboo. The result (3.9%) is lower than that of Oxytenanthera Abyssinica (5.6%) reported by (Amsalu et al., 2017). It is in the range of 3.2 – 5.2 % as reported for Asian bamboo species by (Kaur et al., 2016) and in close agreement with 3.93% for highland Ethiopian bamboo as reported by (Mahelete et al., 2020).

ii. Cellulose

Cellulose is main polysaccharide which forms the skeletal structure of plants and trees. The value 52.2% observed in this experiment is within the range (49.83 – 56.50%) as reported by (Amsalu et al., 2019) for Ethiopian highland bamboo grown in Amhara region and in close agreement with 53.3% reported by (Erimias, 2020).

iii. Hemicellulose

Hemicellulose value of 17.3% observed in this experiment is within the range of 12-33% reported for African bamboo and in close agreement with 16.9% for Oxytenanthera Abyssinica reported by (Amsalu et al., 2017) and 18.1% reported by (Erimias, 2020).

iv. Lignin

Lignin composed of phydroxyphenyl, guaiacyl and syringyl units contributes to the compression strength of woody stems. Lower lignin content is desired for pulp and paper industries (Kaur et al., 2016). Lignin content of 26.9% observed in this experiment is within the range 20.0 – 27.0% reported by (Kaur et al., 2016) and 24.5 – 27.0% reported by (Amsalu et al., 2019). It is in close agreement with 24.2% reported by (Erimias, 2020) and 25.27±1.52% reported by (Mahelete et al., 2020)for Ethiopian highland bamboo.

v. Ash

Ash is the inorganic residue remaining after ignition at high temperature (Song, 2013). Ash content of 2.6% observed in this experiment is lower than that of Oxytenanthera Abyssinica (5.30%) reported by (Amsalu et al., 2017). It is in close agreement with 2.2% reported by (Erimias, 2020) for highland bamboo and B. procera achor.

4.1.2 Bamboo Pulping Experimental Results

The following experimental result for the cooking conditions described in Table 7 is observed.

Table 10: Experimental results of the bamboo pulping

	Factor 1	Factor 2	Factor 3	Response 1	Response 2
Run	A:Alkali Concentration	B:Temperature	C:Time	Pulp Yield	Kappa Number
	%	ºC	min	%	
1	15	160	150	51.22	14.31
2	15	160	150	50.93	14.21
3	10	180	150	33.12	20.38
4	15	140	120	52.25	32.32
5	20	140	150	46.11	25.46
6	15	160	150	51.25	14.42
7	15	180	180	36.06	12.79
8	10	160	180	37.39	17.56
9	10	160	120	46.22	30.11
10	10	140	150	38.21	21.62
11	20	180	150	36.64	13.92
12	15	140	180	48.06	18.46
13	15	160	150	51.23	14.41
14	15	160	150	50.85	14.21
15	20	160	180	42.87	15.61
16	20	160	120	52.14	29.32
17	15	180	120	49.49	25.25

The experimental observation shown was used to examine what effects the parameters: active alkali concentration, cooking temperature and pulping time have on the responses pulp yield and kappa number. To minimize the effect of unexpected variability that may occur due to extraneous factors, the runs were conducted in a randomized order and the design summary is presented in Table 11 below.

Table 11: Design summary

Build Information

File Version	13.0.1.0		
Study Type	Response Surface	**Subtype**	Randomized
Design Type	Box-Behnken	**Runs**	17.00
Design Model	Quadratic	**Blocks**	No Blocks

Factors

Factor	**Name**	**Units**	**Type**	**SubType**	**Min.**	**Max.**	**Mean**	**Std. Dev.**
A	Alkali Concentration	%	Numeric	Continuous	10.00	20.00	15.00	3.54
B	Temperature	ºC	Numeric	Continuous	140.00	180.00	160.00	14.14
C	Time	min	Numeric	Continuous	120.00	180.00	150.00	21.21

Responses

Response	**Name**	**Units**	**Observations**	**Min.**	**Max.**	**Mean**	**Std. Dev.**	**Ratio**
R1	Pulp Yield	%	17.00	33.12	52.25	45.53	6.69	1.58
R2	Kappa Number	-	17.00	12.79	32.32	19.67	6.51	2.53

Pulp Yield: Model Summary Statistics

Source	**Std. Dev.**	**R^2**	**Adjusted R^2**	**Predicted R^2**	**PRESS**	
Linear	5.44	0.4635	0.3397	0.0855	655.20	
2FI	5.98	0.5000	0.2000	-0.7017	1219.19	
Quadratic	**0.1599**	**0.9998**	**0.9994**	**0.9989**	**0.7684**	**Suggested**
Cubic	0.1905	0.9998	0.9992		*	Aliased

*Case(s) with leverage of 1.0000: PRESS statistic not defined.

Kappa Number: Model Summary Statistics

Source	Std. Dev.	R^2	Adjusted R^2	Predicted R^2	PRESS	
Linear	4.36	0.6356	0.5515	0.4414	378.46	
2FI	4.69	0.6759	0.4815	0.1840	552.79	
Quadratic	**0.0798**	**0.9999**	**0.9998**	**0.9998**	**0.1049**	**Suggested**
Cubic	0.1026	0.9999	0.9998		*	Aliased

*Case(s) with leverage of 1.0000: PRESS statistic not defined.

4.1.3 ANOVA and diagnostic plots for pulp yield and kappa number

Table 12: ANOVA for Quadratic model (pulp yield)

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	716.25	9	79.58	3112.73	< 0.0001	significant
A-Alkali Concentration	65.09	1	65.09	2546.00	< 0.0001	
B-Temperature	107.46	1	107.46	4202.96	< 0.0001	
C-Time	159.49	1	159.49	6238.08	< 0.0001	
AB	4.80	1	4.80	187.59	< 0.0001	
AC	0.0484	1	0.0484	1.89	0.2113	
BC	21.34	1	21.34	834.84	< 0.0001	
A^2	217.85	1	217.85	8520.68	< 0.0001	
B^2	122.01	1	122.01	4772.03	< 0.0001	
C^2	2.38	1	2.38	93.13	< 0.0001	
Residual	0.1790	7	0.0256			
Lack of Fit	0.0339	3	0.0113	0.3110	0.8177	not significant
Pure Error	0.1451	4	0.0363			
Cor Total	716.43	16				

Factor coding is **Coded**.

Sum of squares is **Type III - Partial**

The **Model F-value** of 3112.73 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case A, B, C, AB, BC, A^2, B^2, C^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

The **Lack of Fit F-value** of 0.31 implies the Lack of Fit is not significant relative to the pure error. There is an 81.77% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

Table 13: Fit Statistics (pulp yield)

Std. Dev.	0.1599		**R^2**	0.9998
Mean	45.53		**Adjusted R^2**	0.9994
C.V. %	0.3512		**Predicted R^2**	0.9989
			Adeq Precision	156.4594

The R-square indicates how well the model can explain variation of data and the adjusted R-square indicates how much R-square over estimates the variance when another predictor is added in the model. The higher the adjusted R-square is, the better the goodness of fit.

The **Predicted R^2** of 0.9989 is in reasonable agreement with the **Adjusted R^2** of 0.9994; i.e. the difference is less than 0.2.

Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 156.459 indicates an adequate signal. This model can be used to navigate the design space.

Equation in Terms of Coded Factors

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor.

Pulp Yield = $+51.10 +2.85*A -3.66* B + -4.47* C -1.10* AB -0.1100* AC -2.31*BC -7.19*A^2 - 5.38*B^2 +0.7520*C^2$

Table 14: ANOVA for Quadratic model (kappa number)

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	677.44	9	75.27	11832.44	< 0.0001	significant
A-Alkali Concentration	3.59	1	3.59	564.53	< 0.0001	
B-Temperature	81.41	1	81.41	12797.25	< 0.0001	
C-Time	345.58	1	345.58	54324.60	< 0.0001	
AB	26.52	1	26.52	4169.27	< 0.0001	
AC	0.3364	1	0.3364	52.88	0.0002	
BC	0.4900	1	0.4900	77.03	< 0.0001	
A^2	51.26	1	51.26	8057.19	< 0.0001	
B^2	27.25	1	27.25	4283.67	< 0.0001	
C^2	120.47	1	120.47	18937.69	< 0.0001	
Residual	0.0445	7	0.0064			
Lack of Fit	0.0025	3	0.0008	0.0776	0.9688	not significant
Pure Error	0.0421	4	0.0105			
Cor Total	677.49	16				

Factor coding is **Coded**.
Sum of squares is **Type III - Partial**

The **Model F-value** of 11832.44 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case A, B, C, AB, AC, BC, A^2, B^2, C^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

The **Lack of Fit F-value** of 0.08 implies the Lack of Fit is not significant relative to the pure error. There is a 96.88% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

Table 15: Fit Statistics (kappa number)

Std. Dev.	0.0798		**R^2**	0.9999
Mean	19.67		**Adjusted R^2**	0.9998
C.V. %	0.4055		**Predicted R^2**	0.9998
			Adeq Precision	319.1818

The **Predicted R^2** of 0.9998 is in reasonable agreement with the **Adjusted R^2** of 0.9998; i.e. the difference is less than 0.2.

Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 319.182 indicates an adequate signal. This model can be used to navigate the design space.

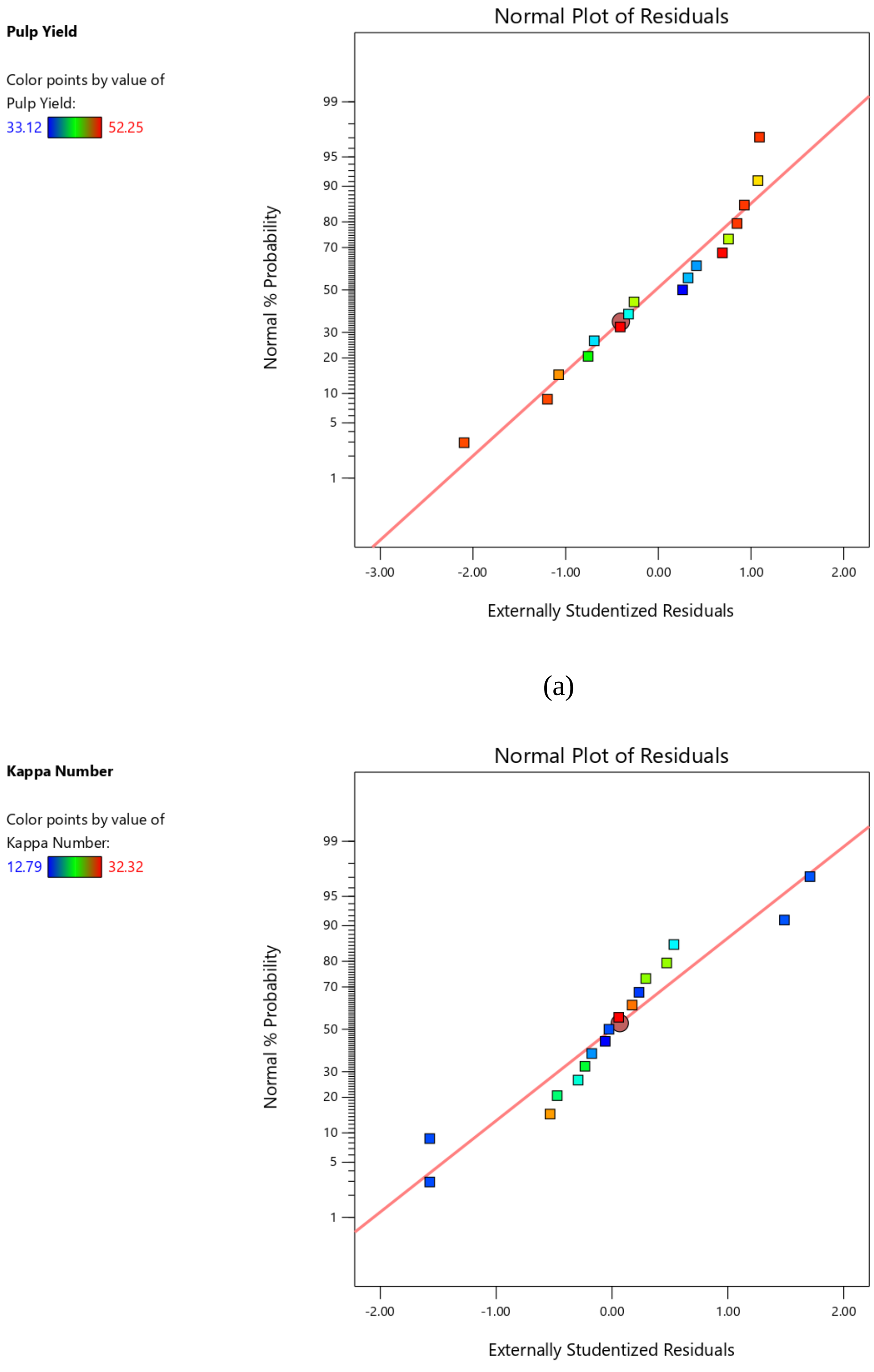
Equation in Terms of Coded Factors

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor.

$\text{Kappa Number} = +14.31 -0.6700*A -3.19*B -6.57*C -2.58*AB -0.2900*AC +0.3500*BC +3.49*A^2 +2.54*B^2 +5.35*C^2$

<u>Diagnostic Plots</u>

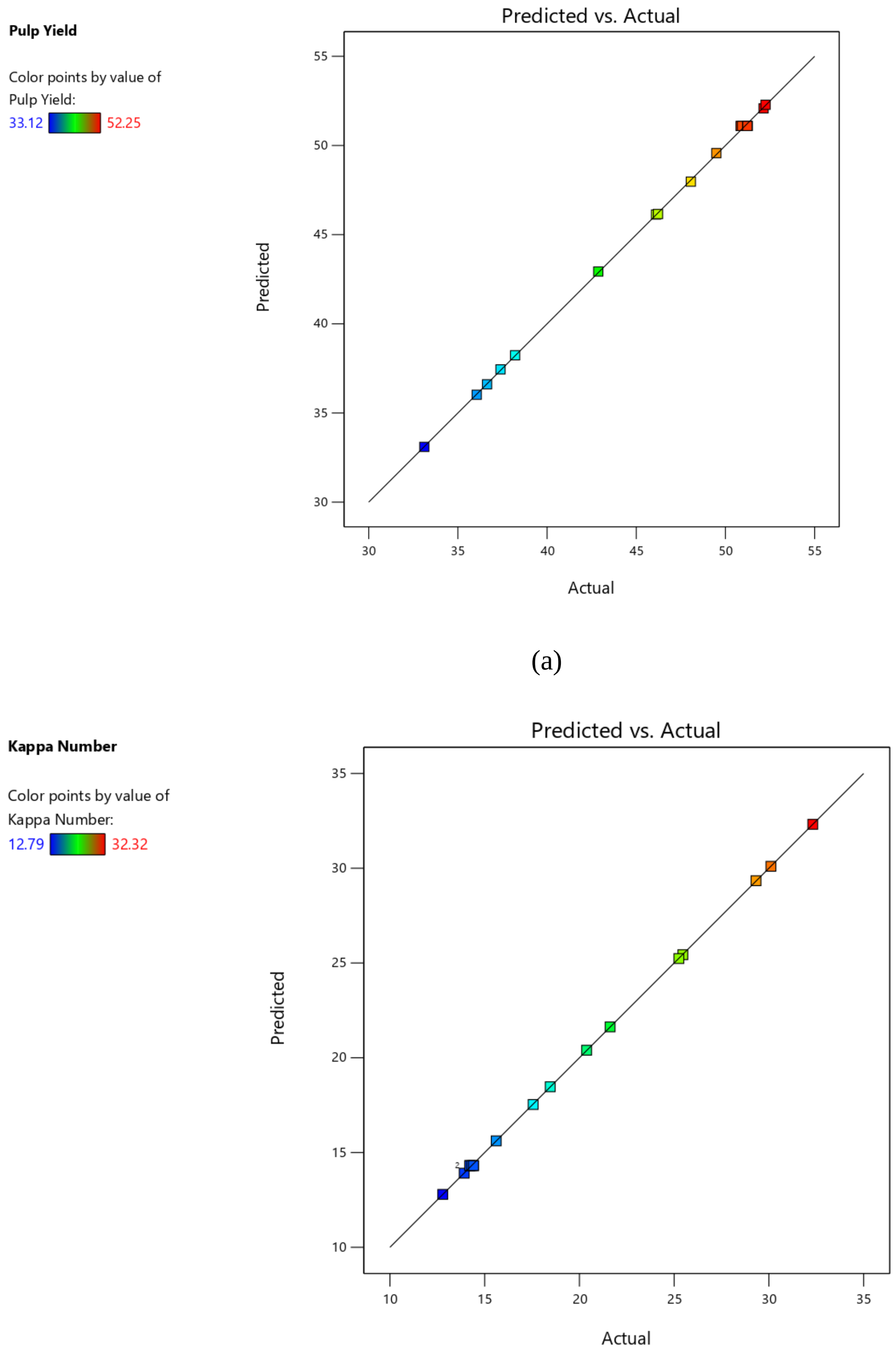
Normal plots of residuals, plots of residuals vs. runs, plots of predicted vs. actual runs and other plots were investigated for pulp yield and kappa number to check for adequacy. Normal plots of residuals for both pulp yield and kappa number and plots of predicted vs. actual runs show that a good correlation exists between experimental and predicted values and the variations between the actually observed and fitted values are minimal. Additional plots are presented in Appendix A.



(a)

(b)

Figure 2: Normal plot of residuals for (a) pulp yield and (b) kappa number



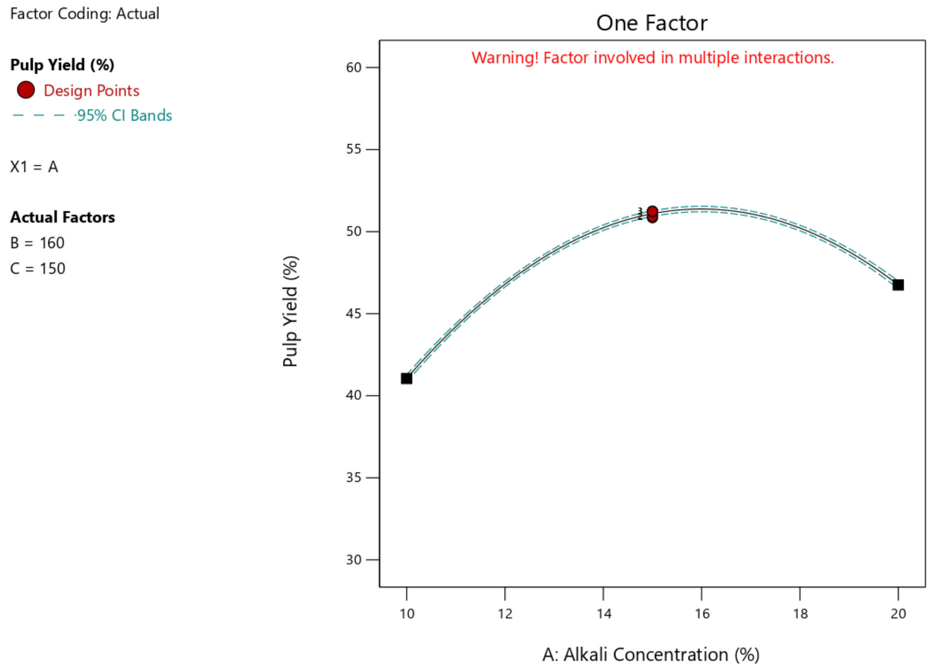
(a)

(b)

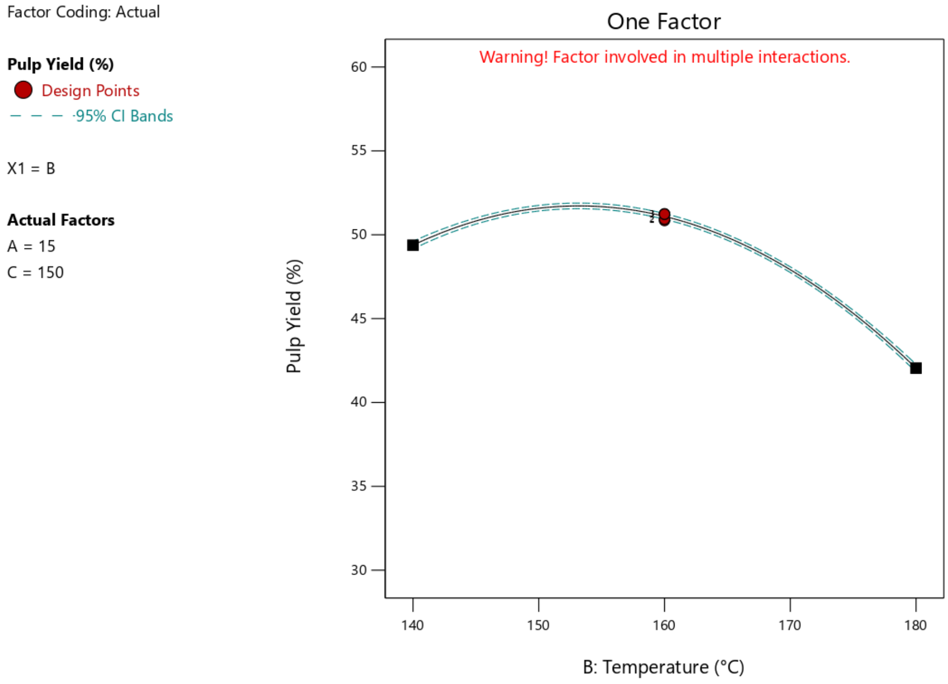
Figure 3: Predicted vs. Actual plots for (a) Pulp Yield and (b) Kappa Number

4.1.4 One factor and Interaction Effects

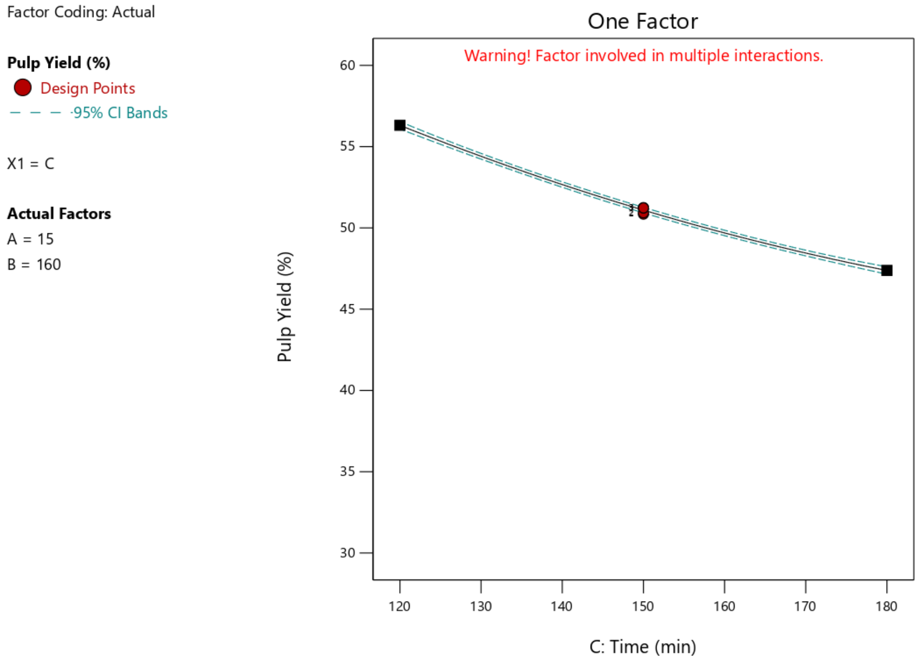
<u>Pulp yield</u>



(a)



(b)

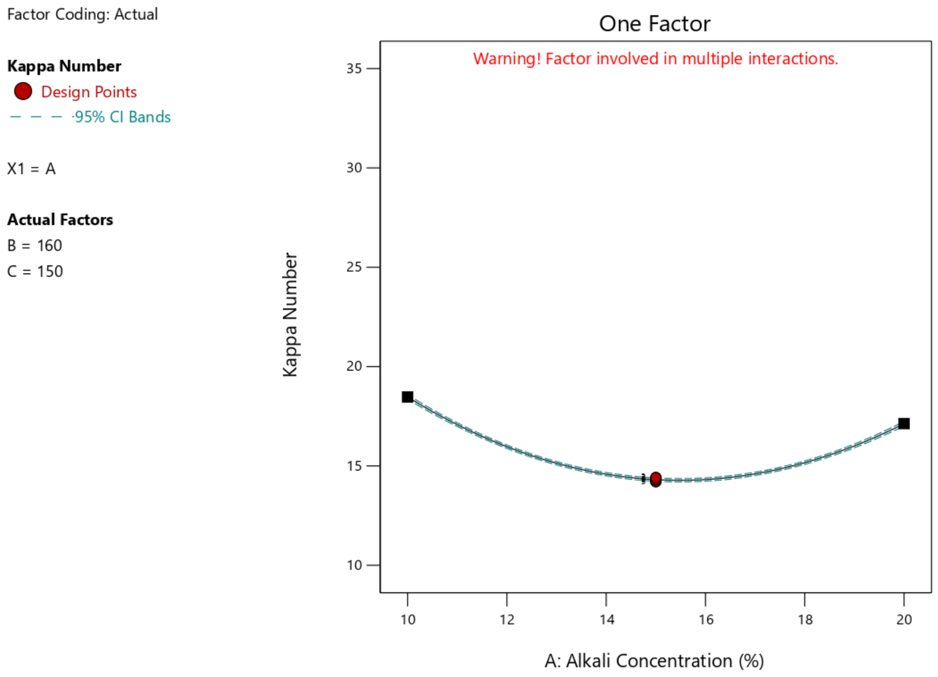


(c)

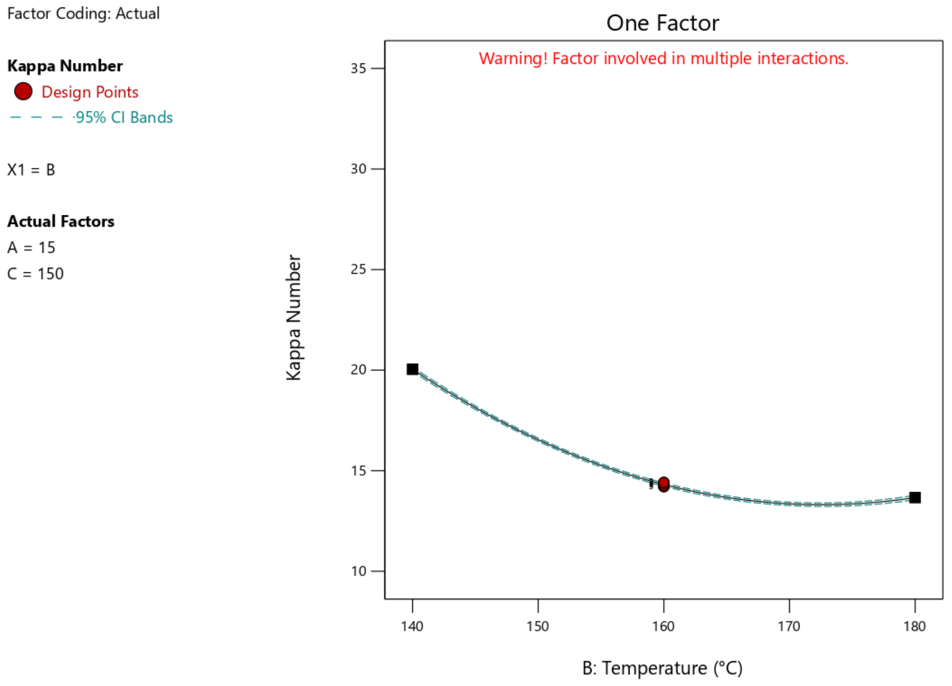
Figure 4: One factor effect on pulp yield (a) alkali concentration, (b) temperature, (c) time

Figure 4 (a) showed that the pulp yield increased with increase in alkali concentration from 10% to 15% and then tends to decline with further increase in concentration. The increase in temperature resulted in an increase in the rate of pulping and hence increased pulp yield. But as the temperature is raised above 150°C the pulp yield dropped sharply as shown in figure 4 (b). The pulp yield decreased with increase in temperature when concentration of cooking liquor and cooking time were kept constant. This could be explained with respect to kinetics of reaction; as reaction proceeds on extended increasing temperature, the speed of molecules decreases beyond equilibrium. Figure 4 (c) indicated that a slight decrease in pulp yield was observed with increase in time. The decrease in pulp yield became significant for a cooking time beyond 150 minutes. This could be as a result of the removal of the hemicellulose components and the partial de-polymerization of cellulose components.

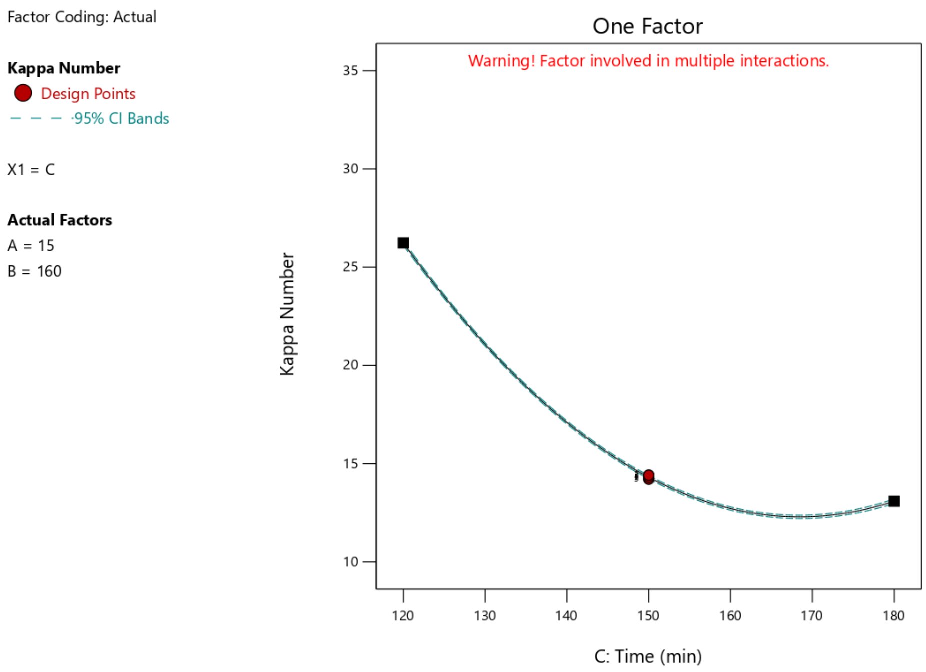
Kappa Number



(a)



(b)



(c)

Figure 5: One factor effect on kappa number (a) alkali concentration, (b) temperature, (c) time

Figure 5 (a) showed that the increase in alkali concentration generally decreased the residual lignin content (kappa number) showing that the cooking liquor reacted with much of the lignin. As depicted in figure 5 (b), the increase in temperature significantly reduced the kappa number. This showed that keeping alkali concentration and reaction time constant, higher temperatures resulted in more lignin getting reacted. Figure 5 (c) indicated that a significant decrease in kappa number (residual lignin) was observed with increase in time. The decrease in residual lignin content became significant for a cooking time beyond 150 minutes. This could be explained with respect to getting sufficient time for more lignin getting reacted.

Interaction Effects

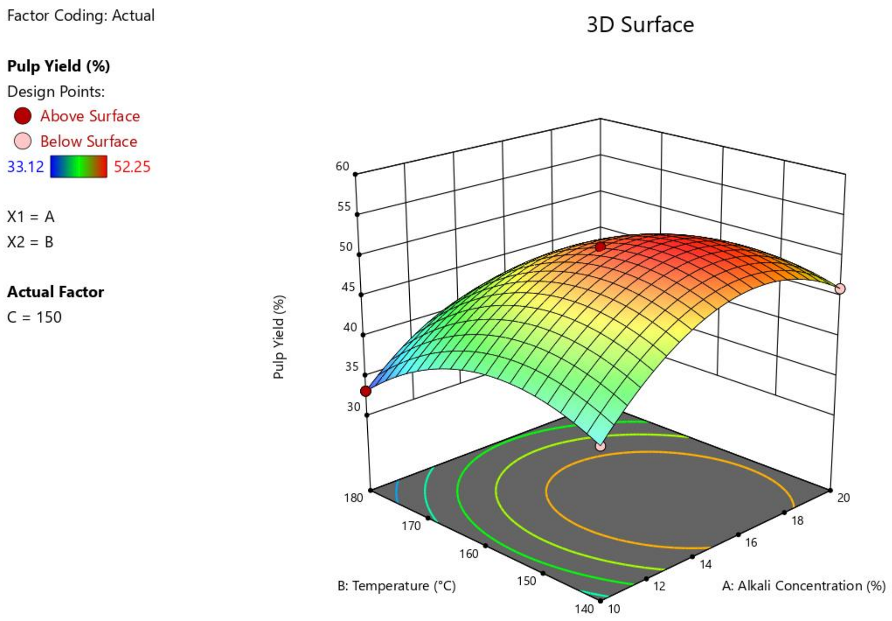


Figure 6: Interaction effect of Alkali Concentration and temperature on pulp yield

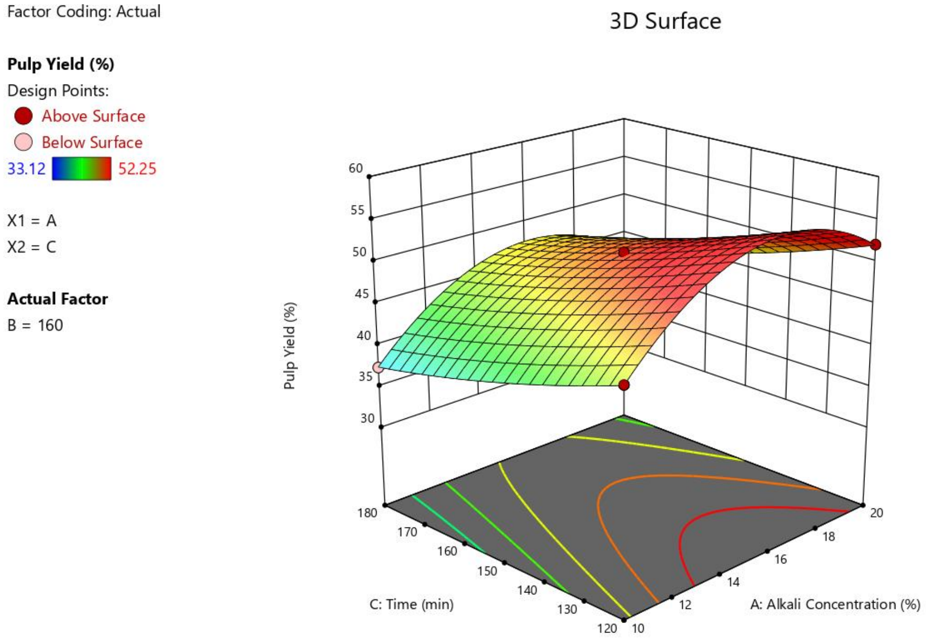


Figure 7: Interaction effect of Alkali Concentration and Time on pulp yield

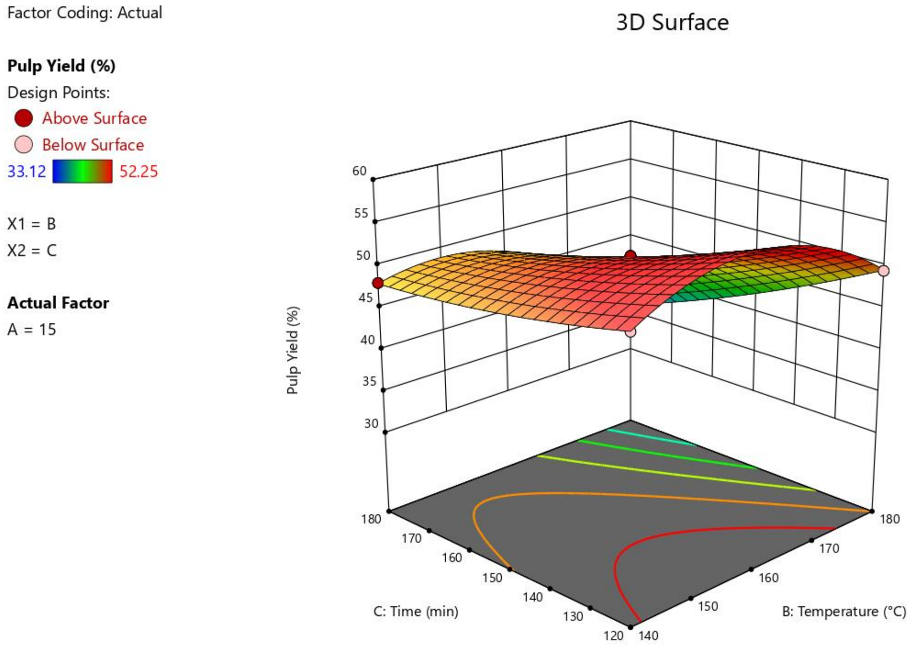


Figure 8: Interaction effect of Temperature and Time on pulp yield

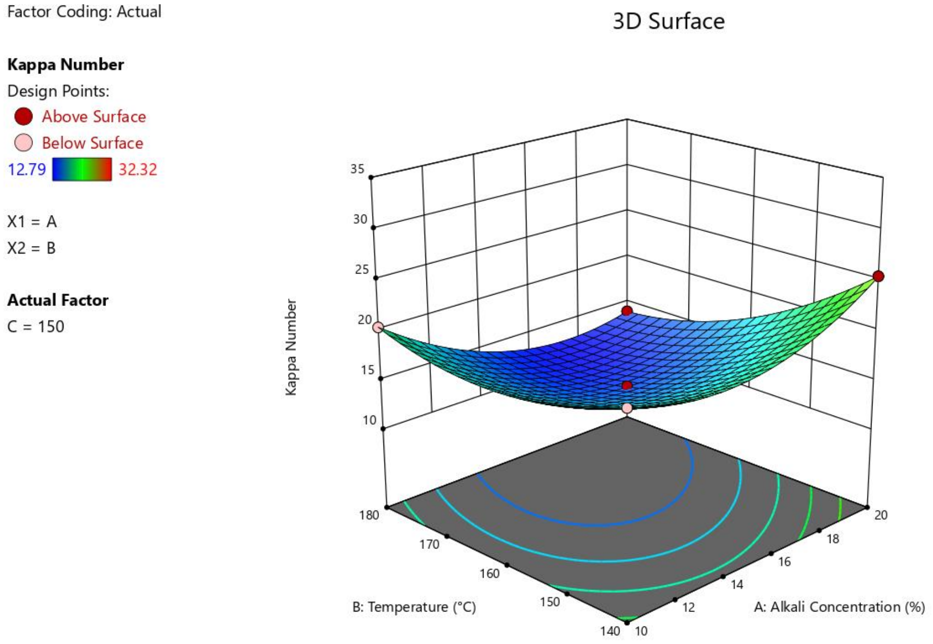


Figure 9: Interaction effect of Alkali Concentration and Temperature on kappa number

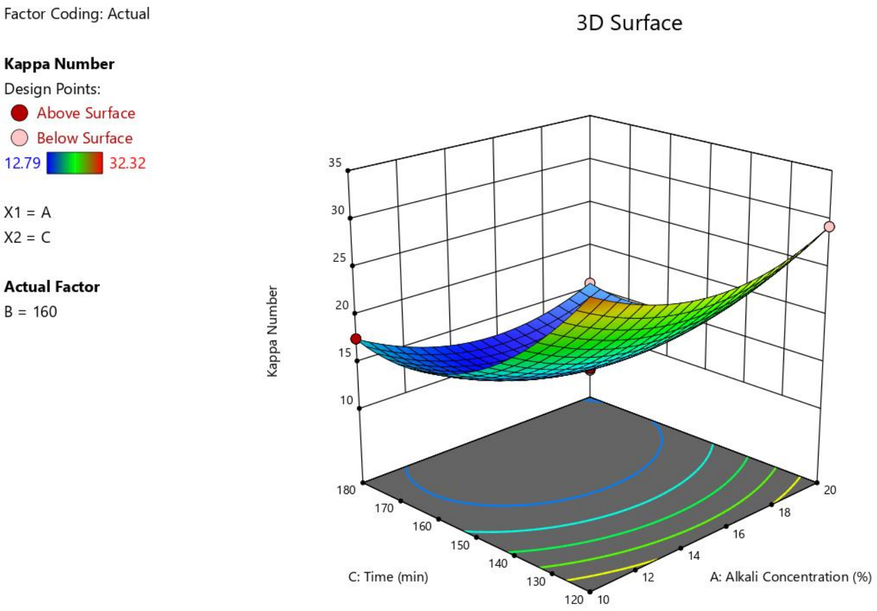


Figure 10: Interaction effect of Alkali Concentration and Time of kappa number

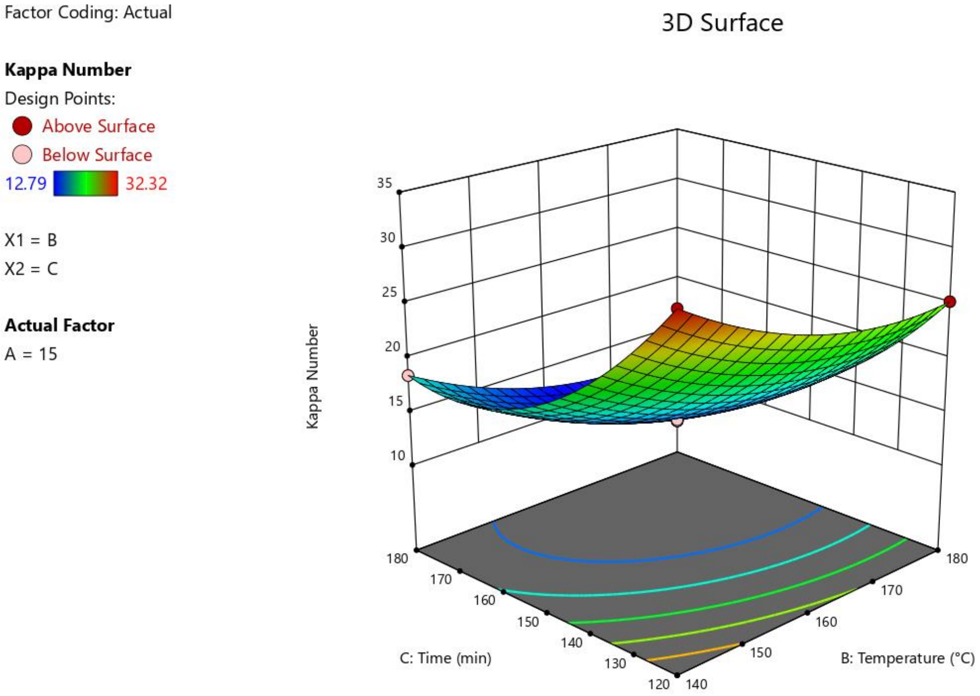


Figure 11: Interaction effect of Temperature and Time on kappa number

4.1.5 Optimization

The purpose of optimization in this research work was maximizing the pulp yield and minimizing the kappa number (the residual lignin content) where the factors alkali concentration, temperature and time were kept within their ranges as in **Table 16** below.

Table 16: Constraints for optimization

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Alkali Concentration	is in range	10	20	1	1	3
B:Temperature	is in range	140	180	1	1	3
C:Time	is in range	120	180	1	1	3
Pulp Yield	maximize	33.12	52.25	1	1	3
Kappa Number	minimize	12.79	32.32	1	1	3

With the constraints imposed the following optimum operating condition was obtained with the desirability of 94.5% at 95% Confidence interval.

Table 17: Optimum operating condition

Number	Alkali Concentration	Temperature	Time	Pulp Yield	Kappa Number	Desirability	
1	15.821	158.624	156.541	50.702	13.362	0.945	Selected

As in Table 17, the optimum operating condition that maximizes the pulp yield and minimizes the kappa number was obtained at active alkali concentration of 15.8%, cooking temperature of 158.6ºC and cooking time of 156 minutes to be 50.7% and 13.4 respectively.

Using the desirability function to get the maximum desirable response for the pulping conditions, the following ramp and bar graphs were obtained.

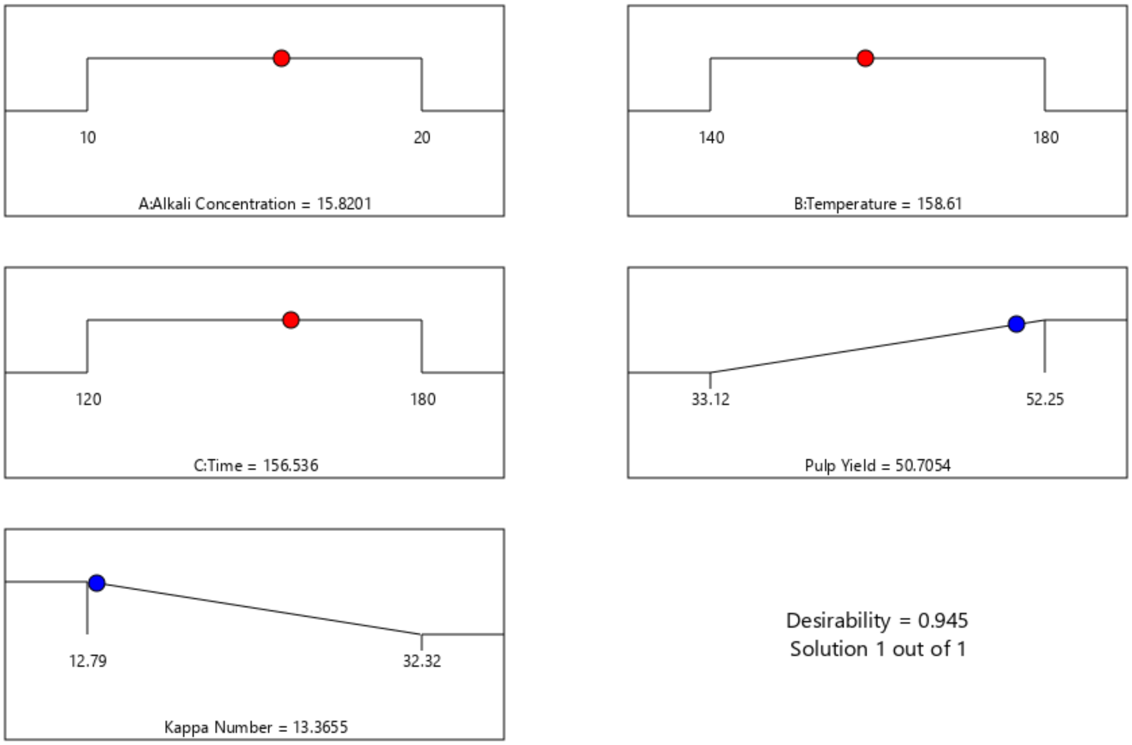


Figure 12: Ramp plot for the optimization conditions

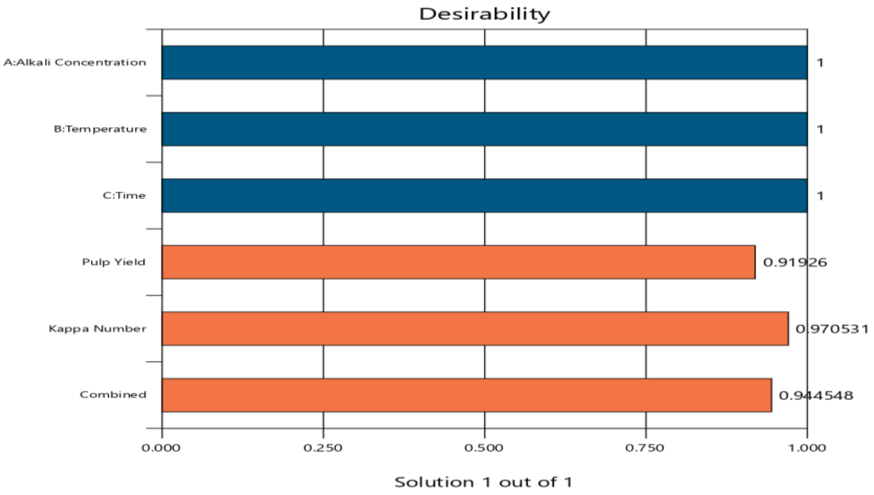


Figure 13: Bar graph for the optimization conditions

4.2 Bamboo Paper Characterization

4.2.1 Paper characterization

The paper made of highland bamboo harvested from western Ethiopia around Jimma zone and pulped in the pulping condition of active alkali concentration of 15.8%, cooking temperature of 158.6ºC and cooking time of 156 minutes were characterized at Ethiopian pulp and paper S.C laboratory and the following test results were obtained.

Table 18: Physical test results of paper made

Physical test parameter	Obtained value	(Ermias, 2020)	(Amsalu, 2015)	Imported pulp*
Tensile index (Nm/g)	*30.2*	48.25	33.58	65-80
Burst index (kPam^2/g)	*1.602*	2.95	1.303	1.5-5.0
Tear index (mNm^2/g)	*12.5*	2.8	11.5	10-12

*Data obtained from Ethiopian pulp and paper S.C (expected properties of imported pulps)

The paper test results obtained in this research work is found to be in close proximity to results obtained by (Ermias, 2020) and (Amsalu, 2015) conducted on Ethiopian highland and low land bamboo respectively. The result in general indicates that quality paper comparable to the one obtained from softwood pulp, with desirable properties can be obtained from Ethiopian highland bamboo with an environmentally friendly pulping procedure of soda – AQ pulping.

CHAPTER FIVE

5. Conclusion and Recommendation

5.1 Conclusion

This research work dealt with the determination of pulp yield from Ethiopian highland bamboo (Yushania Alpina) by soda-AQ (Anthraquionone) catalytic pulping.

Five independent factors were identified to be determinants of the pulp yield that can be made of the raw material bamboo in this catalytic process. Two of the factors: bamboo chips to cooking liquor ratio and anthraquionone concentration were kept constant at 1:4 (w/v) and 0.1% on bamboo oven dry (o.d) basis respectively. Three of the independent factors: active alkali concentration (10%-20%), cooking temperature (140ºC-180ºC) and cooking time (120-180 minutes) were varied in their respective ranges to determine optimum operating condition that maximizes the pulp yield and minimizes the residual lignin content (kappa number).

Response Surface Methodology (RSM) was employed for optimization of the process and the data analysis in this experiment was performed using DESIGN EXPERT® 13 (file version 13.0.1.0). A randomized Box-Behnken design type with seventeen (17) runs was carried out.

The optimum operating condition that maximizes the pulp yield and minimizes the kappa number was obtained at active alkali concentration of 15.8%, cooking temperature of 158.6ºC and cooking time of 156 minutes to be 50.7% and 13.4 respectively. This optimum condition was used to make laboratory scale paper and its mechanical properties were assessed. The tensile, bursting and tearing indexes were found to be 30.2Nm/g, 1.602kPam^2/g and 12.5mNm^2/g respectively. These values indicate that quality paper comparable to the one obtained from softwood pulp, with desirable properties can be obtained from Ethiopian highland bamboo with an environmentally friendly pulping procedure of soda – AQ pulping.

5.2 Recommendation

Over one million hectare of potential resource of bamboo found in Ethiopia, highland bamboo covers more than 300,000 hectares.

This bamboo resource is mainly being utilized for fencing, construction, furniture production and making of bamboo based panels. The result of this research work showed that highland bamboo can be used to produce bamboo pulp that can be made into paper sheet of good quality.

Therefore, it is recommended that this resource be utilized for such a valued commodity that the country imports with a large sum of foreign currency. It is important that further research studies be conducted and preliminary design of a pilot plant be carried out together with feasibility study so that commercial scale pulp and paper production can be implemented using an environmentally friendly soda-AQ catalytic pulping of Ethiopian highland bamboo (*Yushania Alpina*).

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Appendix A: model summaries and plots for pulp yield and kappa number

Fit Summary

Response 1: Pulp Yield

Source	**Sequential p-value**	**Lack of Fit p-value**	**Adjusted R^2**	**Predicted R^2**	
Linear	0.0388	< 0.0001	0.3397	0.0855	
2FI	0.8639	< 0.0001	0.2000	-0.7017	
Quadratic	**< 0.0001**	**0.8177**	**0.9994**	**0.9989**	**Suggested**
Cubic	0.8177		0.9992		**Aliased**

Model Summary Statistics

Source	**Std. Dev.**	**R^2**	**Adjusted R^2**	**Predicted R^2**	**PRESS**	
Linear	5.44	0.4635	0.3397	0.0855	655.20	
2FI	5.98	0.5000	0.2000	-0.7017	1219.19	
Quadratic	**0.1599**	**0.9998**	**0.9994**	**0.9989**	**0.7684**	**Suggested**
Cubic	0.1905	0.9998	0.9992		*	Aliased

*Case(s) with leverage of 1.0000: PRESS statistic not defined.

Response 2: Kappa Number

Source	**Sequential p-value**	**Lack of Fit p-value**	**Adjusted R^2**	**Predicted R^2**	
Linear	0.0036	< 0.0001	0.5515	0.4414	
2FI	0.7459	< 0.0001	0.4815	0.1840	
Quadratic	**< 0.0001**	**0.9688**	**0.9998**	**0.9998**	**Suggested**
Cubic	0.9688		0.9998		**Aliased**

Model Summary Statistics

Source	**Std. Dev.**	**R^2**	**Adjusted R^2**	**Predicted R^2**	**PRESS**	
Linear	4.36	0.6356	0.5515	0.4414	378.46	
2FI	4.69	0.6759	0.4815	0.1840	552.79	
Quadratic	**0.0798**	**0.9999**	**0.9998**	**0.9998**	**0.1049**	**Suggested**
Cubic	0.1026	0.9999	0.9998		*	Aliased

*Case(s) with leverage of 1.0000: PRESS statistic not defined.

ANOVA for Quadratic model

Response 1: Pulp Yield

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	716.25	9	79.58	3112.73	< 0.0001	significant
A-Alkali Concentration	65.09	1	65.09	2546.00	< 0.0001	
B-Temperature	107.46	1	107.46	4202.96	< 0.0001	
C-Time	159.49	1	159.49	6238.08	< 0.0001	
AB	4.80	1	4.80	187.59	< 0.0001	
AC	0.0484	1	0.0484	1.89	0.2113	
BC	21.34	1	21.34	834.84	< 0.0001	
A^2	217.85	1	217.85	8520.68	< 0.0001	
B^2	122.01	1	122.01	4772.03	< 0.0001	
C^2	2.38	1	2.38	93.13	< 0.0001	
Residual	0.1790	7	0.0256			
Lack of Fit	0.0339	3	0.0113	0.3110	0.8177	not significant
Pure Error	0.1451	4	0.0363			
Cor Total	716.43	16				

Factor coding is **Coded**.
Sum of squares is **Type III - Partial**

The **Model F-value** of 3112.73 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case A, B, C, AB, BC, A^2, B^2, C^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The **Lack of Fit F-value** of 0.31 implies the Lack of Fit is not significant relative to the pure error. There is an 81.77% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

Fit Statistics

Std. Dev.	0.1599		**R^2**	0.9998
Mean	45.53		**Adjusted R^2**	0.9994
C.V. %	0.3512		**Predicted R^2**	0.9989
			Adeq Precision	156.4594

The **Predicted R^2** of 0.9989 is in reasonable agreement with the **Adjusted R^2** of 0.9994; i.e. the difference is less than 0.2.

Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 156.459 indicates an adequate signal. This model can be used to navigate the design space.

Coefficients in Terms of Coded Factors

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	51.10	1	0.0715	50.93	51.27	
A-Alkali Concentration	2.85	1	0.0565	2.72	2.99	1.0000
B-Temperature	-3.66	1	0.0565	-3.80	-3.53	1.0000
C-Time	-4.47	1	0.0565	-4.60	-4.33	1.0000
AB	-1.10	1	0.0799	-1.28	-0.9060	1.0000
AC	-0.1100	1	0.0799	-0.2990	0.0790	1.0000
BC	-2.31	1	0.0799	-2.50	-2.12	1.0000
A^2	-7.19	1	0.0779	-7.38	-7.01	1.01
B^2	-5.38	1	0.0779	-5.57	-5.20	1.01
C^2	0.7520	1	0.0779	0.5677	0.9363	1.01

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multi-colinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

Final Equation in Terms of Coded Factors

Pulp Yield	=
+51.10	
+2.85	A
-3.66	B
-4.47	C
-1.10	AB
-0.1100	AC
-2.31	BC
-7.19	A^2
-5.38	B^2
+0.7520	C^2

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

Response 2: Kappa Number

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	677.44	9	75.27	11832.44	< 0.0001	significant
A-Alkali Concentration	3.59	1	3.59	564.53	< 0.0001	
B-Temperature	81.41	1	81.41	12797.25	< 0.0001	
C-Time	345.58	1	345.58	54324.60	< 0.0001	
AB	26.52	1	26.52	4169.27	< 0.0001	
AC	0.3364	1	0.3364	52.88	0.0002	
BC	0.4900	1	0.4900	77.03	< 0.0001	
A^2	51.26	1	51.26	8057.19	< 0.0001	
B^2	27.25	1	27.25	4283.67	< 0.0001	
C^2	120.47	1	120.47	18937.69	< 0.0001	
Residual	0.0445	7	0.0064			
Lack of Fit	0.0025	3	0.0008	0.0776	0.9688	not significant
Pure Error	0.0421	4	0.0105			
Cor Total	677.49	16				

Factor coding is **Coded**.
Sum of squares is **Type III - Partial**

The **Model F-value** of 11832.44 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case A, B, C, AB, AC, BC, A^2, B^2, C^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The **Lack of Fit F-value** of 0.08 implies the Lack of Fit is not significant relative to the pure error. There is a 96.88% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

Fit Statistics

Std. Dev.	0.0798		**R^2**	0.9999
Mean	19.67		**Adjusted R^2**	0.9998
C.V. %	0.4055		**Predicted R^2**	0.9998
			Adeq Precision	319.1818

The **Predicted R^2** of 0.9998 is in reasonable agreement with the **Adjusted R^2** of 0.9998; i.e. the difference is less than 0.2.

Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 319.182 indicates an adequate signal. This model can be used to navigate the design space.

Coefficients in Terms of Coded Factors

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	14.31	1	0.0357	14.23	14.40	
A-Alkali Concentration	-0.6700	1	0.0282	-0.7367	-0.6033	1.0000
B-Temperature	-3.19	1	0.0282	-3.26	-3.12	1.0000
C-Time	-6.57	1	0.0282	-6.64	-6.51	1.0000
AB	-2.58	1	0.0399	-2.67	-2.48	1.0000
AC	-0.2900	1	0.0399	-0.3843	-0.1957	1.0000
BC	0.3500	1	0.0399	0.2557	0.4443	1.0000
A^2	3.49	1	0.0389	3.40	3.58	1.01
B^2	2.54	1	0.0389	2.45	2.64	1.01
C^2	5.35	1	0.0389	5.26	5.44	1.01

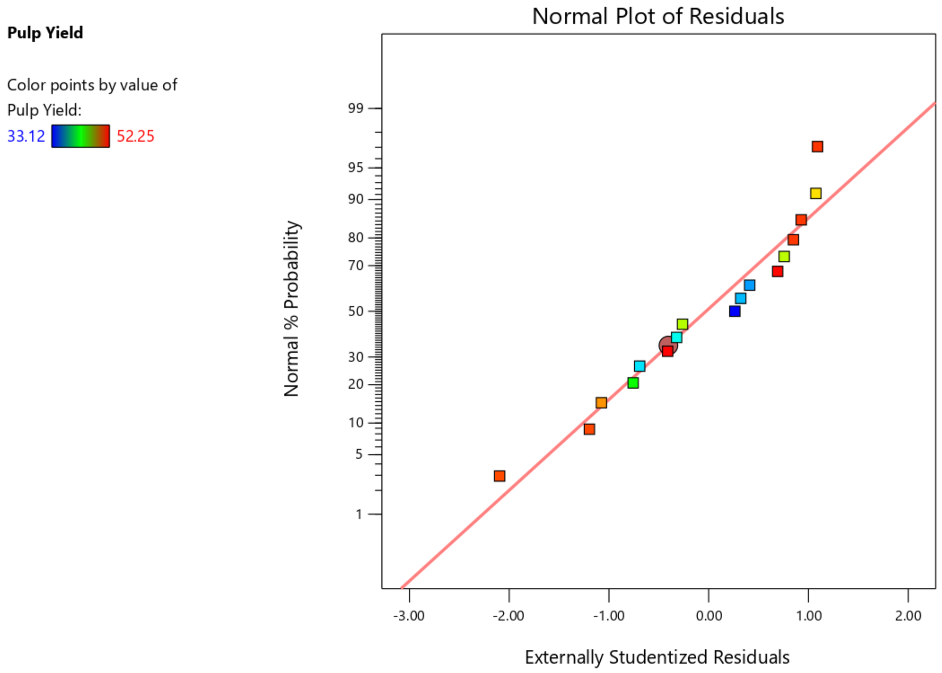
The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multi-colinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

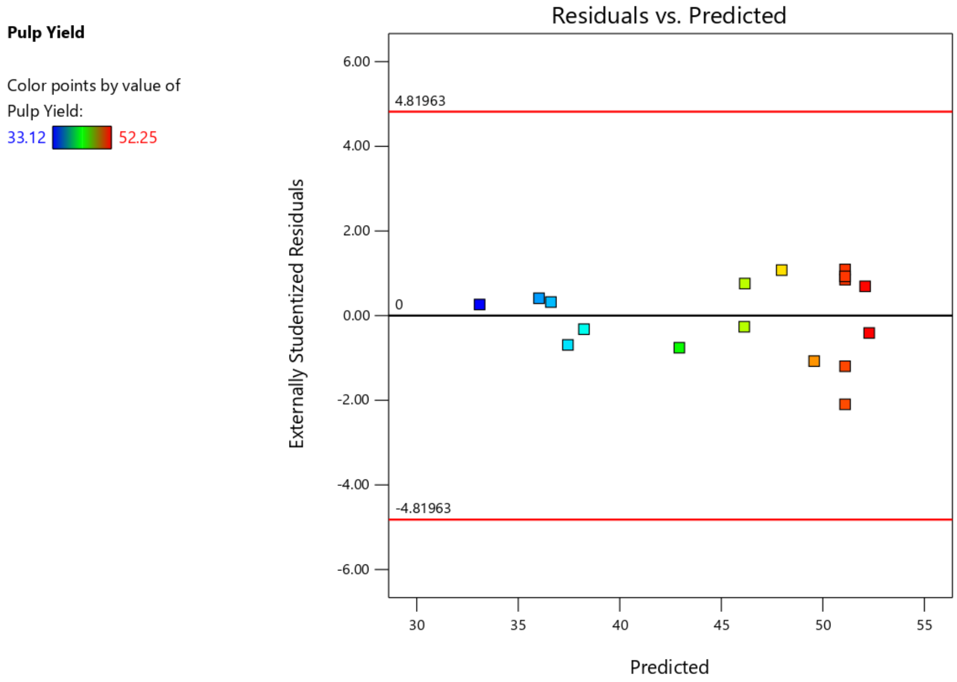
Final Equation in Terms of Coded Factors

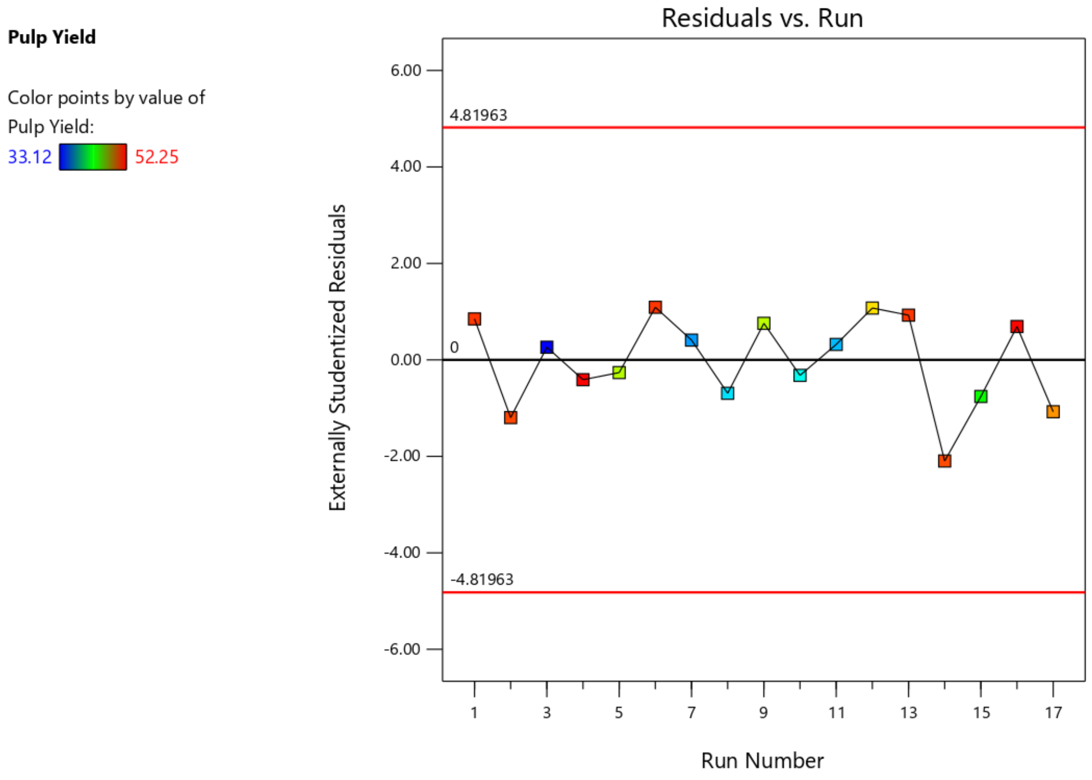
Kappa Number	=
+14.31	
-0.6700	A
-3.19	B
-6.57	C
-2.58	AB
-0.2900	AC
+0.3500	BC
+3.49	A^2
+2.54	B^2
+5.35	C^2

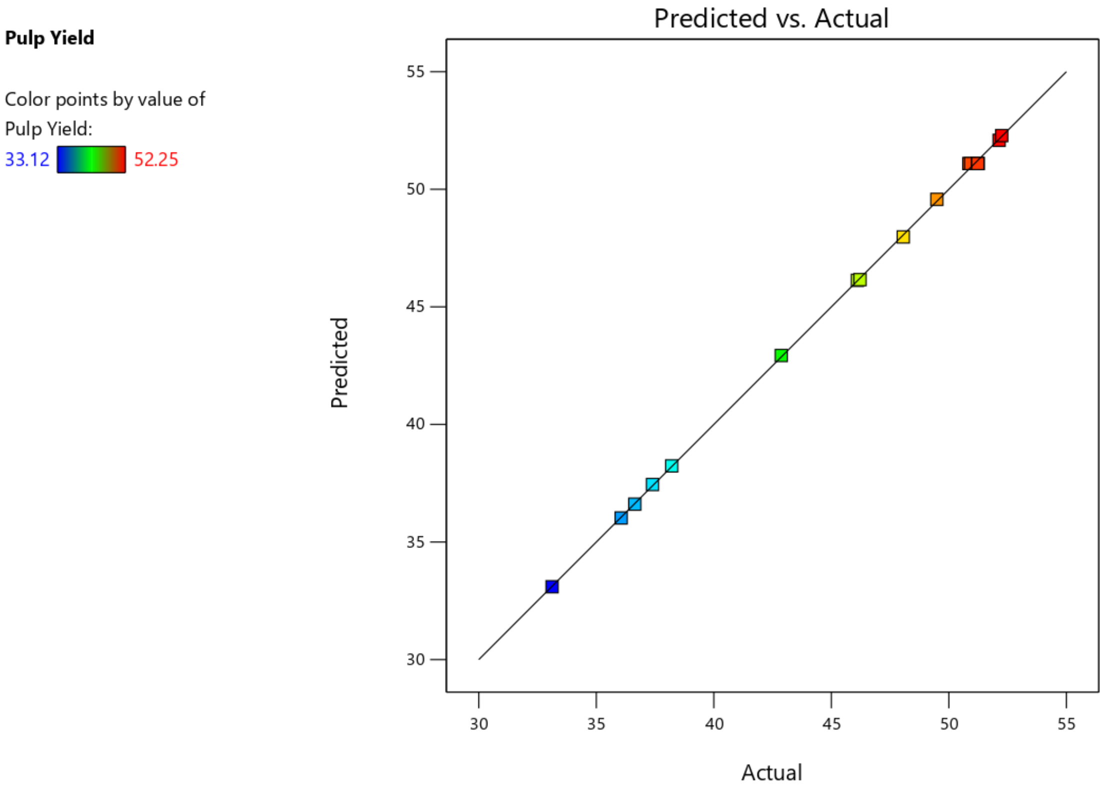
The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

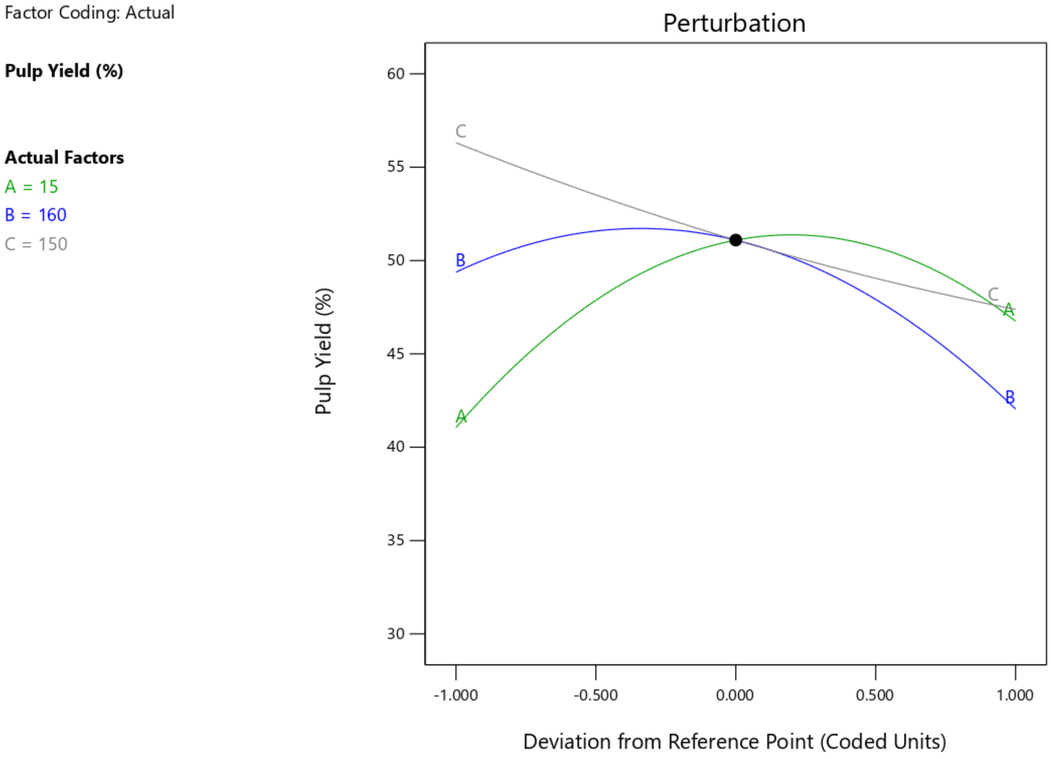
Diagnostic plots and 3D model graphs for kappa number

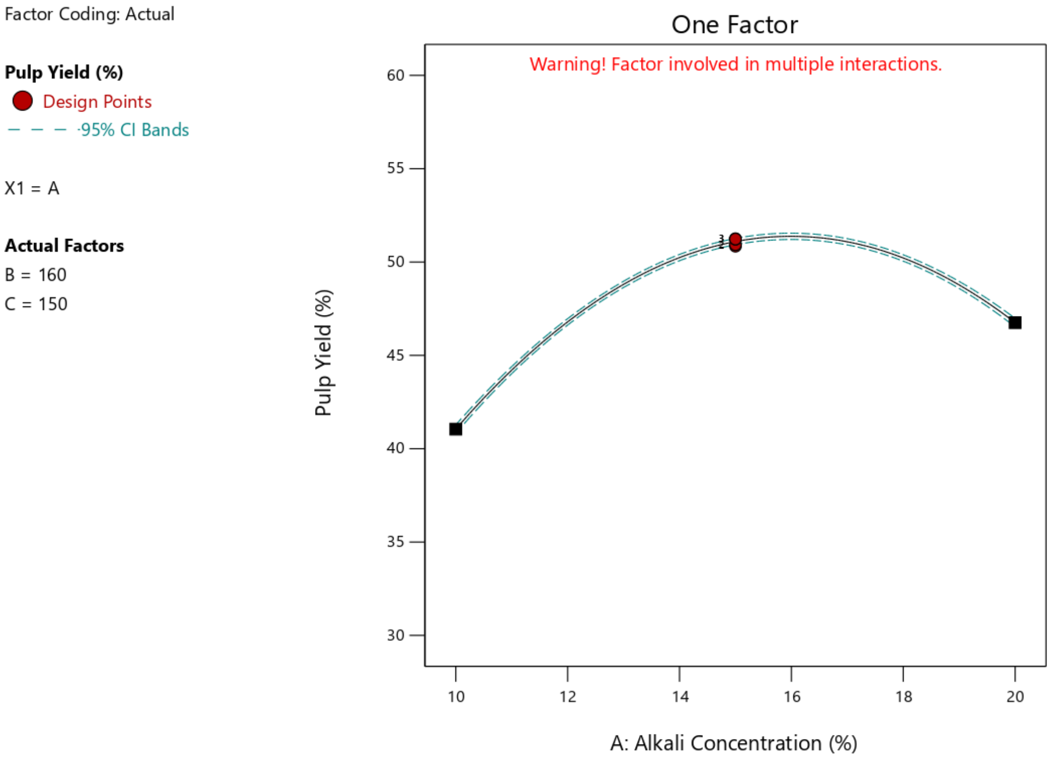


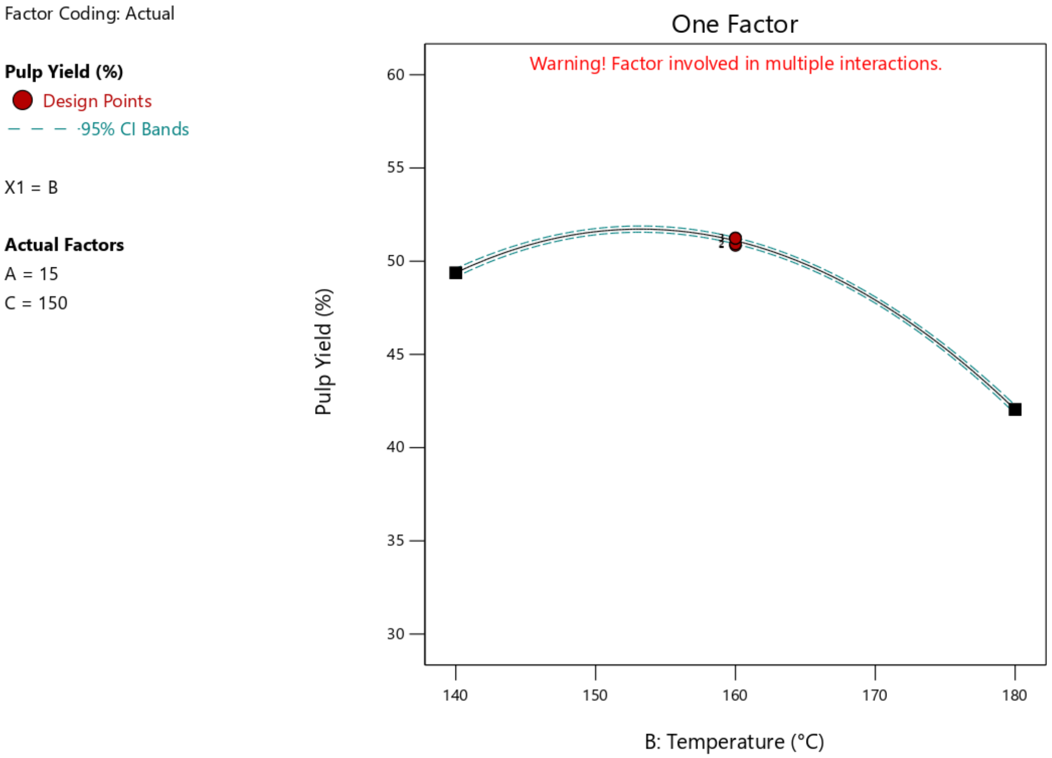


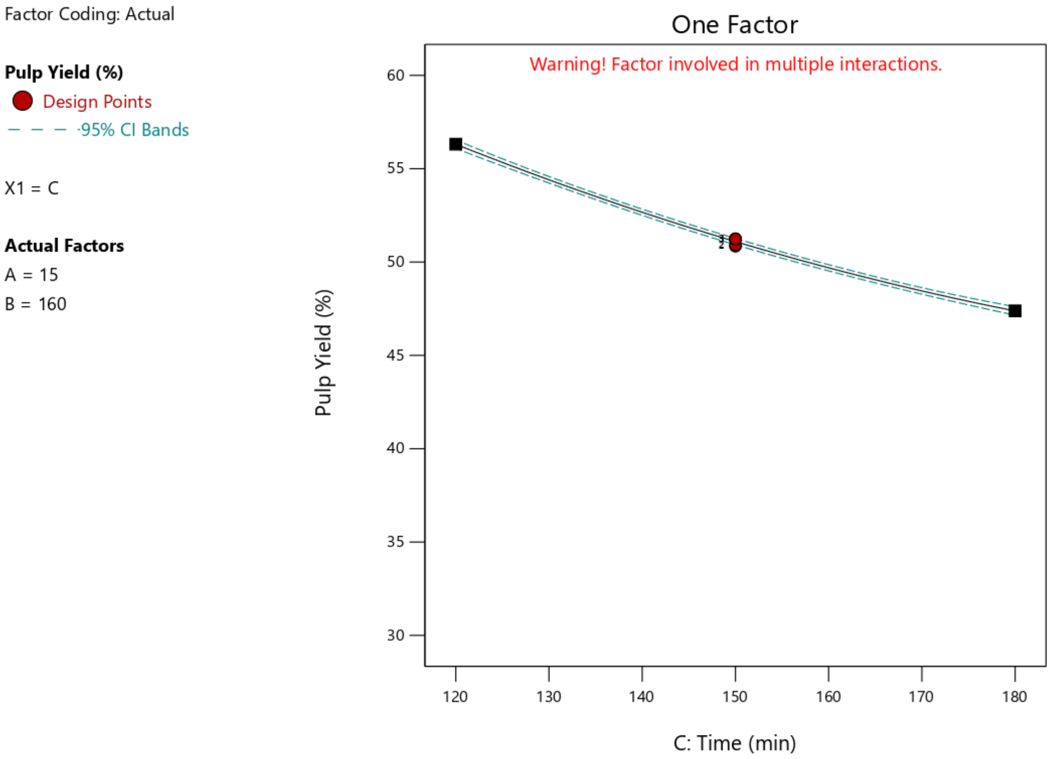

Pulp Yield
Color points by value of
Pulp Yield:
33.12
52.25
Residuals vs. Run
Externally Studentized Residuals
6.00
4.00
2.00
0.00
-2.00
-4.00
-6.00
4.81963
-4.81963
0
Run Number
1
3
5
7
9
11
13
15
17

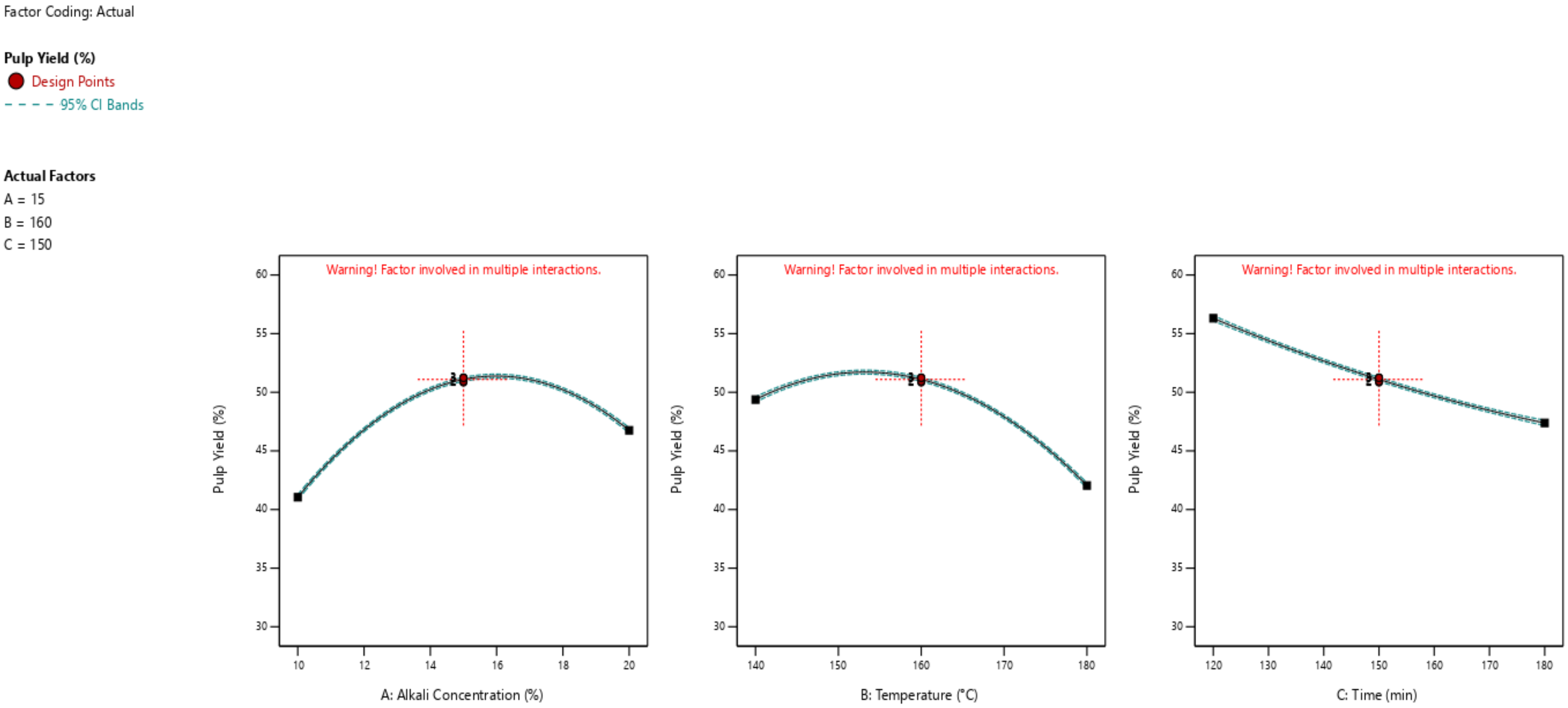

Pulp Yield
Color points by value of
Pulp Yield:
33.12
52.25
Predicted vs. Actual
Predicted
55
50
45
40
35
30
30
35
40
45
50
55
Actual

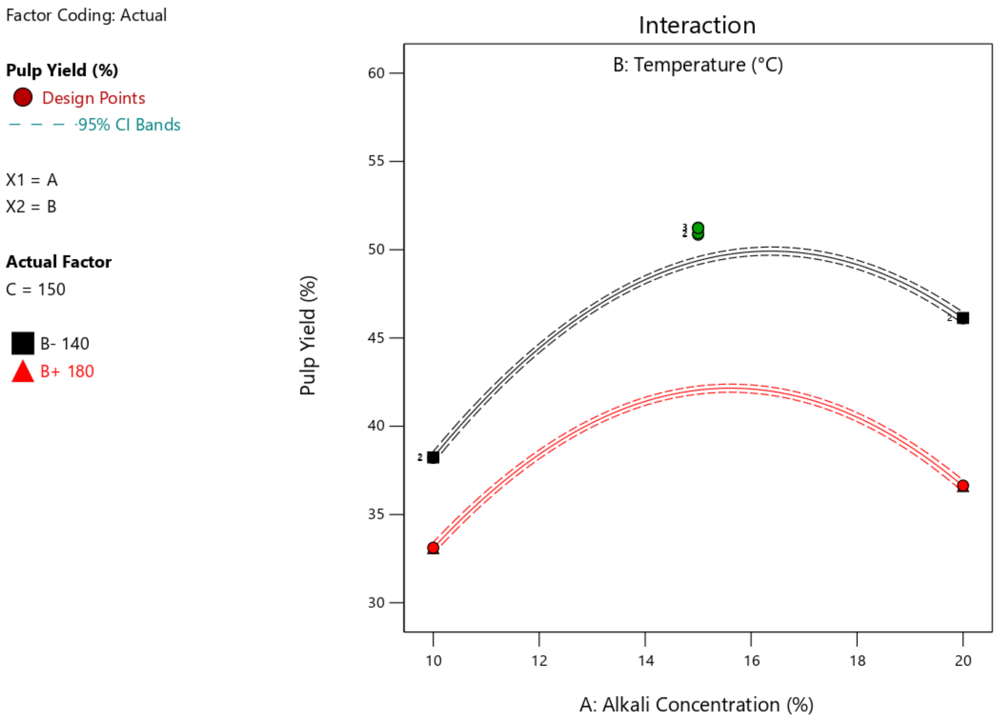

Factor Coding: Actual
Pulp Yield (%)
Actual Factors
A = 15
B = 160
C = 150
Perturbation
Pulp Yield (%)
60
55
50
45
40
35
30
C
B
A
C
A
B
-1.000
-0.500
0.000
0.500
1.000
Deviation from Reference Point (Coded Units)

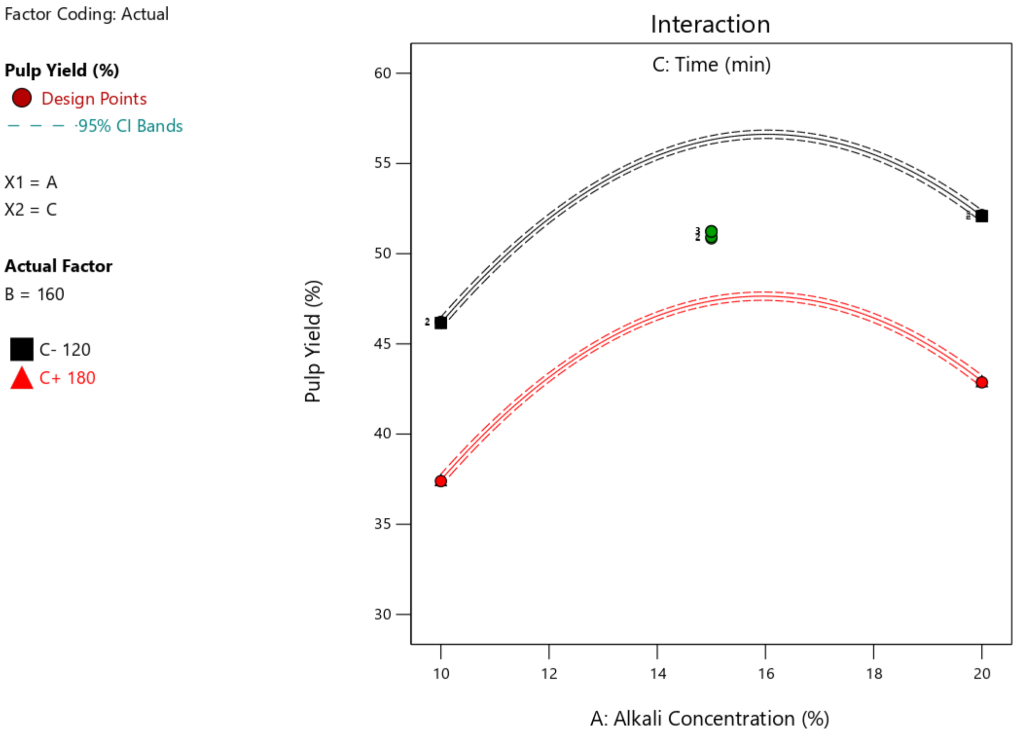

Factor Coding: Actual
Pulp Yield (%)
● Design Points
– – – 95% CI Bands
X1 = A
Actual Factors
B = 160
C = 150
One Factor
Warning! Factor involved in multiple interactions.
Pulp Yield (%)
60
55
50
45
40
35
30
10
12
14
16
18
20
A: Alkali Concentration (%)
3
2

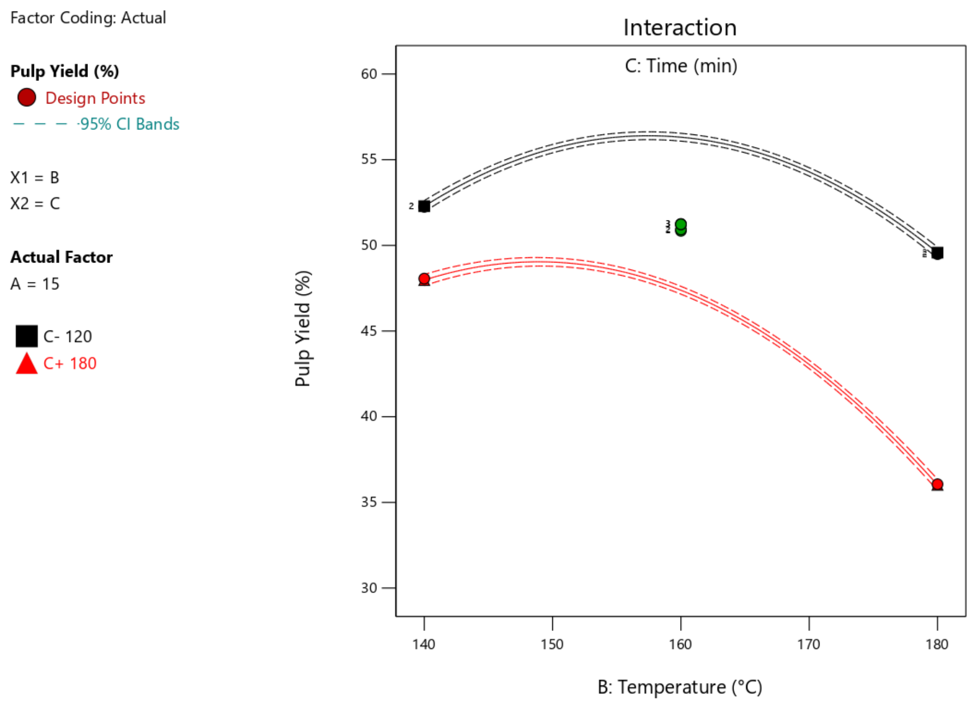

Factor Coding: Actual
Pulp Yield (%)
● Design Points
– – – 95% CI Bands
X1 = B
Actual Factors
A = 15
C = 150
One Factor
Warning! Factor involved in multiple interactions.
Pulp Yield (%)
60
55
50
45
40
35
30
140
150
160
170
180
B: Temperature (°C)
3
2

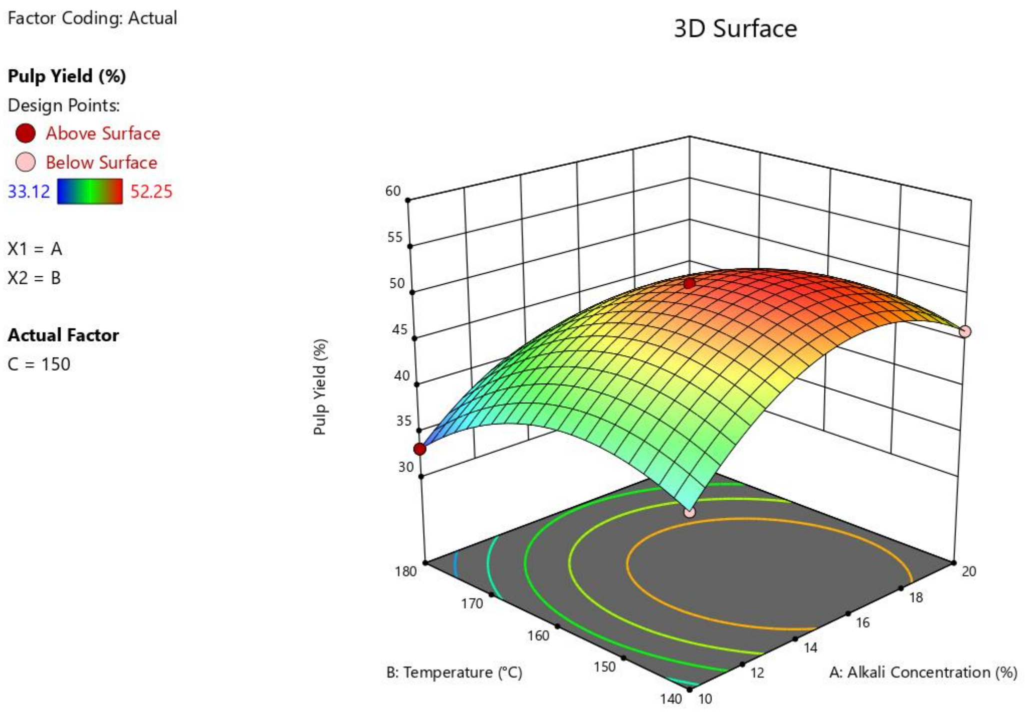

Factor Coding: Actual
Pulp Yield (%)
● Design Points
95% CI Bands
X1 = C
Actual Factors
A = 15
B = 160
One Factor
Warning! Factor involved in multiple interactions.
Pulp Yield (%)
60
55
50
45
40
35
30
2
120
130
140
150
160
170
180
C: Time (min)

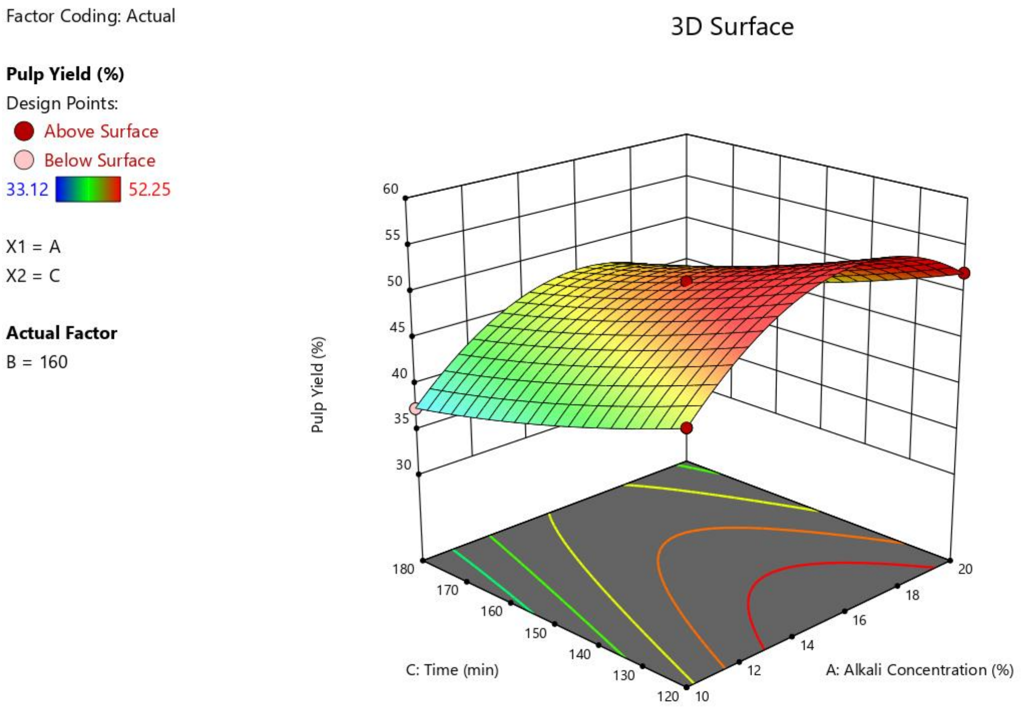

Factor Coding: Actual
Pulp Yield (%)
● Design Points
- - - - 95% CI Bands
Actual Factors
A = 15
B = 160
C = 150
Warning! Factor involved in multiple interactions.
Pulp Yield (%)
60
55
50
45
40
35
30
10
12
14
16
18
20
A: Alkali Concentration (%)
Warning! Factor involved in multiple interactions.
Pulp Yield (%)
140
150
160
170
180
B: Temperature (°C)
Warning! Factor involved in multiple interactions.
Pulp Yield (%)
120
130
140
150
160
170
180
C: Time (min)

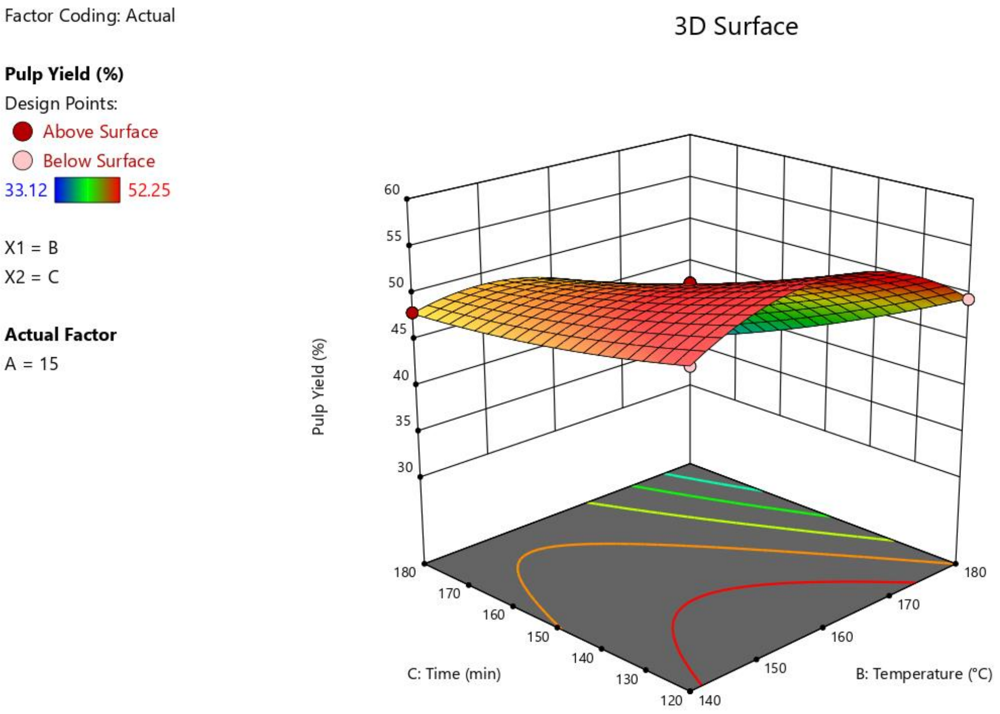

Factor Coding: Actual
Pulp Yield (%)
Design Points
95% CI Bands
X1 = A
X2 = B
Actual Factor
C = 150
B- 140
B+ 180
Interaction
B: Temperature (°C)
Pulp Yield (%)
60
55
50
45
40
35
30
10
12
14
16
18
20
A: Alkali Concentration (%)
2
3
2


Factor Coding: Actual
Pulp Yield (%)
Design Points
95% CI Bands
X1 = A
X2 = C
Actual Factor
B = 160
C- 120
C+ 180
Interaction
C: Time (min)
Pulp Yield (%)
60
55
50
45
40
35
30
10
12
14
16
18
20
A: Alkali Concentration (%)
2
3
2

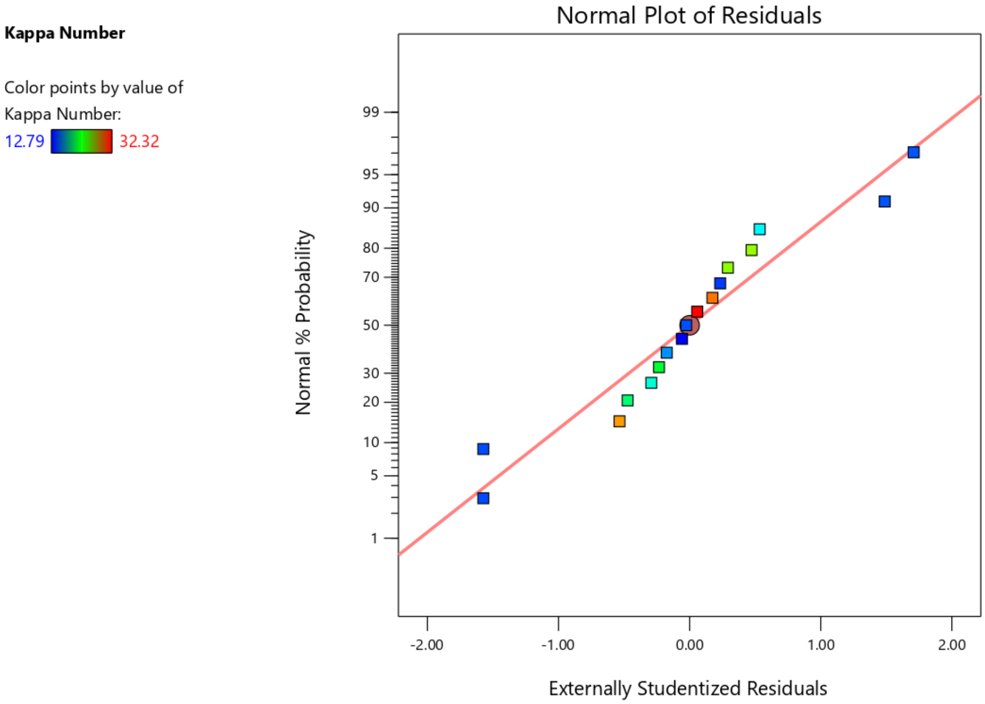

Factor Coding: Actual
Pulp Yield (%)
● Design Points
95% CI Bands
X1 = B
X2 = C
Actual Factor
A = 15
■ C- 120
▲ C+ 180
Interaction
C: Time (min)
Pulp Yield (%)
60
55
50
45
40
35
30
140
150
160
170
180
B: Temperature (°C)

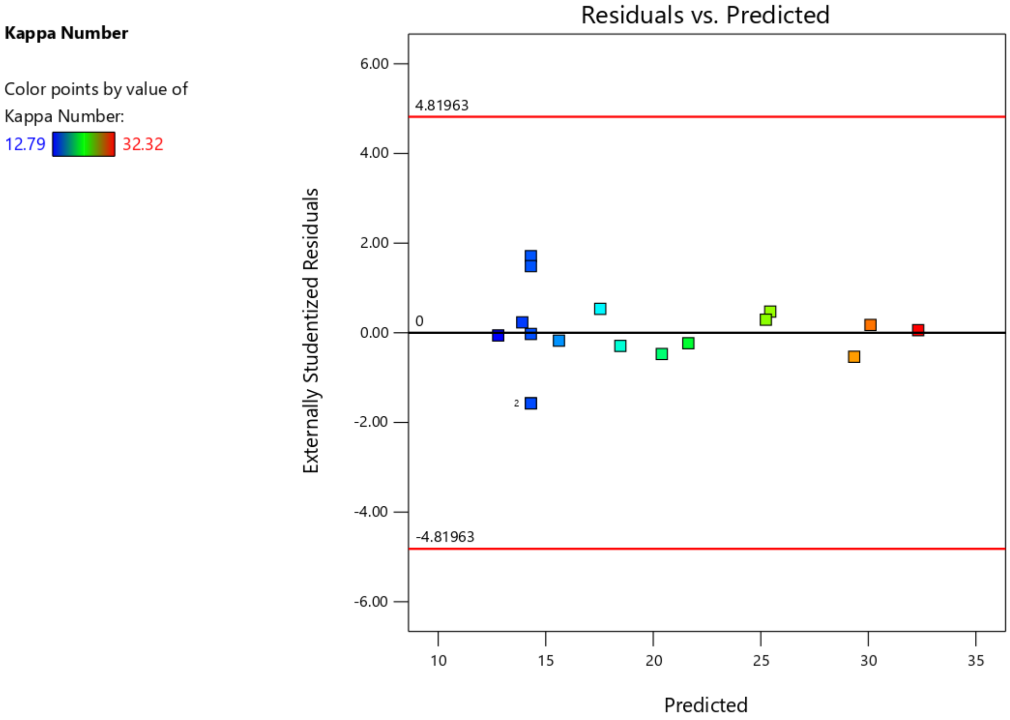

Factor Coding: Actual
Pulp Yield (%)
Design Points:
● Above Surface
○ Below Surface
33.12
52.25
X1 = A
X2 = B
Actual Factor
C = 150
3D Surface
Pulp Yield (%)
60
55
50
45
40
35
30
180
170
160
150
140
B: Temperature (°C)
10
12
14
16
18
20
A: Alkali Concentration (%)

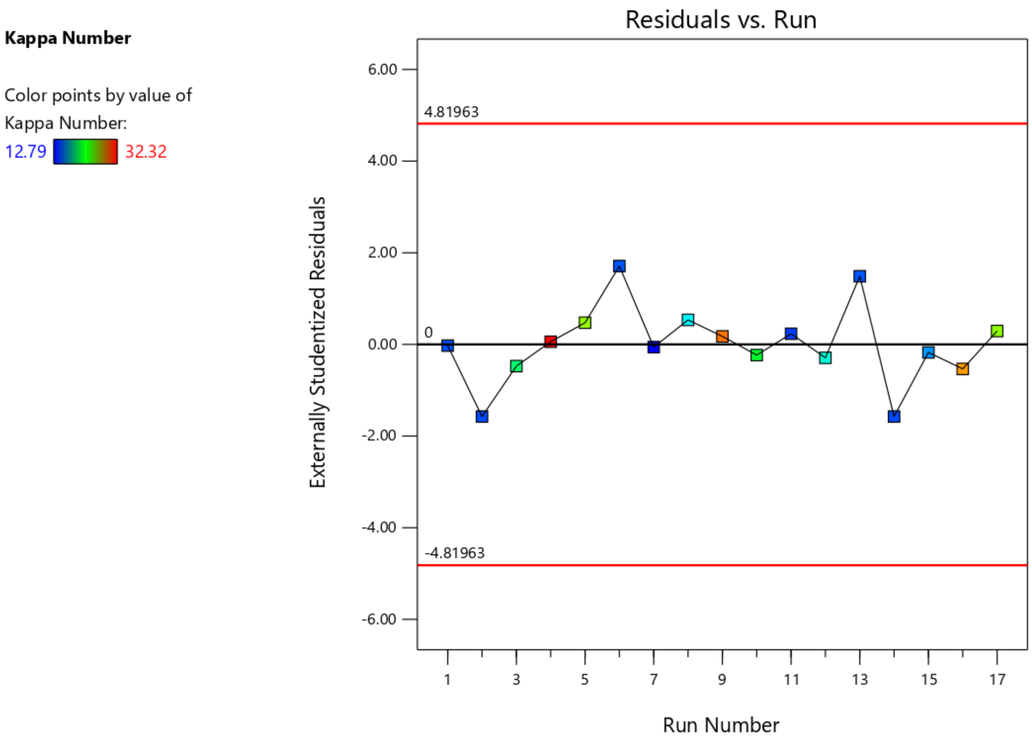

Factor Coding: Actual
Pulp Yield (%)
Design Points:
Above Surface
Below Surface
33.12
52.25
X1 = A
X2 = C
Actual Factor
B = 160
3D Surface
Pulp Yield (%)
60
55
50
45
40
35
30
C: Time (min)
180
170
160
150
140
130
120
A: Alkali Concentration (%)
10
12
14
16
18
20

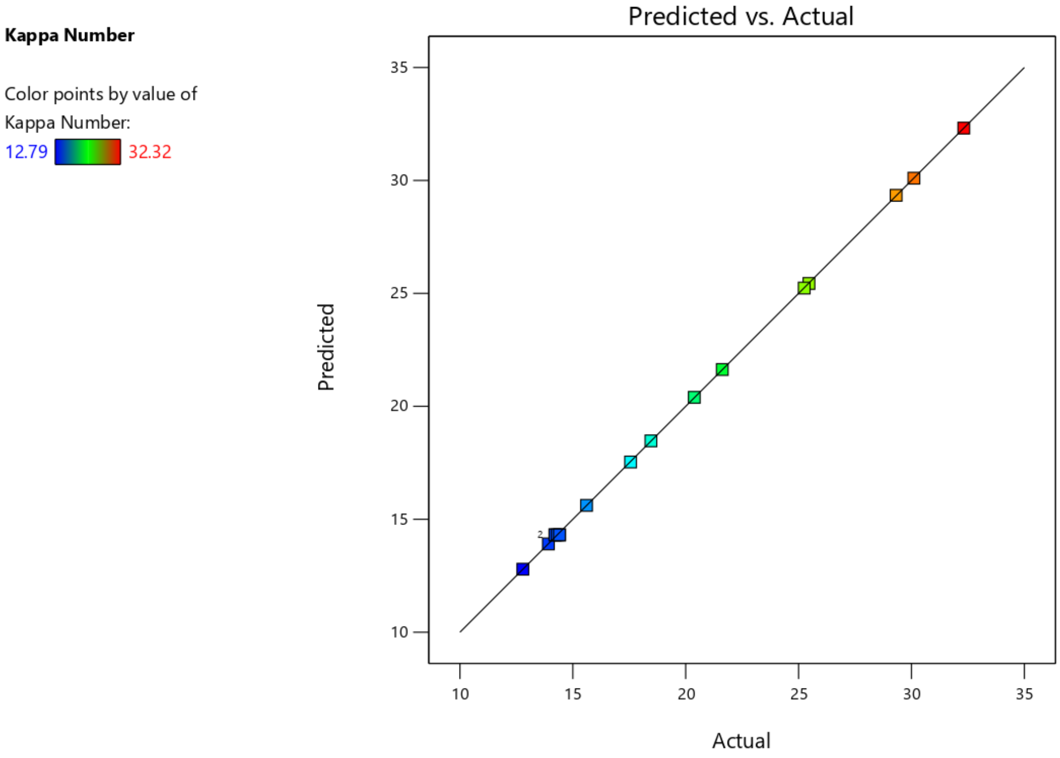

Factor Coding: Actual
Pulp Yield (%)
Design Points:
Above Surface
Below Surface
33.12
52.25
X1 = B
X2 = C
Actual Factor
A = 15
3D Surface
Pulp Yield (%)
60
55
50
45
40
35
30
C: Time (min)
180
170
160
150
140
130
120
B: Temperature (°C)
140
150
160
170
180

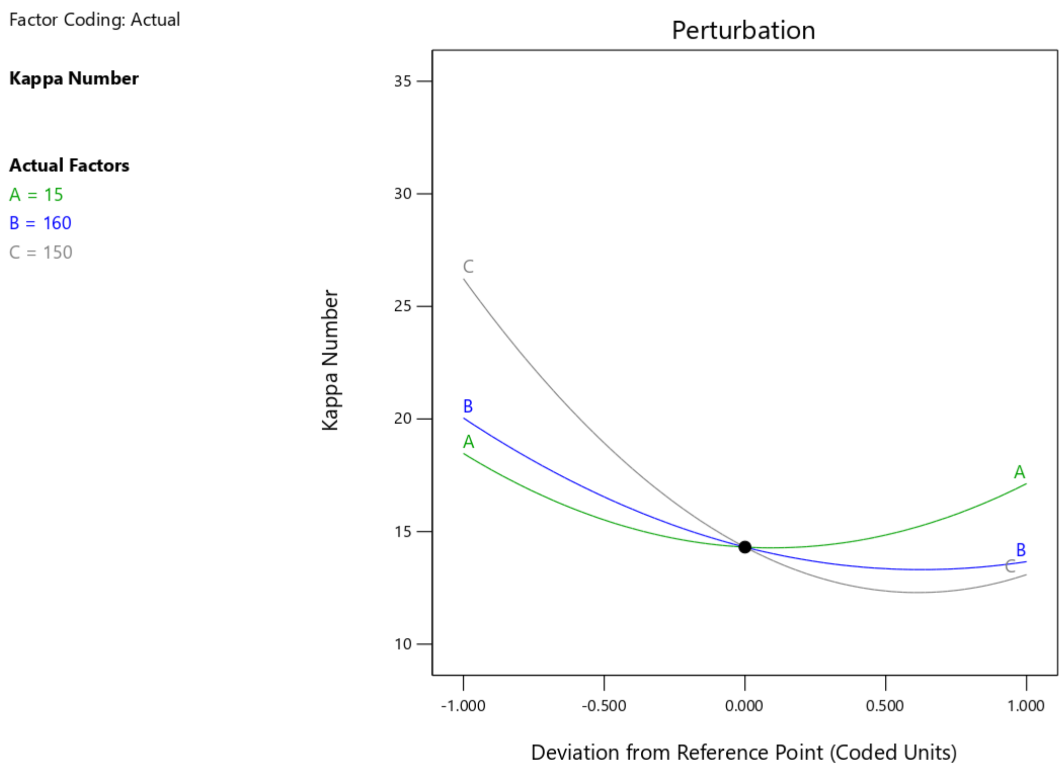
Diagnostic plots and 3D model graphs for kappa number

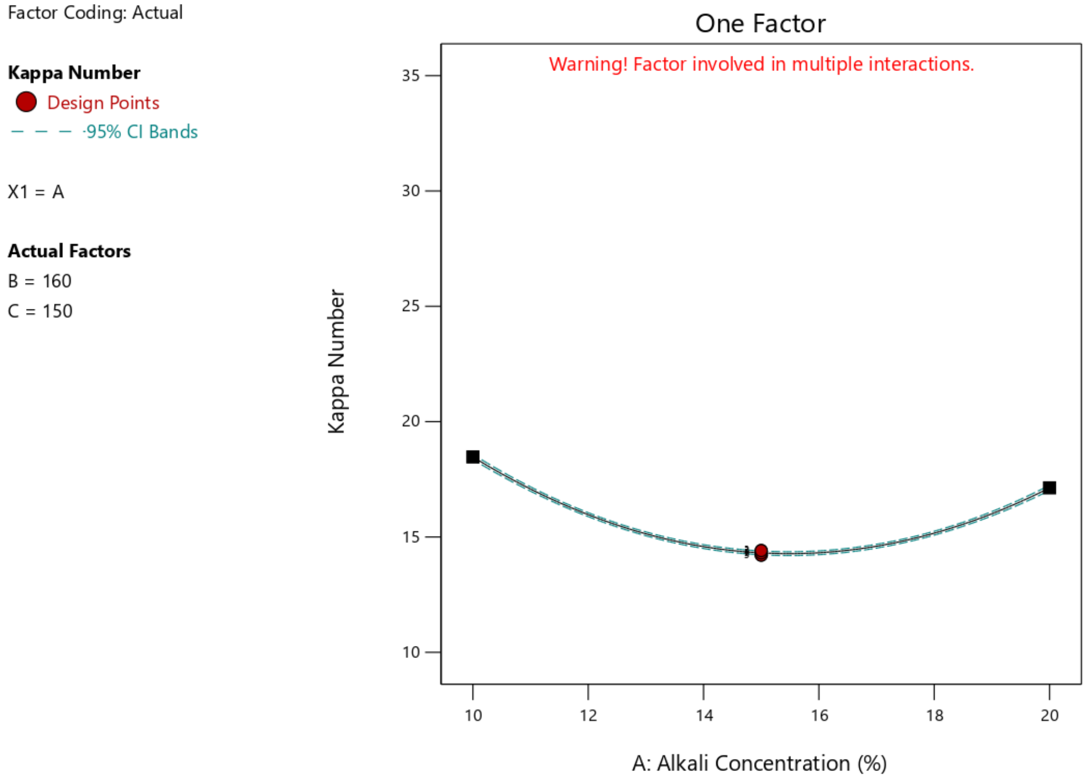


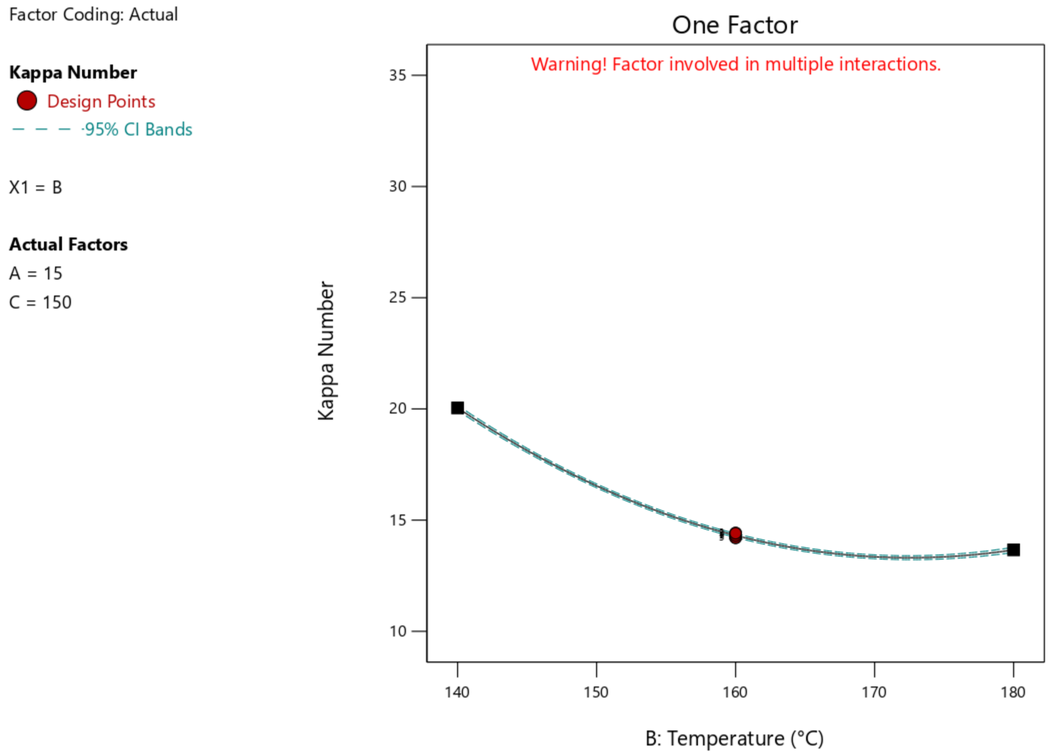


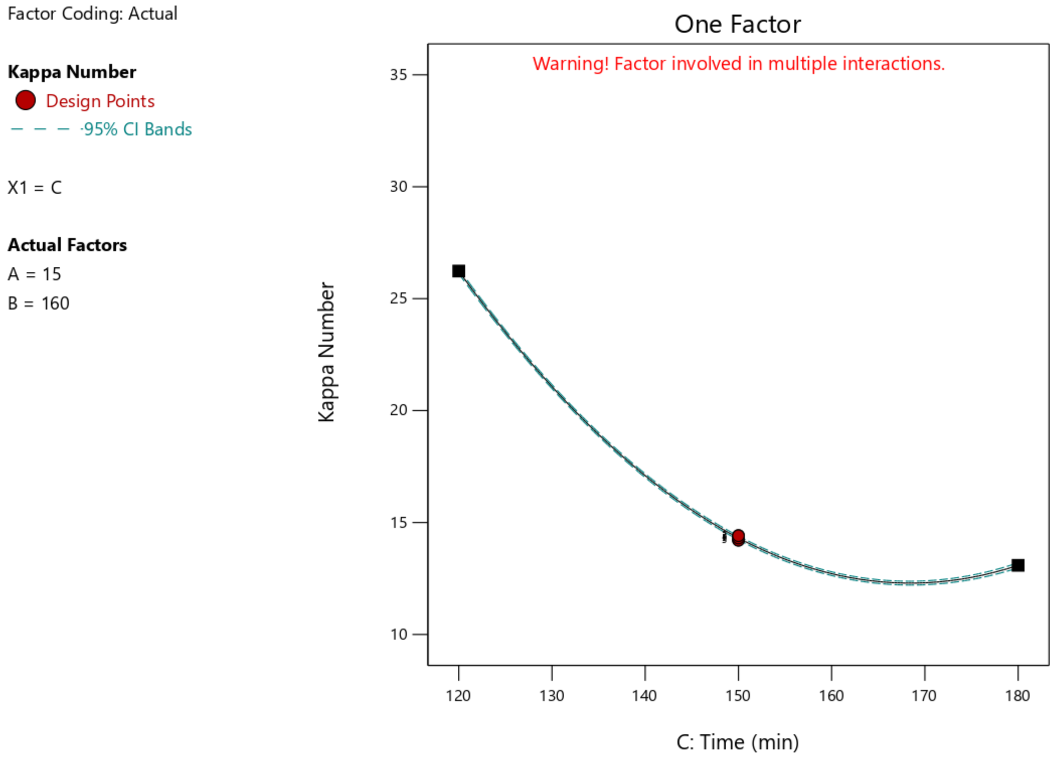

Kappa Number
Color points by value of
Kappa Number:
12.79
32.32
Residuals vs. Run
Externally Studentized Residuals
6.00
4.00
2.00
0.00
-2.00
-4.00
-6.00
4.81963
0
-4.81963
1
3
5
7
9
11
13
15
17
Run Number

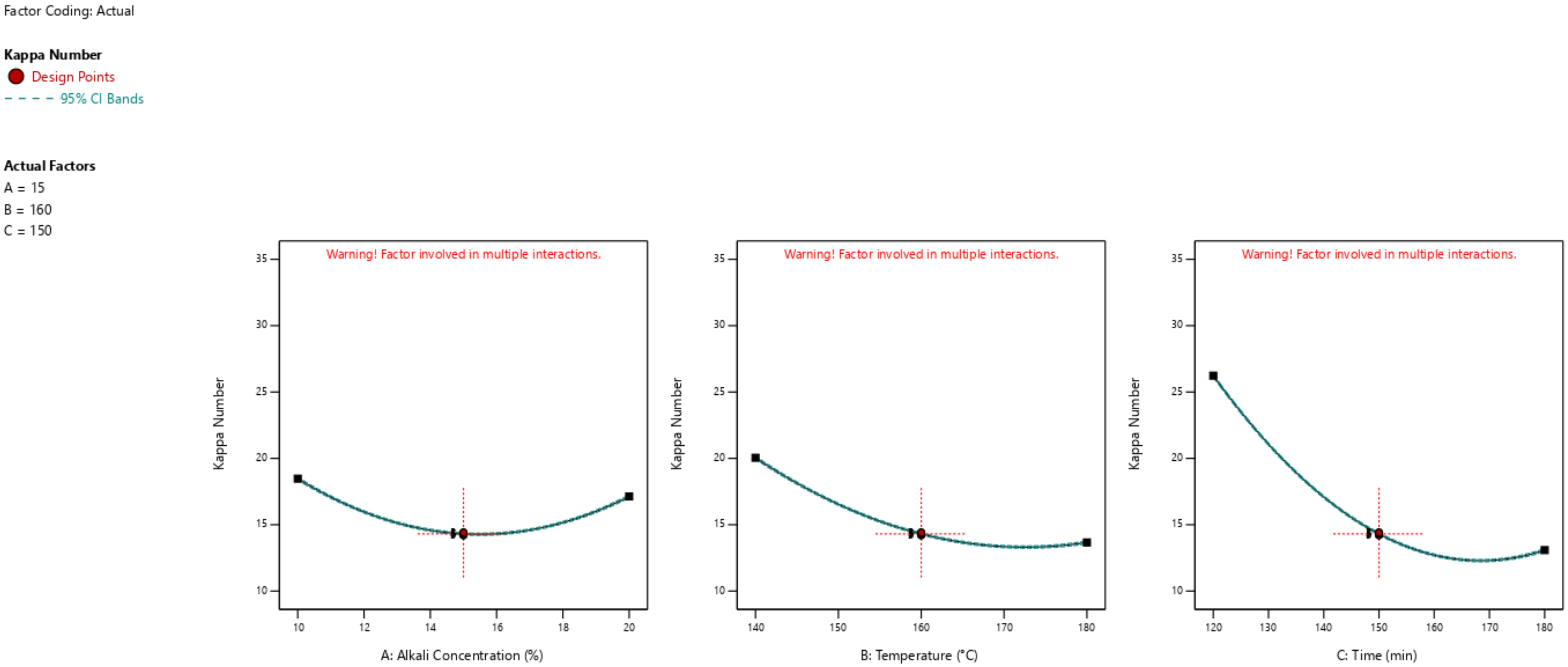

Kappa Number
Color points by value of
Kappa Number:
12.79
32.32
Predicted vs. Actual
Predicted
35
30
25
20
15
10
2
10
15
20
25
30
35
Actual

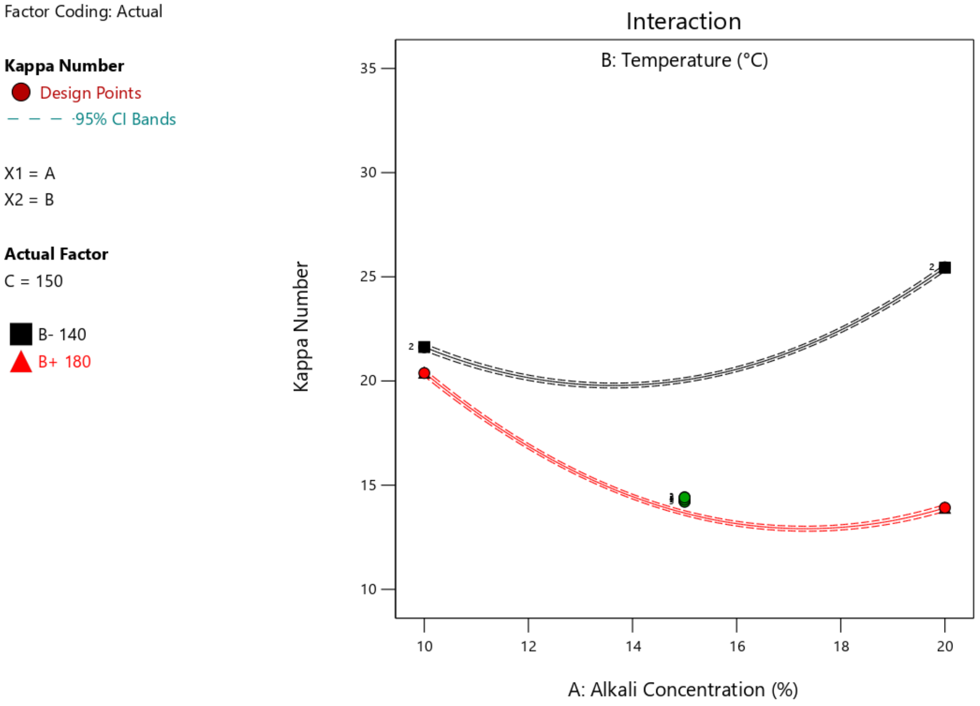

Factor Coding: Actual
Kappa Number
Actual Factors
A = 15
B = 160
C = 150
Perturbation
Kappa Number
35
30
25
20
15
10
C
B
A
A
B
C
-1.000
-0.500
0.000
0.500
1.000
Deviation from Reference Point (Coded Units)

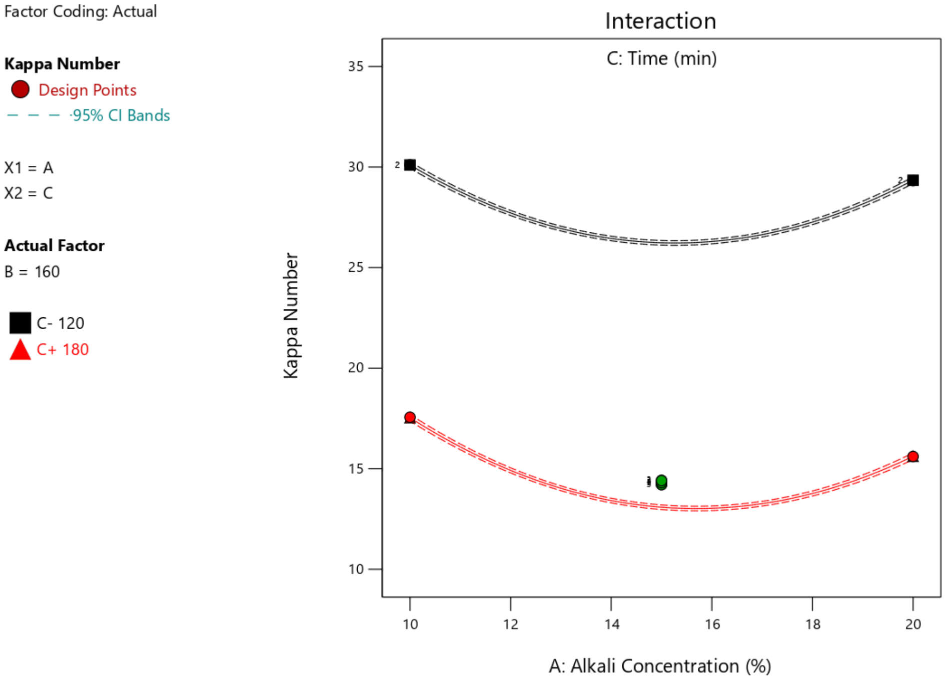

Factor Coding: Actual
Kappa Number
● Design Points
– – – 95% CI Bands
X1 = A
Actual Factors
B = 160
C = 150
One Factor
Warning! Factor involved in multiple interactions.
Kappa Number
35
30
25
20
15
10
10
12
14
16
18
20
A: Alkali Concentration (%)

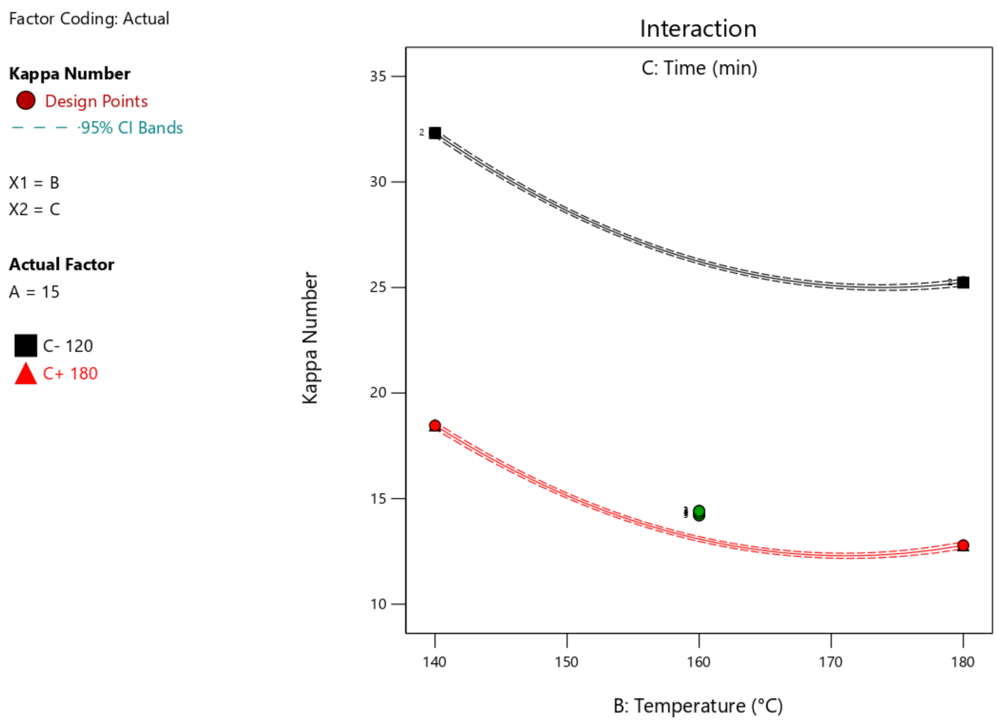

Factor Coding: Actual
Kappa Number
● Design Points
– – – 95% CI Bands
X1 = B
Actual Factors
A = 15
C = 150
One Factor
Warning! Factor involved in multiple interactions.
Kappa Number
35
30
25
20
15
10
140
150
160
170
180
B: Temperature (°C)

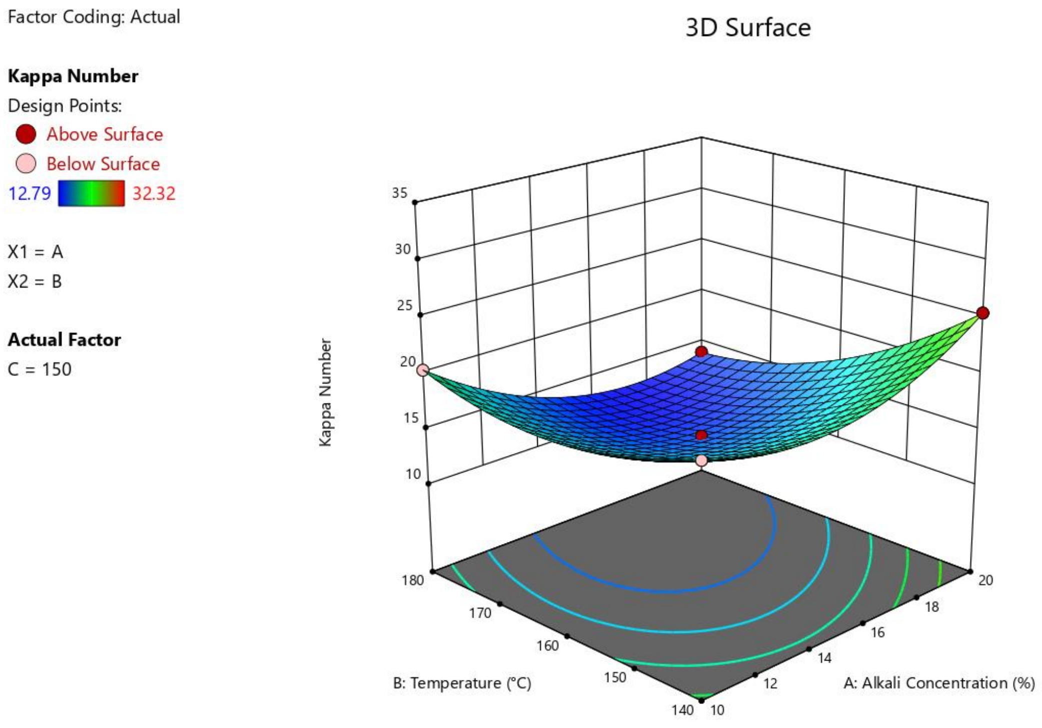

Factor Coding: Actual
Kappa Number
● Design Points
– – – 95% CI Bands
X1 = C
Actual Factors
A = 15
B = 160
One Factor
Warning! Factor involved in multiple interactions.
Kappa Number
35
30
25
20
15
10
120
130
140
150
160
170
180
C: Time (min)

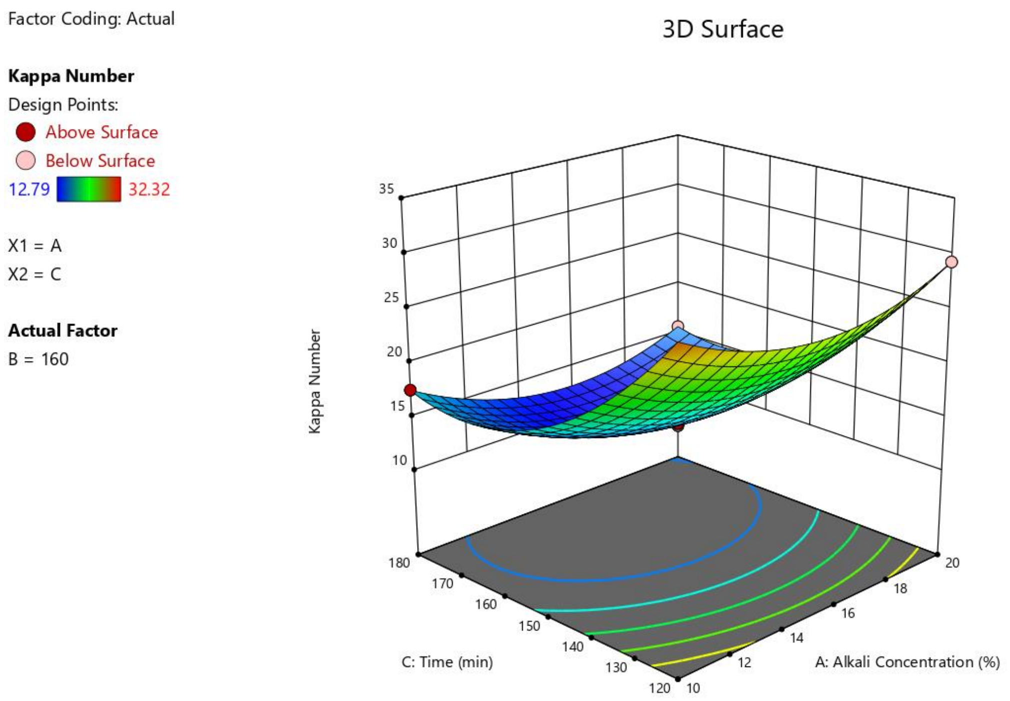

Factor Coding: Actual
Kappa Number
● Design Points
- - - - 95% CI Bands
Actual Factors
A = 15
B = 160
C = 150
Warning! Factor involved in multiple interactions.
Warning! Factor involved in multiple interactions.
Warning! Factor involved in multiple interactions.
Kappa Number
10
15
20
25
30
35
A: Alkali Concentration (%)
10
12
14
16
18
20
B: Temperature (°C)
140
150
160
170
180
C: Time (min)
120
130
140
150
160
170
180

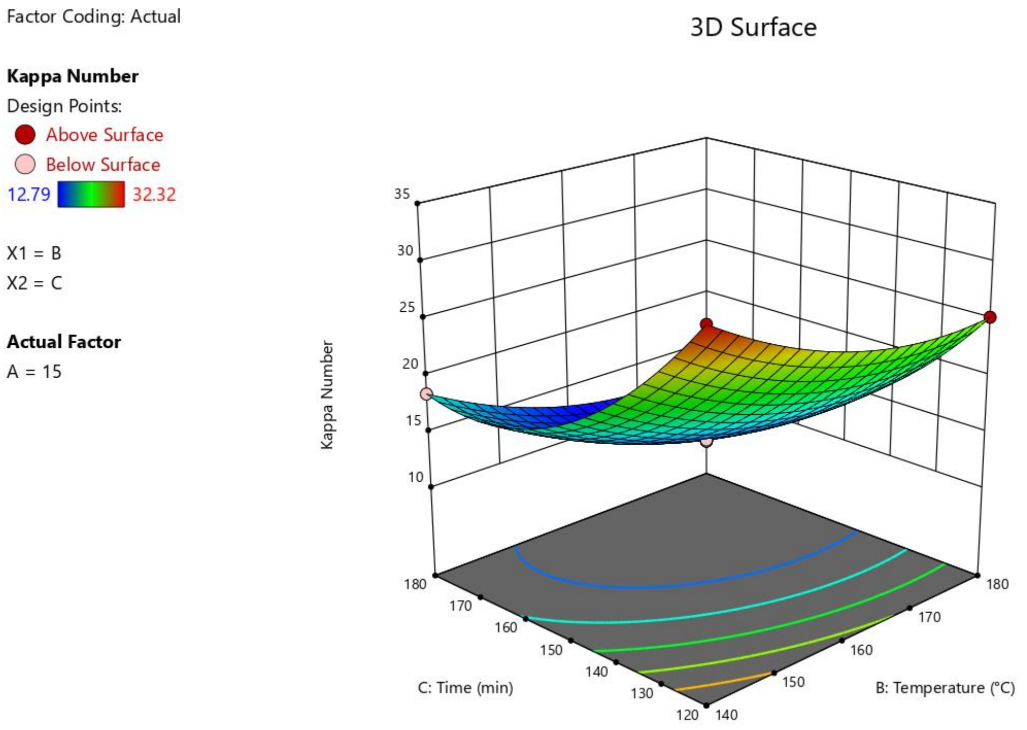

Factor Coding: Actual
Kappa Number
Design Points
95% CI Bands
X1 = A
X2 = B
Actual Factor
C = 150
B- 140
B+ 180
Interaction
B: Temperature (°C)
Kappa Number
35
30
25
20
15
10
10
12
14
16
18
20
A: Alkali Concentration (%)
2
2


Factor Coding: Actual
Kappa Number
Design Points
95% CI Bands
X1 = A
X2 = C
Actual Factor
B = 160
C- 120
C+ 180
Interaction
C: Time (min)
Kappa Number
35
30
25
20
15
10
10
12
14
16
18
20
A: Alkali Concentration (%)
2
2


Factor Coding: Actual
Kappa Number
● Design Points
– – – 95% CI Bands
X1 = B
X2 = C
Actual Factor
A = 15
■ C- 120
▲ C+ 180
Interaction
C: Time (min)
Kappa Number
35
30
25
20
15
10
2
140
150
160
170
180
B: Temperature (°C)


Factor Coding: Actual
Kappa Number
Design Points:
● Above Surface
○ Below Surface
12.79
32.32
X1 = A
X2 = B
Actual Factor
C = 150
3D Surface
Kappa Number
35
30
25
20
15
10
180
170
160
150
140
B: Temperature (°C)
10
12
14
16
18
20
A: Alkali Concentration (%)


Factor Coding: Actual
3D Surface
Kappa Number
Design Points:
● Above Surface
○ Below Surface
12.79
32.32
X1 = A
X2 = C
Actual Factor
B = 160
Kappa Number
35
30
25
20
15
10
C: Time (min)
180
170
160
150
140
130
120
A: Alkali Concentration (%)
10
12
14
16
18
20


Factor Coding: Actual
3D Surface
Kappa Number
Design Points:
● Above Surface
○ Below Surface
12.79
32.32
X1 = B
X2 = C
Actual Factor
A = 15
Kappa Number
35
30
25
20
15
10
C: Time (min)
180
170
160
150
140
130
120
B: Temperature (°C)
140
150
160
170
180

Appendix B: Pictures taken during the research work

1. Bamboo milling machine

2. Screening

3. Electrical autoclave

4. Pulp washing

5. Pulp beating machine

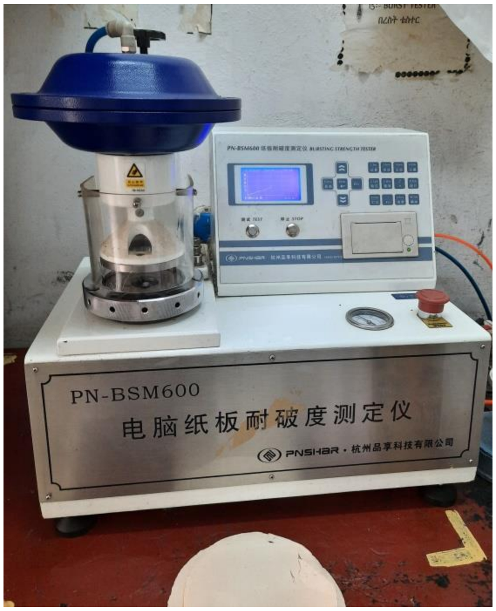
6. Pulp disintegrating machine

7. Sheet former

8. Sheet press

9. Tensile strength testing equipment

10. Burst strength testing equipment



Annex A: pulp and paper import data

Import data obtained from Ethiopian Revenue and Customs Authority (ERCA) (table 1 and 2 below) and data collected from FDRE’s Chemical and Construction Inputs Industry Development Institute (CCIIDI) show that the demand for different pulp products and the downstream product paper has increased by 24% and 33% respectively over the last five years (from 2015 to 2019).

Table -1: The last five years total imported pulp*

Year	Gross Wt. (Kg)	CIF Value (ETB)	CIF Value (USD)
2015	7,323,832.40	125,226,097.22	6,025,699.99
2016	7,949,797.12	138,462,101.92	6,310,023.24
2017	3,836,176.73	70,305,115.30	2,980,015.23
2018	6,465,125.01	146,093,521.65	5,410,871.17
2019	9,094,073.40	221,881,928.00	7,396,064.27
Average	**6,933,800.93**	**140,393,752.82**	**5,624,534.78**

* Source: Ethiopian Revenue and Customs Authority

Table -1 shows an average of 6.9338 million kilogram (6,933.8 tons) of pulp is imported each year and the country spends about 5.6 million American dollars for it.

Table 2: -The past five years average national paper import*

Year	**Gross Wt. (Kg)**	**CIF Value (ETB)**	**CIF Value (USD)**	**Total tax (ETB)**
2015	109,039,701.32	2,815,071,522.01	135,457,199.60	1,091,250,074.11
2016	152,425,105.08	3,665,370,032.41	167,038,993.06	1,458,822,360.16
2017	126,310,752.83	3,047,523,091.13	129,175,027.81	1,214,160,566.58
2018	136,587,493.36	3,906,963,711.07	165,604,043.33	1,505,890,402.23
2019	146,864,233.89	4,766,404,331.00	202,033,058.85	1,797,620,237.87
Average	134,245,457.30	3,640,266,537.52	159,861,664.53	1,413,548,728.19

* Source: Ethiopian Revenue and Customs Authority

Table 2 shows that an average of 134.245 million kilogram (134,245.5 tons) of paper is imported each year and the country spends an average of 160 million USD for it.