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**ADDIS ABABA UNIVERSITY
ADDIS ABABA INSTITUTE OF TECHNOLOGY
SCHOOL OF CHEMICAL AND BIO ENGINEERING
ENVIRONMENTAL ENGINEERING STREAM**

**TEXTILE WASTEWATER TREATMENT BY USING MORINGA SEED
AS A NATURAL COAGULANT**

**BY
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**June, 2015
Addis Ababa, Ethiopia**

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**A Thesis Submitted to the School of Graduate Studies of Addis Ababa University
in Partial Fulfillment of the Degree of Master of Science in Chemical Engineering
(Environmental Engineering Stream)**

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Acknowledgement

Above all my gratitude goes to the Almighty Allah for strengthening me in all of my moves from the beginning up to the end.

Next, I would like to express my gratitude to Eng. Gezachew Shiferaw who has provided me constructive advice, support, and encouragement during the accomplishment of this work.

My gratitude also extends to my family members, especially my father Hamid Saliya who has supported me both morally and materially while doing this research. On top of this I would like to mention and appreciate his contribution for collecting the Moringa seeds which are used in this study. Similarly, I am strongly indebted Emuye Hamid (my sister) who has provided me important support in my life.

Finally, I thank all persons who have direct or indirect contribution on the accomplishment of this work. On top of that Habib Jemal deserve special place for his endless support and encouragement from the beginning up to the end of this work. Similarly, Nuriya Jemal did so many good things for me during this long journey.

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Acronyms

AAiT	Addis Ababa University Institute of Technology
APHA	American Public Health Association
BOD	Biological Oxygen Demand
COD	Chemical Oxygen Demand
EEPA	Ethiopian Environmental Protection Authority
EPA	Environmental Protection Authority
KDa	Kilodaltons
LDC	Liquid Crystal Display
NTU	Nephelometric Turbidity Unit
PLC	Private limiting company
Pt-Co	Platinum Cobalt Unit
Rpm	Revolution per minute
TDS	Total Dissolved Solid
TS	Total Solid
TSS	Total Suspended Solids

Abstract

Currently, there are a number of textile factories which are operating in Ethiopia. Consequently, the magnitude of textile wastewater generation rate of the county has been increased. However, wastewater discharge practice in Ethiopia does not meet the national discharge standard. Because, there is a trend of discharging untreated or little treated textile wastewater into environment [3]. Therefore, to limit the bad consequence of such trend on environment it is indispensable to apply the most effective wastewater treatment system. In this connection, coagulation is one of the most effective treatment methods to remove toxic pollutants of textile wastewaters that technically need adding of either chemical or natural coagulant. However, use of chemical coagulants has been criticized to have some limitations. Consequently, recently there is an interest to replace chemical coagulants by natural materials.

The objective of this study was to evaluate the effectiveness of Moringa seed as a coagulant for the treatment of textile wastewater. This research followed quantitative approach and used primary data. Accordingly, experiments were carried out using jar tests apparatus by varying some operating parameters such as dose of coagulant, pH, and mixing time in order to study their effect as well as optimum condition on coagulation.

The optimum conditions were at 6g/l of coagulant dose, pH 4 and 45 minutes of mixing time with rapid mixing 100 rpm for 2 minutes and slow mixing 40 rpm for 45 minute and 30 minutes of settling time. At these optimum conditions 94 % color reduction, 90 % turbidity reduction, 82.7 % TDS and 86.8 % TSS reduction were obtained. Hence, it is recommended to use Moringa seed as coagulant since it is found effective for removal of color, turbidity, TDS and TSS from textile industry wastewater.

Keywords: coagulation, coagulant, textile wastewater, wastewater treatment Moringa seed, Moringa.

1. Introduction

1.1. Background

One consequence of economic development, which is part of human being's desire to improve quality of life, is environmental pollution [1]. For this reason there are efforts to protect environment at international, regional, national, and local levels via formulation and execution of policies, and regulations. In narrow sense pollution which arises from an industrial activity and its effect on water quality is a prime environmental concern throughout the world [2]. Similarly, in Ethiopia wastewater discharge from industrial activities is one of the major problems [3].

Textile industries are one of the most common and essential sector in Ethiopia. And wastewater discharge from these industries is one major issue which need due attention for the following reasons. On one hand, textile wastewater has enormous volume and composition of toxic pollutant [4]. Because on one hand the sector is expected to utilize (consume) about 200L of water to produce 1 kg of textile [5]. On the other hand, textile effluents are highly colored and saline as well as contain non-biodegradable compounds which have the potential result in high biological and chemical oxygen demand [6]. Besides, it also contains trace metals such as Chrome, Arsenic, Cupper, and Zinc, and oils, greases, and waxes [7]. As a response to the problem various wastewater treatment methods have been studied and applied [8]. But most of such methods are proved to be either too expensive or harmful due to usage of chemicals.

Coagulation is one of the effective textile wastewater treatment techniques. This technique is affirmed to remove, dissolved, and suspended matter as well as color from dyes which are responsible for turbidity as well as, organic matters which contribute to BOD and COD content of wastewater [9]. The method needs adding of chemicals such as salts of aluminum, iron and polyelectrolytes coagulants in to textile wastewater [10]. These chemical coagulants are acknowledged for their effectiveness on treatment of the wastewater. However the use of such chemical coagulants is not free from some limitations. The limitations include detrimental effects on human health and toxicity to aquatic life, production of large sludge volume and non biodegradable, ineffectiveness in low-temperature, change pH value of the treated water. [8] Besides, such chemical coagulants incur high procurement costs both for import and sludge disposal [11]. In recent years, there has been a move on the part of researchers to identify and apply natural materials instead of chemical coagulants [12]. In

this connection the Moringa seed is proved to be active, safe, and inexpensive coagulant to treat textile wastewater [13].

Moringa tree belongs to the family Moringaceae which is shrubs trees cultivated across the whole of the tropical belt including Ethiopia used for a variety of purposes such as food, medicinal, and others.[47] The seeds of the Moringa tree contain a coagulant protein that can be used in the treatment of industrial wastewater. Therefore, the purpose of this study was to evaluate the effectiveness of Moringa seed as a coagulant for textile wastewater treatment. The effect of some operating parameters on coagulation namely pH, coagulant dose and mixing time to remove color, turbidity, TDS and TSS was studied. Besides, the optimal conditions of such parameters were also investigated.

1.2. Statement of the Problem

Now days water quality change due to industrial pollution is one of the major environmental concerns throughout the world. Textile sector is one of highly water polluted industries because as several processes produce multi-component wastewater. It is difficult to treat textile wastewater and also expected to cause serious environmental problems due to their high color, large amount of suspended solids and high chemical oxygen demand [3, 6].

In Ethiopia textile sub sector is one area which is assumed to play promising role in economic development. There are a number of textile factories currently operating in Ethiopia which in turn increased the magnitude of wastewater generation rate of the textile sector. Despite this move effluent discharge practice does not meet the national discharge standard due to poor treatment enforcement of policies and regulations, and lack of awareness and experience [6]. Actually, there is a trend of textile wastewater direct discharge untreated or a little toxic effluent into nearby environment [3]. This trend is not appropriate and unsustainable as it possibly causes serious environmental problem. Thus, the wastewater management system of textile factories needs effective and efficient treatment system which is one important problem area to be researched.

There are several methods have been developed to treat textile wastewater, but most of them are either too expensive or harmful to environment. Coagulation, using natural coagulant, is one of the most effective and efficient textile wastewater treatment methods. As indicated above, textile wastewater treatment practice in Ethiopia is suffered from problems that should be researched. Therefore, this study examine the effectiveness of Moringa seed as a coagulant which is recognized to

be low-cost and eco-friendly material, and effective to remove color, turbidity, TDS, and TSS from textile effluent which is necessary for sustainable development.

1.3. Objective of the Study

1.3.1. General Objective

The general objective of this study is to evaluate the effectiveness of Moringa seed as a coagulant for the treatment of textile wastewater.

1.3.2. Specific Objectives

- Characterize the textile industry wastewater, based on COD, BOD, TDS, TSS, TS, color, turbidity, and pH.
- Determine the effect of the operating parameters, namely coagulant dose, pH and mixing time on the treatment performance of coagulation process.
- Determine the optimum value of these operating parameters.

1.4. Research questions

The following are the core questions of the researcher in this study.

- What are the COD, BOD, TDS, TSS, TS, color, turbidity, and pH level of the textile industry wastewater?
- Does Moringa seed effectively treat the textile wastewater?
- What is the effect of coagulant dose, pH and mixing time on the treatment?
- What is the optimum value of operating parameters like coagulant dose, pH and mixing time?

1.5. Scope of the Study

This study generally objected at assessing the effectiveness of Moringa seed as a coagulant in treating textile wastewater. Thus, this study does not concern methods and processes which may be applicable for textile wastewater treatment other than coagulation. Besides, this research does not attempt to examine every parameters of textile wastewater. Instead it is delaminated on parameters namely color, turbidity, TSS, and TDS. Furthermore, although there are different factors which can be studied in relation to coagulation process, this study pays attention on the major factors like coagulant dose, pH and mixing time.

1.6. Significance of the Study

This study is to introduce the application of Moringa seed as coagulant which is natural material. This natural coagulant has numerous advantages such as: safe for human health, environmental friendly behavior, locally availability as well as cost effective, produce lesser volume and biodegradable sludge, and unchanged pH value of treated water. The significance of the study is fully recognized in solving the environmental problems of the textile sector by suggesting a coagulation process using natural coagulant. Therefore, this research is beneficial in Ethiopia to have effective textile wastewater treatment method, by using Moringa seed, which has fundamental importance for maintaining people's health, protecting the quality of the environment and ultimately to reduce the costs of wastewater treatment chemicals.

1.7. Limitation of the Study

The study would be more and fully beneficial if it has assessed all parameters textile wastewater and factors affecting the coagulation process. However, because of resource and time constraints the researcher was not able to address all questions related to textile wastewater treatment and coagulation process. Hence, the researcher suggests that any further study on the area would benefit more if it continued on related areas not covered in this study. On the other hand there is also inadequate response in the chemical engineering laboratory to make ready the necessary chemicals both in quality and quantity. Besides, the absence of equipments which fit both the required quality and quantity are also another limitation, in addition to time limits on the use of the laboratory.

1.8. Organization of the Study

The paper is organized in such a way that the first chapter embrace the introductory part, statement of the problem, general and specific objectives, research questions, significance, its scope, limitation and organization of the study. The literature review is displayed in chapter two. The third chapter discusses the materials and methods were used to carry out this study. The fourth chapters assess the results & discussions the outcomes. The last chapter five consist of conclusion and recommendations part.

2. Literature Review

2.1. Introduction

In Ethiopia, recently, textile sub-sector is expected to play promising role in industrial development. According to current governmental strategy, textile industry is given more attention [14]. However, there is a practice of direct discharge of the untreated or partially treated effluent into environment [3]. This is not a good international practice, since textile effluents are highly colored and saline as well as contain non-biodegradable compounds. Therefore, the problem associated with textile wastewater discharge in Ethiopia need immediate solution.

In this chapter an attempt is made by the researcher to review literatures which are expected to provide theoretical underpinning for the problem sought to be achieved under this thesis. Eventually, issues like the major process of textile industries, the sources of wastewater, characteristics of wastewater, its environmental problems associated with industry wastewater, wastewater disposal standards, and the treatment options used in the treatment of textile wastewater were discussed. Lastly, special attention has been given to coagulation process using Moringa seed as a coagulant for textile wastewater treatment and factor affecting the process.

2.2. Major Categories of Textile Production Processes

In general, the overall processes of textiles can be categorized in to two major classes, namely the dry and the wet processing. Spinning and weaving/knitting are the major sub-processes of the former whereas sizing, desizing, scoring, bleaching, mercerizing, dyeing and printing are the major sub-processes of the latter [18]. On the following paragraphs an attempt is made to present explanations provided in literatures on the nature of each processes of textile production. Since the scope of this study is limited on textile wastewater it is found essential to pay attention on wet processing. Because, wet processing technique in a textile industry, expected to use large amount of water and results effluent production.

Thus, it is important to provide short discussion on the three sub-process stages which involve dry process. To start with spinning, it is a process which converts raw fiber into yarn or thread. The fibers are prepared and then drawn out and twisted to form the yarn, which is then wound onto a cone [5]. On the other hand, knitting is carried out by interlocking a series of yarn loops, usually using sophisticated, high speed machinery [19]. This process is almost completely dry, although some oils

may be applied during the process for lubrication. These are removed by subsequent processing and enter in to the wastewater stream. Finally, weaving is the most common method used for producing fabrics. This process is carried out on a loom which interlaces lengthwise yarns (warp yarns) with widthwise ones (weft yarns) [20].

Having said some textile production sub-processes that involve dry process let us see the natures of production stages which involve wet process. Firstly, sizing is a process which carried out before weaving process that uses starch to increase the tensile strength and smoothness of the yarn, to reduce yarn breakages [19]. Second, desizing process uses either acid or enzymes so as to remove size or starch the fabric, so that chemical penetration of the fabric in later stages is not inhibited [14]. Desizing effluents have very high organic concentrations, contributing 40 – 50 % of the total organic load from the preparatory sequences. Gums and polyvinyl alcohol may be removed by a simple hot wash but starch and its derivatives have to be made soluble by soaking with acids, enzymes or oxidants before being removed by a hot wash. Third, scouring process is carried out to remove impurities that are present in cotton, both natural (e.g. waxes, fatty acids, proteins, etc.) and acquired (such as size, dirt and oil picked up during processing). This is usually done at high temperatures (above 100 °C) with sodium hydroxide and produces strongly alkaline effluents (around pH = 12.5) with high organic loads. [20] They tend to be dark in color and have high concentrations of TDS, oil and greases. Common scouring agents include detergents, soaps, alkalis, antistatic agents, wetting agents, foamers, defoamers and lubricant. Fourth, bleaching process is used to improve the whiteness of the textile fabric and can be achieved using either hydrogen peroxide or sodium hypochlorite. The wastewater from bleaching process is low in BOD and COD, but high in TDS. Moreover, it may contain the colored impurities, removed from the fabric and is alkaline in nature [19, 5]. Five, under mercerizing process, cotton yarn or fabric is treated with an alkali (sodium hydroxide) to improve luster, strength and dye uptake [18]. It also removes immature fibers. The process is normally carried out on dry fabric; wet mercerization reduces the steam consumption, but requires stringent control of the operational parameters, such as sodium hydroxide concentration [22]. Six, dyeing process is used to add color and intricacy to textiles and increase product value. Some of the dyes also contribute to the complexes of toxic metals, like chromium and copper [23]. There are different types of dyes which are applicable in textile industries. Dyes can be classified into different types depending on their chemical compositions and properties. Therefore, the usage of dyes varies from industry to industry depending on the fabrics they manufacture. The most commonly used dye for cellulose

textiles is reactive dyes. The other types of dyes used are direct, vat, sulphur, indigo and naphthol dyes [3]. Seven, printing is a process that is used for applying color to a fabric. Unlike dyeing, it is usually only carried on prepared fabric where it is applied to specific areas to achieve a planned design [17]. The color is applied to the fabric and then treated with steam, heat or chemicals to fix the color on the fabric. Finally, finishing process refers to a process used to improve the quality of the fabric after dyeing [22]. Almost all the finishing processes consume extensive energy but their contribution towards wastewater generation is not significant.

2.3. The Sources of Wastewater in Textile industry

The main sources of wastewater normally come from cleaning water, pretreatment, dyeing and finishing process water, non-contact cooling water and others. The water consumption varies widely in the industry depending on the mill, processes, equipment used, and type of materials produced. Each textile processes utilizes large amount of water, which will finally become wastewater [24].

The most significant sources of wastewater among various process stages are pretreatment, dyeing, printing, and finishing of textile materials. At the pretreatment stage, desizing is the industry's largest source of wastewater [21]. During desizing, all the sizes used during weaving are removed from the fabric and discarded into the wastewater. In scouring, dirt, oil, waxes from natural fibers are removed from the fabric and washed into wastewater stream. Normally desizing and scouring are combined and these two processes may contribute to 50 % of BOD in the wastewater in the wet processing. Wastewater from the peroxide bleaching is not a major problem [25, 26]. Dyeing process generate the largest portion of the total wastewater. The source of wastewater is from the dye preparation, spent dye bath and washing processes. Dyeing wastewater contains high salt, alkalinity and color. Finishing processes generate organic pollutants such as residue of resins, softeners and other auxiliaries [27].

In general, wastewater generated by different production steps of a textile mill have a high pH, temperature, detergents, oil, suspended and dissolved solids, dispersants, leveling agents, toxic and non biodegradable matter, color and alkalinity [28].

2.4. The Characteristics of Textile Wastewater

The characteristics of textile effluents vary and depend on the type of raw materials, chemicals used, and the type of textile manufactured [16]. The textile wastewater contains high amounts of agents causing damage to the environment and human health including suspended and dissolved solids,

biological oxygen demand, chemical oxygen demand, heavy metal, odor and color [15]. Most of the BOD/COD ratios are found to be around 1:4, indicating the presence of non-biodegradable substances. Generally, the effluent from a textile industry is characterized by extreme fluctuations in many parameters such as COD, BOD, pH, strong color and salinity [6]. The composition of the wastewater will depend on the different organic-based compounds, chemicals and dyes used in the dry and wet-processing steps [5]. Recalcitrant organic, colored, toxicant, surfactant, chlorinated compounds and salts are the main pollutants in textile effluents. As indicated under chapter one the first specific objective of this research is to characterize textile effluent from sample taken from Ayka Addis textile industry of Ethiopia. Thus, it is possible to compare the final result of the experiment with the typical characteristics of textile effluent described below in table 2.2.

Table 2.1: Characteristics of textile mills wastewater [15]

S/No.	Physico-chemical parameter	Units	Range	Trace elements in (mg/l)	Range
1	pH	-	6-10	Iron	<10
2	Temperature	°C	35-45	Zinc	<10
3	Total dissolved solids	mg/l	8000-12000	Copper	<10
4	Biological oxygen demand	mg/l	80-6000	Arsenic	<10
5	Chemical oxygen demand	mg/l	150-12000	Nickel	<10
6	Total suspended solids	mg/l	15-8000	Boron	<10
7	Total dissolved solids	mg/l	2900-3100	Mercury	<10
8	Chlorine	mg/l	1000-6000	Manganese	<10
9	Free chlorine	mg/l	<10	Vanadium	<10
10	Sodium	mg/l	70%	Curium	<10
11	Oil & grease	mg/l	10-30		
12	NO ₃ -N	mg/l	<5		
13	Free ammonia	mg/l	<10		
14	SO ₄	mg/l	600-1000		
15	Silica	mg/l	<15		
16	Total Kjeldahl Nitrogen	mg/l	70-80		
17	Color (Pt-Co)	Pt-Co	50-2500		

2.5. Environmental Impact of Textile Wastewater

In terms of wastewater volume generation as well as composition textile industries considered to take the leading position. The improper and indiscriminate disposal of textile effluents in natural waters and land is posing serious problems [6]. The textile effluent contains organic and inorganic chemical species which have adverse effect on water quality and growth of all plants and animals. Its chemical intensive nature, applied at different processes, has caused environmental impacts at every stage in the life cycle of textiles industry [7]. Hence, every production process within a this industry has an environmental aspect that should be considered. If it is discharged into our environment, it results an immediate or long-term harmful effect on the environment. On the following paragraphs an attempt is made to see the response of literatures as to the environmental impacts arise from textile wastewaters discharge.

Recent studies have shown that environmental problems associated with textile activities mainly arise from the extensive use of organic dyes with complex and highly varied chemical structures [18]. The dyestuff lost through the processes of the textile industry poses a major problem for wastewater management. Primary concern about color is not only its toxicity but also its undesirable aesthetic impact on receiving waters [30]. Dyes in water give out a bad color and can cause diseases like hemorrhage, lung and skin irritations, headaches, congenital malformations and nausea [25]. They can block the penetration of sunlight from water surface preventing photosynthesis. Dyes also increase the biochemical oxygen demand of the receiving water and in turn reduce the reoxygenation process and hence hamper the growth of photoautotrophic organisms [26]. Besides, the dye of the effluent discharges into receiving water affects the aquatic flora and fauna. Some of dyes are carcinogen and others after transformation or degradation yield such as aromatic amines, which may carcinogen or otherwise toxic [6]. In addition, dyes accumulate in sediments at many sites, especially at location of wastewater discharge, which has an impact on the ecological balance in the aquatic system. These pollutants because of leaching from soil also affect ground water system [8]. Therefore, color is the first contaminant to be recognized in the dyeing effluents and has to be removed before discharging into the receiving environment.

Similarly, inorganic substances in the textile effluents make the water unsuitable for use due to the presence of excess concentration of soluble salts. These substances even in a lower quantity are found to be toxic to aquatic life [4]. Some of the inorganic chemicals like hydrochloric acid, sodium

hypochlorite, sodium hydroxide, sodium sulphide and dyes are poisonous to marine life. The organic components are found to undergo chemical and biological changes that result in the removal of oxygen from water [1]. The suspended solid concentrations in the effluents play an important role in affecting the environment as they combine with oily scum and interfere with oxygen transfer mechanism in the air-water interface [31]. Also the discharge of organic pollutant like COD or BOD to the receiving stream can lead to the depletion of dissolved oxygen and thus creates anaerobic condition. Under anaerobic condition foul smelling compound such as hydrogen sulfides may be produced. This will consequently upset the biological activity in the receiving stream [24].

The textile mill also generate effluent contain heavy metals like Cadmium, Chrome, Cupper, Lead, Nikcel, and Zink etc which are capable of harming the environment [6]. The water containing textile effluent used for irrigation contains heavy metals, which accumulate in various parts of plants that result in various clinical problems in animals as well as in human beings through food web [15]. The presence of heavy metals in the effluent has a cumulative effect, and higher possibilities for entering into the food chain [1]. The inorganic heavy metal are directly absorbed by the marine and fresh water biota or are incorporated in ground water in both cases i.e. the polluted ground water and the marine foods which are being used extensively by human being may lead to the diseases like cancer, tumor, brain and liver diseases, psychiatric diseases, and sexual diseases [16].

In sum textile wastewater has bad environmental consequences. This consequence is expected to be high in Ethiopia due to different reasons. These include there is a trend to discharge industrial wastewater directly to water body or land, instead of employing waste treatment [2]. In addition, there are a number of investments on textile sub-sector in Ethiopia which in turn increased the amount of effluent generation rate significantly as well as its environmental impact. In short, to limit the above bad environmental consequences that emanate from textiles wastewater, it is important to apply appropriate treatment methods, the major ones are discussed in the next section.

2.6. Textile Wastewater Disposal Standards

While there are many technologies available in treating the textile wastewater, a majority of them are relatively expensive to be applied by the small and mid-size industries. Furthermore, many of the physico-chemical technologies only transform the pollutants from one form or one phase to another and therefore do not provide any ultimate solution to the problem [3].

Several environment protection agencies worldwide have imposed rules entrusted with the protection of human health and guarding the environment from pollution caused by the textile industry. These agencies imposed certain limits on the disposal of effluents into the environment [15]. The disposal limits are found to differ from country to country [16]. However, a constant check is to be kept on these discharge limits every now and then to maintain a safe and a healthy environment. It is urgently required that industries should continuously monitor textile effluents and take necessary actions to properly treat the wastewater prior their disposal to water bodies and save already depleting natural water resources. The Ethiopia EPA emission limits for textile industry describe in appendix I. The treatment methods in textile industry wastewater, the major ones are discussed in the next section.

2.7. Textile Wastewater Treatment Methods

The variety of raw materials, chemicals, processes and also technologic variations applied to the processes cause complex and dynamic structure of environmental impact of textile industry [19]. These dynamic structures affect the treatability and the applied treatment. The concentrations of pollutants in textile wastewater vary depending upon the wastewater management practices and amount of water used in the production [1]. Therefore, it is of overriding significance to implement an appropriate treatment processes.

There are many ways for treating the textile effluent. The best combination of methods differs from plant to plant depending on the size, type of waste and degree of treatment needed. Generally, the treatment options used in the treatment of textile wastewater are divided into three main categories namely biological, physical and chemical methods [32]. Since textile wastewater are complex, several factors such as dye type, wastewater composition, dose and costs of required chemicals, operation costs, environmental fate and handling costs of generated waste products determine the technical and economic feasibility of each treatment option [33].

2.7.1. Biological Methods

There are different types of biological treatment methods. Among them include trickling filters, activated sludge process, aerobic/anaerobic process, sequencing batch reactor, oxidation ponding etc [1]. To date the commonest treatment of textile wastewater has been based on mainly on aerobic. Biological process consisting mainly conventional, and extended activated sludge system [3]. The trickling filters simulate stream flow by spraying wastewater over a broken, medium such as stone or

plastic. The medium serves as a base for biological growth, which attacks the organic matter of wastewater, and uses it as food.

In activated sludge process, the wastewater flows into a tank after primary settling. The microorganism in activated sludge is suspended in the wastewater as aggregates. The sludge and wastewater is kept in suspension by compressed air, which also supplies the oxygen, necessary for biological activities [34]. The aerated waste is continuously withdrawn and settled and a portion of the sludge is returned to the influent.

Normally the methods is to removing pollutants from wastewater using natural process involving bacteria and other microorganism for oxidation of the organic waste. Biological methods are generally cheap and simple to apply and are currently used to remove organic and color from dyeing and textile wastewater [35]. Most of existing process includes an initial step of activated sludge treatment to remove the organic matters, followed by oxidation, membrane, activated carbon etc.

Anaerobic and aerobic treatments have been used together or separately for the treatment of textile effluents. Hence aerobic treatment is not effective in color removal from textile wastewater containing azo dyes [4].

Generally, biological treatment methods are not effective for treating dyestuff wastewater because many commercial dyestuff are toxic to organism being used and result in the problems of sludge bulking, rising sludge and pin flock [32]. Biological treatment of dyeing wastewater is cheaper than other methods, but it is less efficient for decolorization due to toxicity of the wastewater and the need for an aeration system [36]. The disadvantage of biological treatment processes are: (a) the presence of toxic heavy metals in the effluent which affects the growth of microorganism (b) most of the dyes used are a non-biodegradable in nature and (c) the long time required for treating the effluent [24].

2.7.2. Physical Methods

Physical method include adsorption, membrane filtration such nano filtration, and reverse osmosis. Adsorption is an effective method of lowering the concentration of dissolved dyes in the effluent resulting in color removal [30]. The adsorption on activated carbon without pretreatment is impossible because the suspended solids rapidly clog the filter. This procedure is therefore only feasible in combination with flocculation-decantation treatment or a biological treatment. The

combination permits a reduction of suspended solids and organic substances, as well as a slight reduction in the color, but the cost of activated carbon is high [3].

Membrane filtration is an effective mean of removing pollutant from the wastewater. But initial investment cost is very huge and the periodic replacement of membrane is remarkably high [32].

Ultra-filtration enables elimination of macromolecules and particles, but the elimination of polluting substances, such as dyes, is never complete; it is only between 31 % and 76 %. Even in the best of cases, the quality of the treated wastewater does not permit its reuse for sensitive processes, such as dyeing of textile emphasize that 40 % of the water treated by ultra-filtration can be recycled to feed processes termed “minor” in the textile industry in which salinity is not a problem [1]. Ultra-filtration can only be used as a pretreatment for reverse osmosis or in combination with a biological reactor.

Micro-filtration is suitable for treating dye baths containing pigment dyes, as well as for subsequent rinsing baths. The chemicals used in dye bath, which are not filtered by micro-filtration, will remain in the bath. It can also be used as a pretreatment for nano-filtration or reverse osmosis [37].

Reverse osmosis membranes have a retention rate of 90 % or more for most types of ionic compounds and produce a high quality of permeate. Decoloration and elimination of chemical auxiliaries in dye house wastewater can be carried out in a single step by reverse osmosis [6]. It permits the removal of all mineral salts, hydrolyzed reactive dyes and chemical auxiliaries. It must be noted that higher the concentration of dissolved salt, the more important the osmotic pressure becomes; therefore, the greater the energy required for the separation process [8].

2.7.3. Chemical Methods

The method includes coagulation or flocculation and oxidation and etc. Most chemical treatment methods have been studies focusing on color removal although effective but are expensive or can cause further secondary pollution [9].

Due to the lack of proper wastewater treatment systems underdeveloped communities, the best immediate option is to use simple and relatively cost effective point-of-use technologies such as coagulation [38]. Coagulation is an essential process in the treatment of industrial wastewater. The main advantage of the conventional coagulation and flocculation is removal of the waste stream due to the removal of dye molecules from the dye bath effluent and not due to partial decomposition of

dyes which can lead to an even more potentially harmful and toxic aromatic compound [35]. More about coagulation process in wastewater treatment is discuss in the next section.

Chemical oxidation uses strong oxidizing agents such as hydrogen peroxides, chlorine and others to force degradation of resistant organic pollutant [39]. Chemical oxidation is the most commonly used method of decolourization by chemical owing to its simplicity and the main oxidizing agent is hydrogen peroxide. Oxidation of by ozone is capable of degrading chlorinated hydrocarbons, phenol, pesticides and aromatic hydrocarbon. One of the major advantage of ozonation it does not increase the volume of wastewater and sludge. A major disadvantage of this process is its short-half life typically being 20 minutes [20]. Table 2.2 presents the pros and cons among the major textile wastewater treatment methods.

Table 2.2: Pros and cons of textile wastewater treatment methods

No.	Processes	Advantages	Disadvantage
1	Biodegradation	Rates of elimination by oxidizable substances about 90%	Low biodegradability of dyes
2	Coagulation-Flocculation	Elimination of insoluble dyes	Production of sludge blocking filter
3	Adsorption on Activated carbon	Suspended solids and organic substance well reduced.	Cost of activated carbon
4	Ozone treatment	Good decolonization	No reduction of the COD
5	Electrochemical processes	Capacity of adaptation to different volumes and pollution loads.	Iron hydroxide sludge
6	Reverse Osmosis	Removal of all mineral salts hydrolyzes reactive dyes, and chemical auxiliaries.	High pressure
7	Nanofiltration	Separation of organic compounds of low molecular weight	-
8	Ultrafiltration-microfiltration	Low pressure	Insufficient quality of the treated wastewater

Physical-chemical treatment allows reducing dissolved, suspended, colloidal and non settleable matter as well as coloring from dyes [9]. These treatment methods are effective to remove color but in comparison with biological treatment processes it consumes more energy and high chemicals [3]. It requires additional treatment process for the disposal of pollutants into solid or liquid side streams. The disadvantages of the physico-chemical treatment process are: (a) the generation and disposal of sludge and (b) the required space [35].

2.8. Coagulation Process

In industrial wastewater different compounds are present like suspended, colloidal and dissolved solids. Suspended solids have a diameter larger than 10^{-6} m, colloidal solids between 10^{-9} m and 10^{-6} m and dissolved solids smaller than 10^{-9} m [40]. This material must be removed prior to discharge. Because of the nature of the colloidal suspension these particles will not sediment or be separated with conventional physical methods (such as filtration or settling) unless they are agglomerated through coagulation [13].

Coagulation is the one of the most popular unit operations in water and wastewater treatment [8]. This process is physico-chemical operations used to facilitate the separate of suspended and colloidal solids from the wastewater. Coagulation is frequently applied to process in the primary purification of industrial wastewater and in some cases in secondary and tertiary treatment. It is the main component of wastewater treatment units and the applications include wastewater treatment, recycling and removal of pollutants [41]. Coagulation process is the destabilization of colloidal particles brought about by the addition of a chemical reagent known as a coagulant. Flocculation is the agglomeration of destabilized particles into microfloc, and later into bulky floccules which can be settled called floc. In commonly, the coagulation-flocculation process consists in destabilizing colloids, aggregating and binding them together into flocculates; the resulting flocs can finally be removed either by settling or flotation [34].

Coagulation, flocculation and clarification, followed by filtration, which are the key steps in wastewater treatment systems [40] are described in Figure 2.2. The coagulation process includes dosing of a coagulant in water. And it results in the destabilization of water. It occurs in several steps intended to overcome the forces that stabilizes the suspended particles, allowing particle collusion and growth of floc [8].

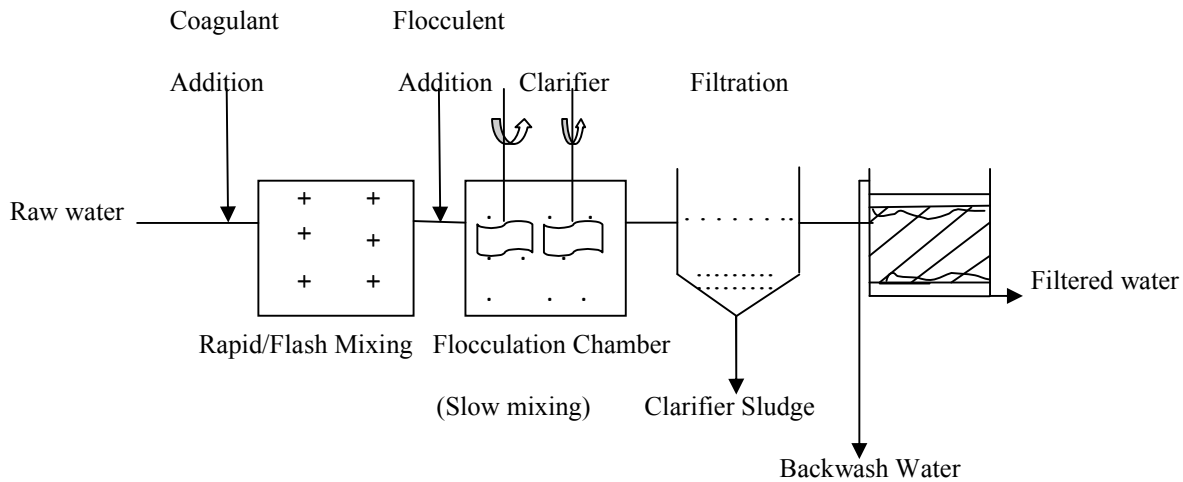


Figure 2.1: Coagulation, sedimentation and filtration steps

The first step in the coagulation process is destabilizing the particle charges in the water. Coagulant of opposite charges added to neutralize the negative charge on the dispersed non settable solids. Once the charge is neutralizes, the smaller particles are capable of sticking together to form a slightly larger particles. Rapid and slow mixing after coagulant dosing is a design parameter. Rapid mixing ensures the coagulant is properly dispersed in the water and promotes particle collision that is needed to achieve good coagulation. Slower mixing helped in promoting floc formation by enhancing particle collisions which led to larger flocs [32].

Depending on the type of colloidal suspension that should undergo coagulation different destabilization mechanisms can be employed such as:

- Repression of the double layer.
- Neutralization of colloid charge by adsorption of counter ions on the surface of the colloid.
- Bridging of colloidal particles via polymer addition.
- Entrapment of colloidal particles by sweeping floc.

The addition of certain chemicals into the raw water causes particles to destabilize and allows agglomeration and floc formation to occur [40]. The general terms used for this purpose are:

- Coagulants, which assist the destabilization of particles (particularly colloidal sizes).
- Flocculants (also known as flocculent aids or coagulant aids), which assist in the joining and enmeshing of the particles together.

In textile wastewater treatment coagulation-flocculation are a commonly treatment methods that can be employed to decolorize effluent and also to reduce the total load of pollutants [37]. The main advantage of these methods is cost effective, simple to operate and energy saving treatment alternative [35]. The inherent disadvantage to this process is the generation of large quantities of chemical sludge and its classification as hazardous waste, necessitating the need for secured land filling of hazardous solid wastes [34]. For this reason, alternative natural materials have been considered for environmental applications. In this study the researcher used the term coagulation, in line with the common trend, to represent the whole process starting from adding of coagulant in wastewater up to their settlement.

2.8.1. Factors Affecting the Effectiveness of Coagulation

The effectiveness of the coagulation is influence by factors such as wastewater type, initial turbidity, temperature, physical and chemical characteristics of coagulant, pH of the solution, the type and dosage of coagulants/flocculent, mixing speed and time, settling time and etc [9]. The review of literatures is limited on coagulant dose, pH and mixing time since they are variables the coagulation experiment in this study. On the following three paragraphs an attempt is made to discuss these parameters one by one.

The coagulant dose is one of the most important parameter that has been considered to determine the optimum condition for the performance of coagulants in coagulation-flocculation process [52]. Essentially, insufficient dosage or overdosing would result in the poor performance in process. There is a range of optimum doses for a coagulant at which maximum settling and removal of pollutants is most effectively achieved. Below this range, the amount of coagulant added is inadequately to destabilize the particles. Above this range, the coagulant essentially serves as a chemical coating which re-stabilizes the particle [8]. Therefore, it is significant to determine the optimum dose in order to minimize the dosing cost and sludge formation and also to obtain the optimum performance in treatment process. Since the range coagulant dose varies with the type of coagulant, the dose Moringa is studied under this research.

The pH is also the second important parameter that has been considered to determine the optimum condition for the performance of treatment [50]. In coagulation process pH will not only affect the surface charge of coagulants, but also the degree of stabilization of the suspension. Besides, pH seems an interesting variable which has a greater influence on the pollutants removal efficiency.

Therefore, effect of pH was one of the parameters was crucial to determine the optimum level in order to minimize the sludge formation and obtain the optimum performance in treatment. Thus, pH must be controlled, by adding either strong acid or base, to establish optimum condition for coagulation process.

In addition to coagulant dose and pH, the effect of mixing time also play an important role on flocs formation and growth in coagulation-flocculation process [37]. The time of macrofloc formation (flocculation time) is one of the operating parameters that is given great consideration in any water and wastewater treatment plant that involves coagulation [36]. Therefore, it is important to determine the optimum mixing time for the treatment system.

There are typically three phases of mixing in a coagulation process: rapid mix, slow mix, and no mix. The rapid mixing phase is a short period of extremely turbulent mixing that allows coagulants contact with suspended particles [45]. The next phase is flocculation time . It is characterized by slow mixing and allows destabilized particles to agglomerate together into larger particles [46]. This mixing period is important for floc formation. Flocs are highly fragile, so sudden movements lead to floc breakage and therefore to a loss of effectiveness. The final phase consists of no mixing. It allows flocculated particles to settle out of the system. Insufficient periods of slow mixing result in poor agglomeration of particles. Prolonged agitation periods, however, lead to ruptures in floc fragments and dis-agglomeration of particles.

In general, optimization of coagulant dose, mixing time and pH is significantly increases the effectiveness and efficiency of coagulation so that it is recommended to take in to consideration. Actually, in this study these three determinant factors are examined.

2.8.2. Types of Coagulants

A viable pre-treatment technology for reducing the pollution problems is the physico-chemical treatment using coagulants and this has been successfully applied to different wastewaters [11]. The coagulants used play a major role in the treatment of wastewater and disposal of sludge [32]. Selecting a coagulant for treatment of water and wastewaters is a function of several factors which include the nature of the suspended particles, raw water and wastewater condition, design of treatment facility, cost of coagulant chemicals, zeta potential (the amount of repulsive forces which ensure that particles stay in water environment), and Van der Waal's forces (weak inter-molecular

forces that influence particle formation) [42]. For any treatment process to be effective, attention must be paid to required effluent quality, sludge handling disposal cost (chemical coagulants generate sludge), and overall dosage cost of coagulant [28].

There are two types of chemicals coagulants used in coagulation process. These include; primary coagulants and coagulant aids [34]. Primary coagulants are involved in neutralize the electrical charges of particles in the water which causes the particles to clump together. Coagulant aids add density to slow-settling flocs and add toughness to the flocs so that they will not break up during the mixing and settling processes [41]. Primary coagulants are always used in the coagulation/flocculation process. Coagulant aids, in contrast, are not always required and are generally used to reduce flocculation time.

Coagulants used in wastewater treatment can be classified into inorganic coagulants (e.g. Aluminum sulphate (Alum), polyaluminum chloride, Ferrous sulphate, and ferric chloride), synthetic organic polymers (e.g. polyacrylamide derivatives and polyethylene amine) and naturally occurring coagulants (e.g. Chitosan, Moringa and etc) [39]. On the following paragraphs an attempt is made by the researcher to discuss the characters and application of some of the major types of coagulants.

As indicated above the type of coagulant used is one determining factor for effective wastewater treatment. In this relation, application the aforementioned most common type of chemical coagulants has some advantages and disadvantages.

Alum is common name for Aluminum Sulphate also known as Sulphate of alumina, and the most widely used coagulants in water and wastewater treatment all over the world because of effectiveness [35]. However, numerous recent studies have raised doubts about introducing aluminum into environment. Also, some studies have reported that this salt is characterized in poisoning factor for encephalopathy, and impact of aluminum on human health; it is strongly evidenced aluminum based coagulants linked in the development of neurodegenerative ill-nesses as sesil dementia and with Alzheimer's disease [12]. There is also the problem of reaction of alum with natural alkalinity present in the water leading to a reduction of pH, and low efficiency in coagulation of cold waters in addition very high sludge formation and disposal cost also non biodegradable [11].

The iron salts are also the most commonly used as coagulants include ferric sulfate, ferric chloride and ferrous sulfate. The iron compounds possess pH coagulation ranges and floc characteristics

similar to aluminum sulfate [11]. The cost of iron compounds may often be less than that of alum. However, the iron compounds are generally corrosive and often present difficulties in dissolving, and their use may result in high soluble iron concentration in process effluents [43]. Iron salts have also been used as coagulant but with limited success, because of same disadvantages as in the case of aluminum salts.

Synthetic organic polymers are the other type of coagulant which has widespread application. This type of coagulants is non-biodegradable by nature. Consequently, some researches indicate that the sludge remained after using syntactic polymers cannot be recycled, or cannot be used as fertilizers or animal feed [4, 39].

In sum chemical coagulants used for treatments process are harmful effects to human being and environments. Therefore, to solve such problem arise from chemical coagulants there is a move to investigate and discover alternative natural coagulants. Several studies have pointed out the introduction of naturally prepared coagulants produced or extracted from microorganisms, animals, or plants [44, 45]. Moringa seed, Nirmali, Okra, Beans, Rice and Maize are the type of coagulants which have been identified from plant. On the other hand, chitosan is affirmed to be good coagulant which has animal origin [9].

Other studies also indicated the presence of four types of plants which have been extensively used as coagulants viz. Moringa seed, Nirmali seeds, Tannin and Cactus has been used for successfully removed pollutant from industrial wastewater [45, 38]. In coagulation Moringa seed has been recognized to remove industrial pollutant namely from textile, dairy, brewery, and tannery [8, 12, 9]. The literature review confirmed Moringa seed to be effective coagulant for wastewater treatment. In the next part an attempt is made to see its application in coagulation.

2.9. Application of Moringa Seed as Coagulant

Literatures show that Moringa tree is found in India, Asia, Latin America, and Sub-Saharan Africa-including Ethiopia. The type of moringa tree that grow in Ethiopia is scientifically named as *Moringa stenopetala*, locally as *shiferaw*. A *Moringa stenopetala* type is often referred to as the African tree because it is native only to southern Ethiopia and northern Kenya [48]. In Ethiopia it grows widely in southern part namely in Keffa, Gamo Gofa, Bale, Sidamo, Arba Minch, Wollayta, Borana, Dehub Omo Zones, Konso and Dherashe areas [47]. The type Moringa tree found in Ethiopia is shown in

Figure 2.3. Moringa is found to grow as high as 2200m and as low as 300m in Ethiopia. It is an extremely fast-growing tree and continued to grow during the exceptionally long dry season.



Figure 2.2: Moringa stenopetala tree found in Ethiopia

Moringa is considered as a multipurpose tree. It is also considered as miracle of nature because it has so many benefits. [12] All parts of the tree except the wood are edible, providing a highly nutritious food for both humans and animals. The flowers are a good nectar source for honey; can be eaten or used to make a tea and the seeds are rich oil sources for cooking, lubricant and different purpose. The wood is very soft; useful for paper but makes low-grade firewood and poor charcoal. Many parts of the plant have been used in medicinal preparations [46]. Recently decades the application of the seeds of Moringa tree as a coagulant to treat water and wastewater has attracted attention.

As many researchers conducted using Moringa seed for water and wastewater treatment on the seed itself and cake powder. As the studies reported the Moringa seeds have content of protein have (26.50% - 32.00%), fiber (5.80% - 9.29%), ash (5.60% -7.50%), fat (42% - 45%) and moisture contents (8.7% - 9.1%) [54]. The study also indicated that Moringa seed contain 45% oil and 55% seed cake powder [48]. In this study used seed cake powder because the contribution of the seed cake powder can be considered as value added because it is a by-product of the oil extract from seeds. The Moringa seed cake (defatted) had higher values in the ash, crude fiber, protein, and carbohydrate contents than seed powder (undefatted) which shows in table 2.3 [39].

Table 2.3: Composition of Moringa seed and cake powder

No.	Parameter	Moringa seed powder	Moringa seed cake powder
1	Moisture content (%)	4.70 ± 0.05	5.03 ± 0.10
2	Ash content (%)	4.10 ± 0.14	10.00 ± 0.21
3	Crude fiber content (%)	7.73 ± 0.35	12.96 ± 0.40
4	Crude fat content (%)	45.84 ± 0.17	3.06 ± 0.11
5	Crude protein content (%)	28.04 ± 0.67	50.80 ± 0.45
6	Carbohydrate content (%)	10.59 ± 0.22	18.15 ± 0.22

The Moringa seeds contain a coagulant protein that can be used either in drinking water clarification or industrial wastewater treatment. This protein can therefore be used as a non-toxic natural polypeptide for purification of water. From environmental scientific community onwards it is suggested that its active coagulating agents are dimeric cationic proteins with molecular mass of 12–14 Kilodaltons and isoelectric point between 10 and 11 and its main coagulation mechanism is adsorption and charge neutralization. However, there have been conflicting reports on the exact nature of the active coagulating agent present in Moringa seed. The reports that the coagulating agent is a protein with a molecular mass of 6.5 Kilodaltons and isoelectric point greater than 10 while suggests that it is a cationic protein with isoelectric point greater than 9.6 and molecular mass less than 6.5 kDa [49]. Conversely, other study argues that the active component from an aqueous salt extraction is not a protein, polysaccharide or lipid, but an organic polyelectrolyte with molecular weight of approximately 3.0 Kilodaltons [35]. Although most research groups seem to agree that the active agent of coagulation are dimeric cationic protein.

Many researchers stated that Moringa seeds have to be one of the most effective natural coagulants. The coagulant has to be applied to transform water constituents into forms that can be separated out physically. The seed of Moringa contain significant quantities of high molecular weight water-soluble proteins which carry a positive charge [13]. When the crushed seeds added to raw water, the protein produces positive charges acting like magnets and attracting the predominantly negatively charged particles (such as clay, silt, bacteria, and other pollutants). Under proper agitation these bound particulates then grow in size to form the flocs, which may be left to settle by gravity or be removed

by filtration [28]. The flocculation process occurs when the proteins bind the negative charges forming floc through the aggregation of particles which are present in water, and flocs are easily removing by filtration [9]. Many previous studies arguably stated that the coagulation mechanism of Moringa seed coagulant protein has been explained in different ways. It has been described as adsorption, charge neutralization and interparticle bridging of the colloidal positive charges that attract the negatively charged impurities in wastewater [45].

The different studies have acknowledged the applicability of Moringa seed as one type of coagulant in industrial wastewater treatment. Besides, it is affirmed to be most effective and viable coagulant wastewater treatment [50]. The same recognition is given by numerous researchers on its application for the treatment of different effluents such as color, phenol, COD, BOD, Cr, TDS, and turbidity from different industries [38, 43, 48]. In connection to this, other has proved it to reduced color, conductivity, absorbance, turbidity, COD, BOD, TDS, and TSS from textile industries [8, 12, 28]. The literature survey has give conformity on the applicability and effectively of Moringa seed acts as coagulants, adsorbents, and antimicrobial agent to remove different pollutants from different industries.

In comparison with chemical coagulants, Moringa seed is considered favorably in terms to reduce the costs of wastewater treatment in developing countries [50]. In addition the sludge produced by Moringa seed as a coagulant is stated to be innocuous and 4-6 times less in volume than the chemical coagulants produced [46]. Besides, if the particulates are removed the sludge that is generated is proven to be non-hazardous and useful for fertilizer and/or soil conditioner after stabilization [28]. Furthermore, the effectiveness of Moringa seed for treating textile effluents is also explained in relation to its availability in many parts of the world and also has a number of advantages [8].

Therefore, the natural coagulants like Moringa seed have bright future and are concerned by many researchers because of their effectiveness, abundance, low cost, eco-friendly, and biodegradable nature in wastewater treatment. Consequently, the consecutive chapters of this paper are focused on coagulation experiment using Moringa seed as coagulant to remove color, turbidity, TSS and TDS from textile wastewater.

3. Materials and Methods

3.1. Materials

The following materials and methods were used to carry out this study.

a) Equipments and Materials

The equipment and tools used throughout the experiments to achieve the objective of the study are: jar test apparatus, BOD incubator, COD analyzer, oven, desiccators, vacuum filter, measuring cylinder, beakers, magnetic stirrer, weight balance, pipit, crucible, domestic mill, sieve, filter paper, pH meter, HACH model spectrophotometer and HACH turbidimeter were used during the experiment work.

b) Chemicals and reagents

The following chemicals are also used during the experimental works such as: NANO COLOR CSB 1500 as COD reagent, to test BOD potassium dichromate and Nitrification inhibitor were used to absorb carbon dioxide gas and to avoid nitrification respectively. Besides, hydrochloric acid & sodium hydroxide are used to adjust pH during the experiment, and ethanol is used to extract oil from Moringa seed. All of these chemicals were obtained from environmental engineering laboratory of AAiT, with exception to ethanol which is purchased from Neway PLC (Addis Ababa, Ethiopia).

3.2. Methods

The methods used to reach at each specific objectives of this study are described as in the following sub-titles.

3.2.1. Sample Collection and Study Area

In this study the sample of wastewater was collected from Ayka Addis textile industry, located in Alemgena town, 19 km from Addis Ababa in south west which is the largest textile industry in Ethiopia. The industry has wastewater treatment plant which has the potential (capacity) to treat of 5760 m³ wastewater day⁻¹.

Wastewater samples were collected, by using Grab sampling technique, from the inlet of the treatment plant on February 9-2015 at 10:00 am and March 3 at 12:00 am and April 27-2015 at 4:00 pm. Plastic containers were used for sample collection after cleaned with detergent, rinsed with tap water, soaked in 10% HNO₃, and rinsed with deionised water. After sample collection and it was

transported in to AAiT laboratory and preserved in a refrigerator at 4°C in order to minimize the chance of their characteristics changes until used for experiment.

3.2.2. Characterization of Textile Wastewater

It is necessary to identify the concentration of physico-chemical parameters before conducting treatment experiments. Thus, an initial experiment is carried out to determine the character of Ayka Addis textile wastewater in term of parameters namely BOD, COD, TSS, TDS, Color, TS, turbidity and pH. This experiment is conducted in chemical engineering laboratory of AAiT based on procedures which are provided under standard methods for the examination of water and wastewater [51]. Some of the experimental procedures which are relevant with the objectives of this study are attached in Annex IV.

3.2.3. Coagulant Preparation

For the purpose of this study the Moringa seeds are collected from Yabelo town which located at the Southern part of Ethiopia (Oromia Regional State). It is possible to use the Moringa seed as coagulant either by the normally seed or extracting the oil from the seed (defatted cake) [15]. To enhance effectiveness on wastewater treatment some studies has recommended the importance of using defatted Moringa seed [12, 44]. In line with this recommendation defatted Moringa seed was prepared, for the purpose of this study, based on the following procedure. See Figure 3. shows the overall steps followed on coagulant preparation. First, the seeds covered with pods were separated manually. The seed with external shells dried in the sun because the green seeds do not possess any coagulation activity. Then seeds are also separated manually from the external shells after dried [28]. See Figure 3.2 shows the pods, seed, and seed kernels of the Moringa stenopetala.

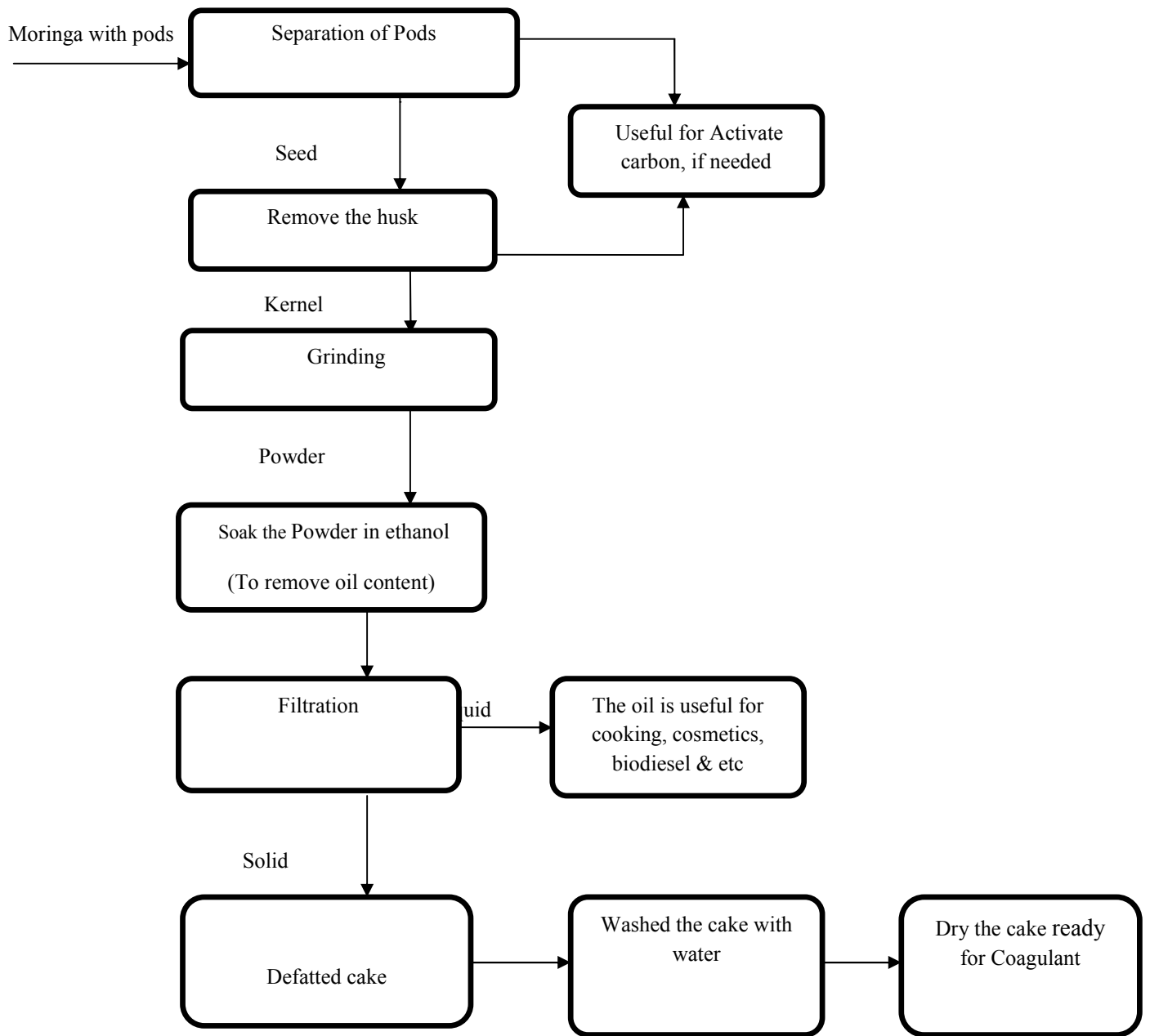


Figure 3.1: Sequential coagulant preparation flow chart of Moringa seed



Pods



Seeds



Seed kernels without husk

Figure 3.2: Moringa pods, seeds and kernel

After, the seeds kernel dried a domestic mill was used to ground to a fine powder of approximate size 600 μ m to achieve solubilization of active ingredients in the seed [28]. To extract oil, the next step was soaked the powder with 95% ethanol at 100g of the powder in 500ml of ethanol for 30 minutes at room temperature while mixed with the help of stirrer from time to time [49]. To obtain the defatted cake, it is required to filter the solution using filter paper No 41 [46]. The remaining solids (press cake) in the filter were then dissolved in water, stirred and filtrated in the same way. And then allow the cake to dry in oven at 40 °C for 24 hours [49]. This is helpful for removing ethanol that may prevail with the seed cake powder. Finally, the dried seed powder is stored under room temperature until it is used for coagulation experiment. Figure 3.3 was showed the powder of Moringa seed after its oil was extracted.



Figure 3.3: *Moringa stenopentala* seed powder

3.2.4. Coagulation Experiments

Jar test is the most widely used experimental method for evaluating and optimizing coagulation-flocculation processes based on standard methods [12]. Similarly this research has used the jar test apparatus consists of the batch test, accommodating a series of four beakers together with four-spindle steel paddles. Before operating the jar test the wastewater samples were mixed homogeneously and their initial pH was adjusted by adding either strong acid (HCl) or strong base (NaOH). Then the samples of 1000 ml were poured in to each beaker. After the different dose (2-8g/l) of the coagulant was added to the beakers and agitated at various mixing time with constant speed consisting of rapid mixing 100 rpm for 2 minutes and slow mixing 40 rpm for 15-60 minutes. The rapid mixing disperses the coagulant uniformly throughout the beakers whereas the slow mixing promotes the formation of floc by enhancing collisions of particles that led to larger flocs [37]. After the agitation being stopped, the suspension was allowed to settle for 30 minutes.

After settlement the supernatant were filtered using filter papers No. 41 [38]. Then the samples of supernatant taken from each beaker were tested for turbidity, color, TDS and TSS. The result of this test has represented the final concentration and it was compared with results of the initial concentration wastewater sample. In the experiment, the study was conducted by varying some operating parameters like coagulant dose (2-8g/l), pH (4-10), and mixing time (15-60 minutes) in order to study their effect in coagulation-flocculation process and which helped to obtain the optimum condition of parameters. This research focuses on finding the optimum condition for operating parameters depend on higher removal efficiency of color, turbidity TDS and TSS. The variables and their levels in coagulation process are depicted in Table 3.1. All coagulation experiments were run in triplicate to ensure the results were highly reproducible. A conventional jar test apparatus for treatment of textile wastewater in natural coagulant was depicted in Figure 3.4.

Table 3.1: Operating parameters and their values corresponding to their levels

No.	Parameters	Units	Range and levels			
			1	2	3	4
1	Coagulant dose	g	2	4	6	8
2	pH	-	4	6	8	10
3	Mixing time	Minute	15	30	45	60



Figure 3.4: A jar test apparatus used for treatment of textile wastewater in coagulation

3.2.5. Analysis Method

Standard procedures [51] were used to measure color, turbidity, TSS, and TDS at school of chemical and bio engineering laboratories AAiT.

3.2.5.1. Color Measurements

A HACH spectrophotometer (Model DR 2500) was used for the color measurements in Pt-Co unit. The following are the experimental procedures followed to determine the color of the wastewater after treatment: Wipe the blank (deionized water) and place it into the cell holder and touch zero, the display will show 0 Pt-Co and, then Wipe the prepared 10ml wastewater sample and place it into the cell holder and touch read, the result will appear in Pt-Co unit. Figure 3.5 show HACH spectrophotometer (model DR 2500) used for color measurement.



Figure 3.5: color measurement by using HACH model Spectrophotometer

3.2.5.2. Turbidity Measurements

The primary purpose of the coagulation/flocculation process is the removal of turbidity from the wastewater [10]. The cloudiness of waters is referred to as turbidity and has its origin from particles suspended in the water. Turbidity is a measure of the extent of light is either absorbed or scattered by suspended material in water [13].

Turbidity measurements were conducted by using a HACH turbidimeter (Model 2100N) when the sample was filled into a sample cell (test tube) and put into the cell holder for measurement. The turbidity of the samples was displayed on the LCD panel of the instrument in NTU. The turbidimeter was calibrated with StablCal stabilized formazin primary standards of 0.01, 20, 100, 200 and 1000 NTU before starting the analysis. HACH turbidimeter (model 2100N) used for turbidity measurement was show in Figure 3.6.



Figure 3.6: wastewater turbidity test by using HACH Turbidimeter

3.2.5.3. Total Solids Analysis

Total solids content defined as the residue remaining after a wastewater sample has been evaporated and dried at a temperature of 103°C to 105°C. Total solids are a measure of the suspended and dissolved solids in water. Matter suspended or dissolved in wastewater is considered as solids [6].

Figure 3.7 show wastewater samples in the oven for TS and TDS analysis.

Experimental procedure:-

1. Heat clean dish at a temperature of 103 – 105⁰C for 1 hour.
2. Store and cool dish in desiccators at room temperature until needed.
3. Weigh the dish immediately before use.
4. Add a measured volume of well mixed sample into the dish.
5. Evaporate the contents by placing the dish on an oven for 24 hrs.
6. Cool dish in desiccators to balance temperature and weigh it immediately.
7. The amounts of total solid in the sample can be computed using equation (3.1).

$$\frac{\text{Total solid, mg}}{1} = \frac{(A - B)}{\text{Sample volume, ml}} \times 1000 \dots \dots \dots (3.1)$$

where; A = weight of dried residue + dish, mg, and B= weight of dish, mg.



Figure 3.7: Wastewater samples in the oven for TS and TDS analysis.

3.2.5.4. Total Dissolved Solid analysis

Experimental Procedure:-

- i. Filter a measure portion of the mixed sample through a filter paper and collect the filtrate in a previously prepared and weight evaporating dish.
- ii. Repeat the step 4 to 6 outlined in total solid procedure
- iii. The amounts of total dissolved solid in the sample can be computed using equation (3.2).

$$\frac{\text{Total Disolved Solid, mg}}{l} = \frac{(A - B)}{\text{Sample volume, ml}} \times 1000 \dots \dots \dots (3.2)$$

where; A = weight of dried residue + dish, mg, and B= weight of dish, mg.

3.2.5.5. Total Suspended Solid Analysis

Total suspended solids consist of silt, clay, fine particles of organic and inorganic matter. Total suspended solids were determined by using the following formula [51].

$$\text{Total suspended solid} = \text{Total solid} - \text{Total dissolved solid}$$

3.2.5.6. pH Measurements

The pH of the samples measurements were carried out using pH meter. The pH meter was calibrated using buffer solutions of pH 4.0 and pH 7.0 before starting the analysis.

3.3. Experimental Design

To apply the jar test in line with the objective of the study, it is necessary to follow the appropriate experimental design. To this end, the following experimental phases were undertaken to determine the optimum dose, pH, and mixing time needed to achieve the best performance of the coagulant in coagulation process. Based on the design, the experiments run were carried out and results obtained for optimization of operational parameter were shown in Annex VII.

1) Determination of Optimum Coagulant Dose without pH Adjustment

First phase: The first run, in which the coagulant dose varies while the mixing time was kept constant to determine the optimum coagulant dose. Here the experimental factor was coagulant dose ranging between 2-8 g/l. The removal efficiency of coagulate dose were determined based on removed amount of color, turbidity, TDS, and TSS.

2) Determination of optimum mixing time

Second phase: Optimum mixing time was investigated by providing optimum dose determined from the first phase. In this case the experimental factor was mixing time which ranges from 15-60 minutes and evaluated based on the removal efficiency of color, turbidity, TDS, and TSS.

3) Determination of optimum pH

Third phase: Using the optimum dose and mixing time taken from the previous phases was used to determine the optimum pH. Here the only experimental factor was pH where ranges from 4-10 and evaluated based on the removal efficiency of color, turbidity, TDS, and TSS.

4) Determination of Optimum Dose at Optimum pH and Mixing Time

Fourth phase: The coagulant dose vary while the pH and mixing time maintained constant at the optimum value which were taken from second and third phase was used to determine the optimum dose. For all phases the optimum value was picking depending on the highest removal efficiency of color, turbidity, TSS and TDS.

3.4. Sources of Error

The procedure of the experiments was done consistently through the whole study to minimize the sources of error. There were many sources of error encountered during the experiments done in this study. The possible errors in the study might rise from the lack of calibration for the laboratory equipment used in measuring turbidity, COD, and color. The other source of error occurs due to the chemical used for calibration equipments are out of date (expired) for example turbidimeter to test the turbidity. On the other hand the time of settlement is 30 minute but the settlement time was quite frequently too long since the measuring equipments were used by others so the setting of the sample analyses makes it one source of error. In addition to these the results variation may be occurred in Moring seed oil extract methods. Besides, the main factors affecting the jar test results are assumed to be the wastewater samples storage time is important since organic matter can be degraded and change concentration pollutant of the sample. In generally, the results of this study might be lower due to the above state problem.

3.5. Data Analysis

The data collected from the above experiment are analysis using Microsoft Excel program. The results are presented using tables and graphs. The percentage removal efficiency of the parameters was calculated using following formula [3].

$$\text{Removal Efficiency(\%)} = \frac{(C_o - C_e)}{C_o} \times 100 \dots \dots \dots (3.3)$$

where, C_o = initial concentrations (before treatment) and

C_e = final concentrations (after treatment).

4. Results and Discussions

4.1. Raw Textile Effluent Characterization

The experiment undertaken to examine the preliminary character of the textile wastewater sample demonstrate the following result. Accordingly, the collected samples had a red and dark blue color and the measurement result was 2500 Pt-Co. The pH of the effluent was found pH 10. This indicated that the effluent from the textile industry is more alkaline in nature. The measurement as to total dissolved solids and total suspended solids were 5000 mg/l and 2500 mg/l, respectively. Similar result had also reported by N. Muralimohan and T. Palanisamy who have conducted study on textile effluents [28]. Besides, the experiment was confirmed that the particles of TDS are higher than TSS in the textile wastewater sample. Furthermore, the experimental result of wastewater sample show 7500 mg/l of total solids. Finally, COD and BOD values were 1200 mg/l and 265 mg/l respectively. In other words textile wastewater contain high amount of non-biodegradable, 3 to 4 times, than degradable organic matters. The preliminary characters of textile wastewater sample were summarized under Table 4.1.

Table 4.1: Characteristics of Ayka Addis textile wastewater before treatment

S/No.	Parameters	Unit	Value
1	Chemical Oxygen Demand	mg/l	1200
2	Biological Oxygen Demand	mg/l	265
3	Total Suspended Solids	mg/l	2500
4	Total Dissolved Solids	mg/l	5000
5	Turbidity	NTU	200
6	Color	Pt-Co	2500
7	Total Solid	mg/l	7500
8	pH	-	10

4.2. The Effects of Operating Parameters on Coagulation Experiment

The effects of coagulant dose, pH, and mixing time are important factors in order to investigate the performance of Moringa seed in coagulation process. The color, turbidity, TDS and TSS level in textile wastewater are considered as the most important parameter, which can be used as the indicator on the coagulant capacity of Moringa seed in these experiments along with other parameters like BOD and COD.

4.2.1. Effect of Coagulant Dose

Coagulant dose is one of the most important parameter that has been considered to determine the optimum condition for the performance of coagulants in coagulation and flocculation [52]. Essentially, insufficient dosage or overdosing would result in the poor performance in process. Therefore, it is significant to determine the optimum dose in order to minimize the dosing cost and sludge formation and also to obtain the optimum performance in treatment process.

The experiments to test the effect of coagulant dose ranging from 2 - 8g/l, while pH was 4 and mixing time of 45 minute consisting of rapid mixing 100 rpm for 2 minutes and slow mixing 40 rpm for 45 minutes and 30 minute settling time, shown the presence of significant removal difference of color, turbidity, TDS and TSS. Accordingly, there was continuous removal of color, turbidity, TDS and TSS with increases in coagulant doses from 2- 6g/l and the removal rate of color, turbidity, TDS and TSS was low when the amount of dose was below 6 g/l. because, the amount of coagulant is inadequately to destabilize the particles. On the other hand, the removal of color, turbidity, TDS and TSS is lower when the coagulant dose is greater than 6g/l. Because the aggregated particles redisperse & disturb particle settling due to excess amount of the coagulant added. Similarly, other literatures also state that when coagulant dose is beyond an optimum concentration it would confer positive charges on the particle surface (a positive zeta potential), and as a result re-dispersion of particles [52].

Thus, the experiment confirmed optimum dose is 6g/l for the Moringa seed as a coagulant. And at this concentration resuspension of solids is possible. At this optimum condition, there were 94 %, 90 %, 82.7 % and 86.8 % removal of color, turbidity, TDS & TSS respectively. Such highest percentage removal with increasing coagulant dose was also reported by other relevant and related works [8]. The result of the experiment is summarized both in the form of figure and table.

Table 4.2: Percentage removal of color, turbidity, TDS, & TSS at various coagulant doses

Coagulant dose (g/l)	Color concentration (Pt-co)	Color Removal (%)	Turbidity concentration (NTU)	Turbidity Removal (%)	TDS concentration (mg/l)	TDS Removal (%)	TSS concentration (mg/l)	TSS Removal (%)
Control	2500	-	200	-	5000	-	3500	-
2	900	64	124.8	37.6	3100	38	1200	52
4	670	73.2	73	63.5	2300	54	1050	58
6	150	94	20	90	863	82.7	330	86.8
8	460	81.6	43	78.5	1820	63.6	1800	60

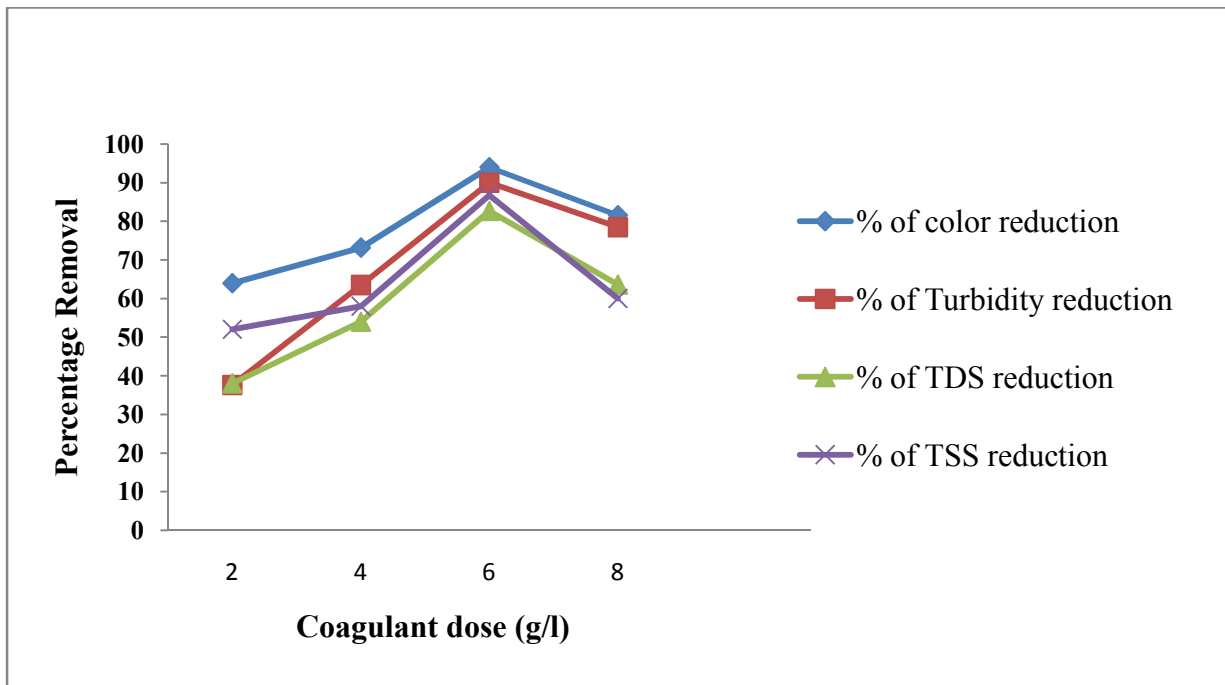


Figure 4.1: Effect of coagulant dose on % removal of color, turbidity, TDS & TSS

The results were presented Figure 4.1 which shows the effects of the coagulant dose on percentage reduction of color, turbidity, TDS and TSS by using Moringa seed as coagulant in coagulation.

In addition to discussing the optimum value and the percentage performance of treatment it is important to explain other results which were observed during the experiment. Accordingly, the initial red and dark blue colored textile wastewater sample was changed in to colorless after adding different doses of the coagulant. This confirms that Moringa seeds to have absorbent properties and effective for removal of color. Similar results are obtained from relevant and related works [12]. With regard to turbidity, initially it 200 NTU and after the coagulation experiment it becomes 20 NTU. This is good evidence on the effectiveness of Moringa seed for decreasing turbidity of textile wastewater. This result is also match with reports made by other researchers [49, 53] who proved that the potential of Moringa seed to remove 90-99% turbidity.

The experiment also confirmed the effectiveness of the Moringa seed which reduced the concentration of total dissolved solids from 5000 to 863 mg/l and total suspended solid from 2500 to 330 mg/l. This result including the range of removal also supported other study [28].

In general, the overall results of the coagulation experiment under this research proved that Moringa seed have an effective coagulant to remove the turbidity, TSS, TDS and color from textile wastewater. This result is described under figure 4.5.

It was also found important to examine the relationship between coagulant dose and Ph from the coagulation experiment. And consequently the result of the experiment observed that unlike chemical coagulants [12] Moringa dose did not have significant effect on the pH of treated effluent. Besides, pH scale of the treated effluent has fulfilled the permissive limit standard which set by EPA of Ethiopia. This assertion is also in agreement with the result obtained by others [46]. Thus, there is no need of pH adjustment for treated textile wastewater after coagulation using Moringa seed. This in turn avoids additional cost which is commonly incurred to adjust pH while using the chemical coagulant. The relationship between pH and coagulant doses are presented in Figure 4.2. Besides, textile wastewater emission limit standard of Ethiopia is attached under Annex I.



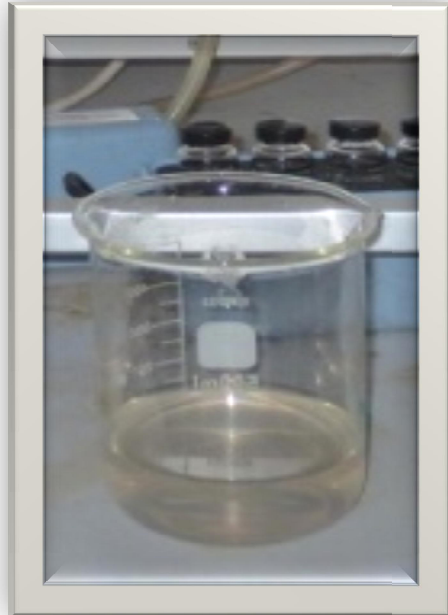
(a)



(b)



(c)



(d)

Figure 4.2: Raw wastewater samples [(a) & (c)], & corresponding results of coagulation [(b) & (d)]

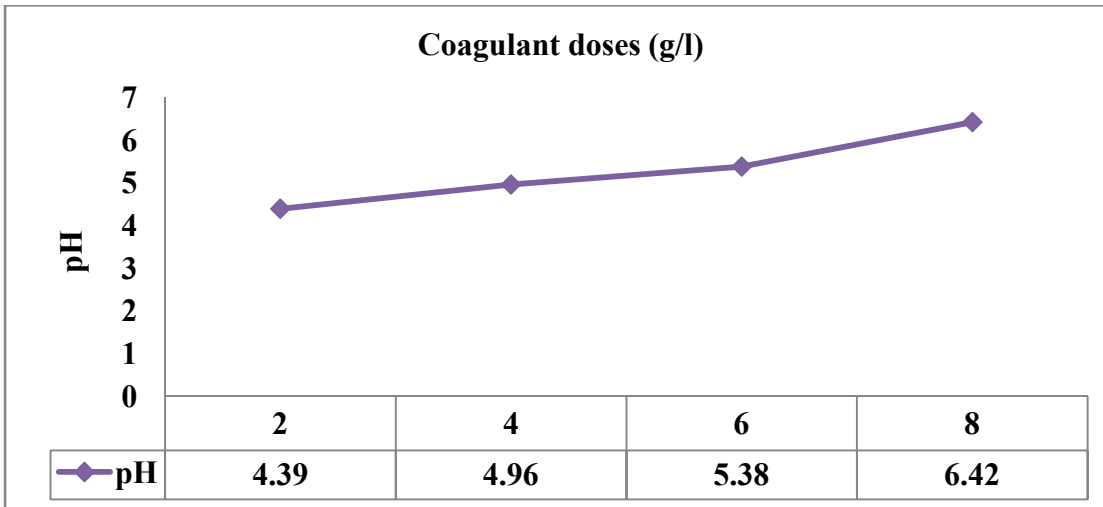


Figure 4.3: The response pH at different coagulant doses

4.2.2. Effect of pH

The effect of pH was analyzed at optimum dose of 6g/l and mixing time is 45 minute which consisting of rapid mixing 100 rpm for 2 minutes and slow mixing 40 rpm for 45 minutes and 30 minute setting time for a range of pH which varied from 4 to 10. And the coagulation experiment clearly affirmed that the percentage removal to be good for color, turbidity, TDS and TSS at acidic condition than basic, particularly at pH 4. Similar results are also obtained in another relevant study [38].

The optimum pH condition was at pH 4. At this point particles surface charge were reduced or neutralized as well as destabilized the suspension. The removal capacity has gradually declined as pH increase beyond 4. And as pH approaches basic condition it is proved to decrease the solubility of Moringa seed. Because, as pH increased beyond the optimum positive charges of Moringa seed surface decreased significantly. Consequently, the contribution charge neutralization roles of the seed to destabilize the particles become less. The properties of the seed, including its cationic behavior and molecular weight, is affirmed by others to be seem useful for such charge neutralization [12]. Since Moringa seed has positively charged it act as cationic behavior and result in protonation of amino groups of the solution. In other words, particles of textile suspension which are negatively charged bind to surface by means of ionic or hydrogen bonding with Moringa seed. Therefore, the particles destabilization by Moringa seed would result in charge neutralization. Moreover, at pH 4 the result showed that flocs produced by Moringa seed were rapid and caused large size of flocs formation

which was important for easiness of the settlement. The following table and figure summarized the effect of pH on Percentage removal of color, turbidity, TDS and TSS.

Table 4.3: Percentage removal of color, turbidity, TDS and TSS at different pH

pH	Color concentration (Pt-co)	Color Removal (%)	Turbidity concentration (NTU)	Turbidity Removal (%)	TDS concentration (mg/l)	TDS Removal (%)	TSS concentration (mg/l)	TSS Removal (%)
Control	2500	-	200	-	5000	-	2500	-
4	150	94	20	90	863	82.7	330	86.8
6	370	85	40	80	1270	74.6	612	75.5
8	1368	45	101	49.5	2150	57	974	61
10	1488	32.5	138	31	2560	48.8	1050	58

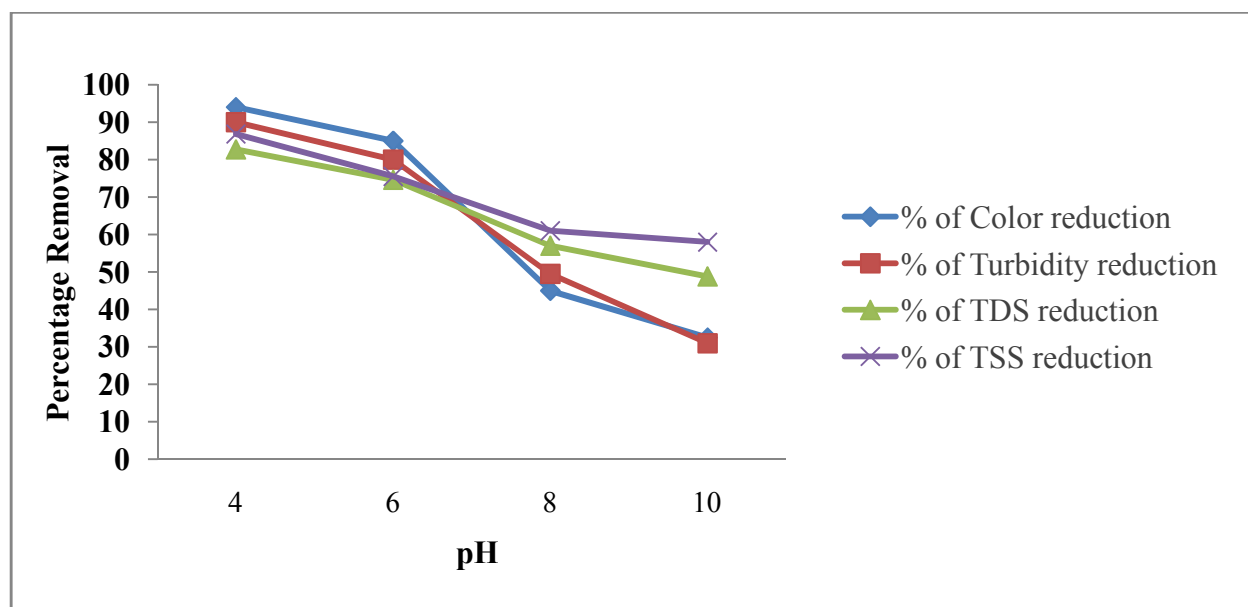


Figure 4.4: Effect of pH on color, turbidity, TDS, and TSS percentage removal

Finally, the result of the experiment demonstrated that at pH 4 there is highest percentage removal of color, turbidity, TDS and TSS; 94 %, 90 %, 82.7 % and 86.8 % respectively. Therefore, Moringa seed is effective for the treatment of textile wastewater in acid condition.

4.2.3. Effect of Mixing Time

The effect of mixing time on removal of color, turbidity, TDS and TSS was tested at 6g/l coagulant dose of and at pH 4, which consists of rapid mixing 100 rpm for 2 minute and slow mixing time from 15 to 60 minutes with 30 minutes of settling time.

The experimental result shows that there was a continuous removal of color, turbidity, TDS and TSS while increasing the mixing time from 15 to 45 minutes. Similar results are observed in other studies [8]. When the mixing time is short (<45 minutes), the collisions between the coagulants and colloids are not efficient to precipitate suspended solids in wastewater. On the other hand, if mixing time longer (>45 min) it would lead to an increase in flocs breakage & limit the size of the floc formed. As a result small size flocs which are not dense to settle down & finally cause the sample to be turbid again. In sum it is also found that the longer or shorter mixing time would result in the poor performance of the coagulant seed for binding and bridging. There were similar results in experiments made by other researchers [28]. The following table and figure summarized the experimental results of mixing time.

Table 4.4: Percentage removal of color, turbidity, TDS and TSS at different Mixing time

Mixing time (min)	Color concentration (Pt-co)	Color Removal (%)	Turbidity concentration (NTU)	Turbidity Removal (%)	TDS concentration (mg/l)	TDS Removal (%)	TSS concentration (mg/l)	TSS Removal (%)
Control	2500	-	200	-	5000	-	2500	-
15	982	60.7	85.	57.5	2300	54	1250	50
30	300	88	35.5	82	1950	61	900	64
45	150	94	20	90	863	82.7	330	86.8
60	630	74.8	66	67	1590	68	760	69.6

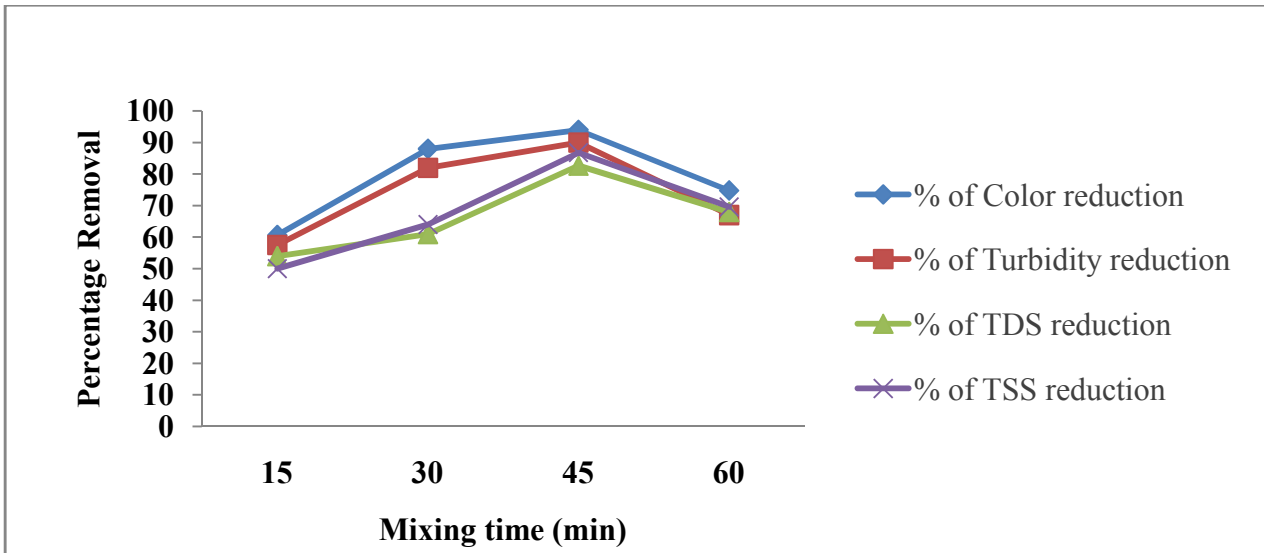


Figure 4.5: Effect of mixing time on color, turbidity, TDS, and TSS percentage removal

There is consistent percentage increment of removal was revealed with increasing flocculation time up to 45 minutes, and it decreases after 45. From this it can be inferred that the optimum mixing time using Moringa seed is rapid mixing at 100rpm for 2 minute and slow mixing at 40 rpm for 45 minute. Therefore, the optimum flocculation time is attained at 45 minute. This result also proximately matched with other study conducted to treat textile wastewater using natural coagulants [52]. There is highest removal percentage of color, turbidity, TDS and TSS 94 %, 90 %, 82.7 % and 86.8 % respectively, at the optimum mixing time.

Thus, the removal percentage of Moringa seed to remove color, turbidity, TDS, and TSS form textile wastewater was influence by mixing time. And it is a great importance to optimize the mixing time so as to increase removal performance of the coagulation.

4.2.4 The Optimum Operational Parameters

The optimum condition for removal of color, turbidity, TDS and TSS using Moringa seed as a coagulant in coagulation process were at 4.0, 6g/l, and 45 minute for pH, coagulant dose, and mixing time, respectively. Therefore, these optimum experimental conditions are needed to achieve the best performance of the Moringa seed. On the following paragraph an attempt is made to present the results of tests made on other parameters such as COD, BOD, and TS.

Similarly, at pH = 4, mixing time = 45 minutes, and coagulant dose of 6g/l there were maximum removal efficiencies of TSS, COD, and BOD showing 86.8%, 77.5% and 69 % respectively in which these results are above the limit value set by the EEPA. Since there is no clear standard which are set for color, turbidity, TDS and TS it made difficult to compare the result of the experiment. The results of these are summarized under Table 4.5. Annex I also present emission limits set by EEPA for textile industry.

Table 4.5 comparisons between treatment result with national discharge standard

No.	Parameters	Before treatment	After treatment	(%) Removal at optimum values	Ethiopian discharge standard
1	Color (Pt-Co)	2500	150	94	-
2	Turbidity (NTU)	200	20	90	-
3	TDS (mg/l)	5000	863	82.7	-
4	TSS (mg/l)	2500	330	86.8	30
5	TS (mg/l)	7500	1323	84	-
6	COD (mg/l)	1200	270	77.5	150 mg/l
7	BOD (mg/l)	265	81	69	50 mg/l
8	pH	4	4.39-6.42	-	6-9

5. Conclusion and Recommendations

5.1. Conclusions

This study concluded that the treatment of textile effluent before disposal is important to ensure the safety of our environment. To this end, there has been different textile wastewater treatment methods are studied and applied. Coagulation, which was applied in the present study, represents a powerful treatment method for toxic pollutants of textile wastewaters. Thus, different types experiments were undertaken to address each of the three specific objectives of the study.

The initial character of the textile wastewater sample showed pH -10, Color – 2500 Pt-Co, COD – 1200 mg/l, BOD – 265 mg/l, TS – 7500 mg/l, TDS – 5000 mg/l, TSS – 2500 mg/l, and Turbidity is 200 NTU.

The coagulation experiment proved coagulant dose, pH, and mixing time for being important operating parameters for the removal of color, turbidity, TDS and TSS from textile wastewater using Moringa seed as a coagulant. And their optimum conditions were at 6g/l of coagulant dose, pH 4, & 45 minutes of mixing time. At these optimal conditions the removal efficiency of color, turbidity, TDS, and TSS was 94 %, 90 %, 82.7%, and 86.8% respectively.

In general, it can be concluded that Moringa seed is an effective coagulant; that has significant potential to remove the level of color, turbidity, TDS & TSS from textile industry wastewater. Therefore, promotion and development of Moringa seed is offers many diverse advantages for developing countries like Ethiopia; cost effective and environmental eco-friendly.

5.2. Recommendations

Based on the above findings, the researcher recommends the wide use of Moringa seed as a coagulant for the treatment of textile industry wastewater which operate in Ethiopia. Because, for one thing it is possible to produce Moringa locally, and for another thing its application as coagulant should be encouraged since it is environmental friendly coagulant. To this end, the following points need attention:

- ❖ It is recommended to apply Moringa seed as coagulant since it has significant potential to remove color, turbidity, TDS, and TSS form textile industries wastewater of Ethiopia.

- ❖ Future research should be done using this technology in combination with other cost effective treatment methods for further treatment to improve the removal efficiency of the system.
- ❖ Additional studies should be conducted to identify other operational variables that may affect the effectiveness of Moringa seed as a coagulant in treating textile wastewater.
- ❖ Further research should be also undertaken on the use of Moringa seed at industrial scale for textile wastewater treatment.
- ❖ It is essential to create awareness to farmers to plant and engage on Moringa tree development all over the countries and creating market to local people to sell the seeds.
- ❖ Policy makers of Ethiopia should pay attention and exert pressure on wider application of Moringa seed as a coagulant in textile industries wastewater treatment system.

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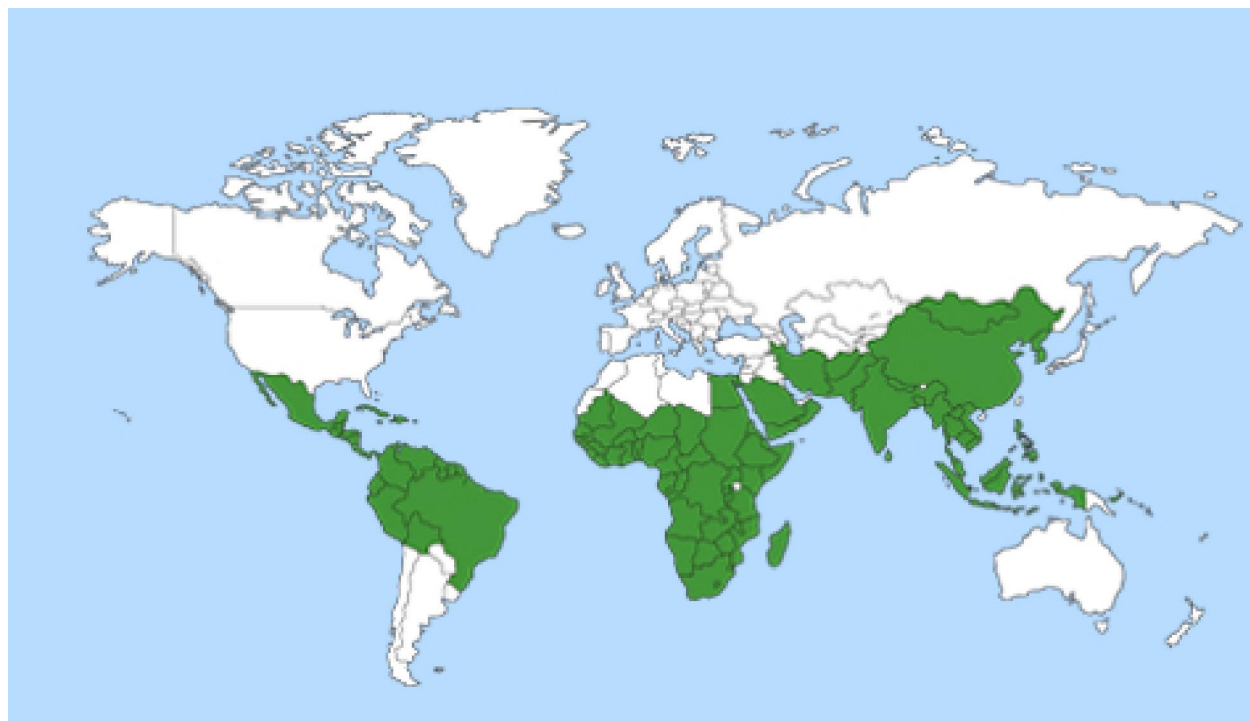
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Annexes

Annexes I: EEPA Emission Limits For Textile Manufacturing

Parameter	Limit values
Temperature	40 °C
pH	6 – 9
BOD ₅	90% removal or 50 mg/l, whichever is less
Total nitrogen (as N)	80% removal or 40 mg/l, whichever is less
COD (mg O ₂ /l)	80% removal or 150 mg/l, whichever is less
Total phosphorus (as P)	80% removal or 10 mg/l, whichever is less
Suspended solids	30
Total ammonia (as N)	20
Oils, fats & grease	20
Phenols	1
Mercury (as Hg)	0.001
Nickel (as Ni)	2
Cobalt (as Co)	1
Lead (as Pb)	0.5
Antimony (as Sb)	2
Tin (as Sn)	5
Chromium (as Cr VI)	0.1
Chromium (as total Cr)	1
Arsenic (as As)	0.25
Cadmium (as Cd)	1
Zinc (as Zn)	5
Copper (as Cu)	2
Mineral oils (Interceptors)	20
Benzene, toluene & xylene (combined)	1
Mineral oils (Biological Treatment)	5
Organochlorine pesticides (as Cl)	0.03
Mothproofing agents (as Cl)	0.003
Organ phosphorus pesticides (as P)	0.003
Adsorbable organic halogen compounds (AOX)	5
Sulphide (as S)	2

Annexes II: The Map of Geographical Distribution of Moringa Tree in World



Annexes III: Sample Collection at the inlet Point of Ayka Addis Textile Industry Wastewater Treatment Plant.



Annexes IV: Laboratorial Procedures for Water and Wastewater Analysis.

i) BOD test

Biochemical oxygen demand represents the amount of oxygen consumed by bacteria and other living microorganisms in the stabilization of organic matter of water/wastewater. Bacteria biodegrade the organic matter under aerobic conditions at a specified temperature. Thus this test determines the organic material present in wastewater.

The method consists of filling with sample, to overflowing, an airtight bottle of the specified size and incubating it at the specified temperature for 5 day. DO is measured initially and after incubation, and the BOD₅ is computed from the difference between initial and final DO. Because the initial DO is determined shortly after the dilution is made, all oxygen uptake occurring after this measurement is included in the BOD measurement.

Five days biological oxygen demand measurements were carried out according to procedures given in standard methods for the examination of water and wastewater, 5 – days BOD test. [49].

Apparatus used

- a) Incubation bottles: Use glass bottles having 60 ml or greater capacity (300 ml bottles having a ground glass stopper and a flared mouth are preferred). Clean bottles with a detergent, rinse thoroughly and drain before use.
- b) Air incubator or water bath, thermostatically controlled at $20 \pm 1^\circ\text{C}$. Exclude all light to prevent possibility of photosynthetic production of DO.

Required reagent

Prepare reagents in advance but discard if there is any sign of precipitation or biological growth in the stock bottles.

- a) Potassium hydroxide (KOH) to absorb carbon dioxide gas (CO₂)
- b) Nitrification inhibitor, 2 – chlorine – 6 - (trichloromethyl) pyridine. to avoid nitrification
- c) Dilution water: use demineralized, distilled, tap, or natural water for making sample dilutions
- d) Acid and alkali solutions, 1 N of NaOH and HCl, for neutralization of caustic or acidic Wastewater samples.

Experimental procedures

Preparation of the sample

- a) Select the volume for the wastewater sample
- b) The sample volume is related to the expected BOD₅ value. The BOD₅ incubator is designed to operate with the following BOD₅ ranges and sample volume allowing BOD₅ measurement.
 - i) BOD₅ range 0 - 400 mg/l use the sample without dilution
 - ii) BOD₅ range 0 – 2000 mg/l, the expected sample volume is 56 ml with 3 drop of nitrification inhibitor and 3 - 4 drop of potassium hydroxide (KOH) addition.
 - iii) BOD₅ range 0 – 4000 mg/l the expected sample volume is 21.2ml with 1 drop of nitrification inhibitor and 3 - 4 drop of potassium hydroxide (KOH) addition.
- c) Carry out the necessary pretreatment of the wastewater sample, setting pH 6.5 - 7.5, if higher or lower adjust by HCl and NaOH and mix well and allow the sample to settle and filtrate of the sample
- d) Measure the wastewater sample precisely using appropriate overflow and if necessary add nitrification inhibitor
- e) Insert magnetic stirring rod
- f) Place 3 - 4 drop of KOH solution into the seal gasket and insert gasket in the neck of the bottle, screw the BOD sensors to the sample bottle and then place the bottle in the bottle rack
- g) Finally, incubate the sample for 5 days at a temperature of 20⁰C.

Calculation

For each test bottle meeting the 2.0 mg/l minimum dissolved oxygen (DO) depletion and the 1.0 mg/l residual DO, calculate BOD₅ as follows:

$$\text{BOD}_5, \text{ mg/l} = \frac{D_1 - D_2}{P}$$

where, D₁ = DO of diluted sample immediately after preparation, mg/l.

D_2 = DO of diluted sample after 5 day incubation at 20 °C, mg/l.

P = decimal volumetric fraction of sample used.

ii) COD Analysis

The chemical oxygen demand is the amount of oxygen required to completely oxidize the organic matters and other chemical reducing substances of the wastewater. Chemical oxygen demand measurements were carried out according to procedures given in standard methods for the examination of water and wastewater, titrimetric method [49].

Apparatus used

Reflux apparatus, Burettes and pipettes

Required reagent

1. Standard potassium dichromate solution 0.25N.
2. Sulphuric acid reagent.
3. Standard ferrous ammonium sulphate.
4. Ferroin indicator solution.
5. Mercuric sulphate.
6. Sulphuric acid crystals.

Experimental procedures

1. Place 50.0ml of sample in a 500ml refluxing flask.
2. Add 1g mercuric sulphate and a few glass beads.
3. Add sulphuric acid to dissolve the mercuric sulphate and cool.
4. Add 25.0 ml 0.25 N potassium dichromate solution and mix well.
5. Attach the flask to the condenser and start the cooling water.
6. Add the remaining acid reagent (70ml) through the open of condenser and mix well.
7. Apply heat and reflux for 5 hours.
8. Cool and wash down the condenser with distilled water.
9. Dilute the mixture to about twice its volume and cool to room temperature.
10. Titrate the excess dichromate with standard ferrous ammonium sulphate using ferroin indicator. (2 to 3 drops).

11. The color change from blue green to reddish indicates the end point.
12. Reflux in the same manner a blank consisting of distilled water of equal volume as that of the sample.

Chemical oxygen demand concentration was calculated using the following formula:

$$\text{COD} \left(\frac{\text{mg}}{\text{l}} \right) = \frac{(V1 - V2)}{V} N \times 8000$$

where; V1= ml ferrous ammonium sulphate used for blank.

V2= ml ferrous ammonium sulphate used for sample.

N= normality of ferrous ammonium sulphate.

V= volume of sample used.

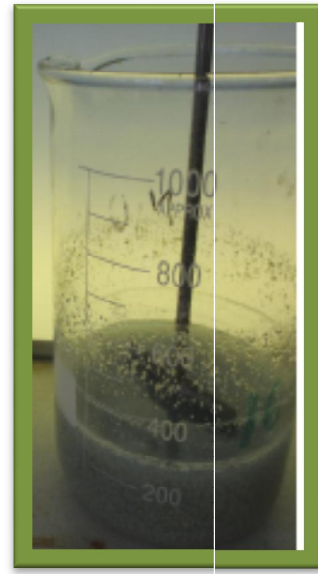
Annexes V: The Treatment Stage of The Coagulation Experiment Consisting of Images of the Raw water under a & b, floc formation under c, and stage of settlement under d & e.



(a)



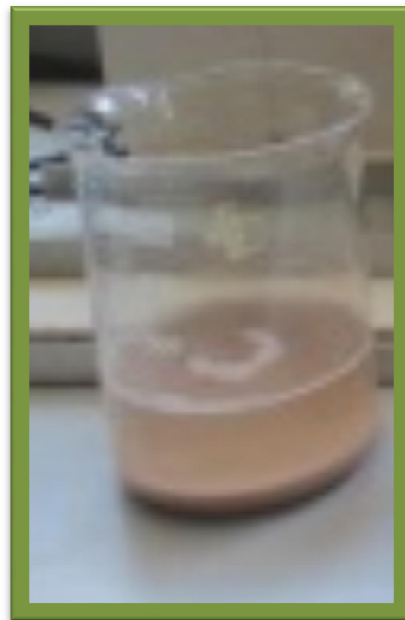
(b)



(c)



(d)



(e)

Annexes VI: Experimental Runs and Results to Optimize Operational Parameter

Determination of optimum coagulant dose without pH adjustment				
Run	1	2	3	
Variable	Coagulant dose	pH	Mixing time	
Level	2g	10	30 minute	
	After treatment			Average results
Parameters	Sample 1	Sample 2	Sample 3	
Color (pt-co)	990	1120	1220	1112
Turbidity (NTU)	63.2	76	58.8	66
TDS (mg/l)	4200	3700	4100	4000
TSS (mg/l)	2532	2751	2969	2750
Run	4	5	6	
Variable	Coagulant dose	pH	Mixing time	
Level	4g	10	30 minute	
	After treatment			Average results
Parameters	Sample 1	Sample 2	Sample 3	
Color (pt-co)	850	950	900	900
Turbidity (NTU)	49.2	50.8	40	46.6
TDS (mg/l)	2700	2800	2900	2800
TSS (mg/l)	1500	1400	1600	1500
Run	7	8	9	
Variable	Coagulant dose	pH	Mixing time	
Level	6g	10	30 minute	
	After treatment			Average results
Parameters	Sample 1	Sample 2	Sample 3	
Color (pt-co)	277	302	258	279
Turbidity (NTU)	40	46	28	38
TDS (mg/l)	1040	990	880	970
TSS (mg/l)	1500	1400	1600	1500

Run	10	11	12	
Variable	Coagulant dose	pH	Mixing time	
Level	8g	10	30 minute	
	After treatment			Average results
Parameters	Sample 1	Sample 2	Sample 3	
Color (pt-co)	517	503	480	500
Turbidity (NTU)	39.5	49	28.5	39
TDS (mg/l)	1200	1400	1300	1300
TSS (mg/l)	1100	1000	1200	1100
Run	13	14	15	
Variable	Coagulant dose	pH	Mixing time	
Level	6g	10	15 minute	
	After treatment			Average results
Parameters	Sample 1	Sample 2	Sample 3	
Color (pt-co)	1150	900	1100	1050
Turbidity (NTU)	108.2	141.8	110	120
TDS (mg/l)	2600	2500	2700	2600
TSS (mg/l)	2160	1910	2140	2070
Run	16	17	18	
Variable	Coagulant dose	pH	Mixing time	
Level	6g	10	30 minute	
	After treatment			Average results
Parameters	Sample 1	Sample 2	Sample 3	
Color (pt-co)	280	258	299	279
Turbidity (NTU)	37	42.4	34.6	38
TDS (mg/l)	1630	1600	1270	1500
TSS (mg/l)	770	700	630	700
Run	19	20	21	
Variable	Coagulant dose	pH	Mixing time	

Level	6g	10	45 minute	
	After treatment			Average results
Parameters	Sample 1	Sample 2	Sample 3	
Color (pt-co)	190	210	185	195
Turbidity (NTU)	31.3	27.5	28.2	29
TDS (mg/l)	910	930	890	910
TSS (mg/l)	470	520	510	500
Run	22	23	24	
Variable	Coagulant dose	pH	Mixing time	
Level	6g	10	60 minute	
	After treatment			Average results
Parameters	Sample 1	Sample 2	Sample 3	
Color (pt-co)	375	450	510	445
Turbidity (NTU)	75.5	66.4	68.1	70
TDS (mg/l)	980	1200	1120	1100
TSS (mg/l)	1472	1370	1358	1400
Determination of optimum pH				
Run	25	26	27	
Variable	Coagulant dose	pH	Mixing time	
Level	6g	4	45 minute	
	After treatment			Average results
Parameters	Sample 1	Sample 2	Sample 3	
Color (pt-co)	137	158	155	150
Turbidity (NTU)	19	16	25	20
TDS (mg/l)	863	863	863	863
TSS (mg/l)	330	338	322	330
Run	28	29	30	
Variable	Coagulant dose	pH	Mixing time	
Level	6g	6	45 minute	

	After treatment			Average results
Parameters	Sample 1	Sample 2	Sample 3	
Color (pt-co)	229	250		244
Turbidity (NTU)	38.3	41	34.7	38
TDS (mg/l)	1100	1200	1300	1200
TSS (mg/l)	640	660	800	700
Run	31	32	33	
Variable	Coagulant dose	pH	Mixing time	
Level	6g	8	45 minute	
	After treatment			Average results
Parameters	Sample 1	Sample 2	Sample 3	
Color (pt-co)	520	610	610	580
Turbidity (NTU)	45	53	52	50
TDS (mg/l)	2530	2620	2530	2560
TSS (mg/l)	980	1130	1190	1100
Run	34	35	36	
Variable	Coagulant dose	pH	Mixing time	
Level	6g	10	45 minute	
	After treatment			Average results
Parameters	Sample 1	Sample 2	Sample 3	
Color (pt-co)	193	205	187	195
Turbidity (NTU)	35.3	26.2	25.5	29
TDS (mg/l)	880	948	902	910
TSS (mg/l)	487	482	531	500
Determination of optimum dose at optimum pH and mixing time				
Run	37	38	39	
Variable	Coagulant dose	pH	Mixing time	
Level	2g	4	45 minute	
	After treatment			Average results
Parameters	Sample 1	Sample 2	Sample 3	

Color (pt-co)	850	750	1100	900
Turbidity (NTU)	110	138.8	124	124.8
TDS (mg/l)	3050	3200	3050	3100
TSS (mg/l)	1300	1160	1140	1200
Run	40	41	42	
Variable	Coagulant dose	pH	Mixing time	
Level	4g	4	45 minute	
	After treatment			Average results
Parameters	Sample 1	Sample 2	Sample 3	
Color (pt-co)	670	650	690	670
Turbidity (NTU)	72	75	72	73
TDS (mg/l)	2100	2400	2400	2300
TSS (mg/l)	1000	1100	1050	1050
Run	43	44	45	
Variable	Coagulant dose	pH	Mixing time	
Level	6g	4	45 minute	
	After treatment			Average results
Parameters	Sample 1	Sample 2	Sample 3	
Color (pt-co)	150	160	140	150
Turbidity (NTU)	20	22	18	20
TDS (mg/l)	893	833	863	863
TSS (mg/l)	310	360	320	330
Run	46	47	48	
Variable	Coagulant dose	pH	Mixing time	
Level	8g	4	45 minute	
	After treatment			Average results
Parameters	Sample 1	Sample 2	Sample 3	
Color (pt-co)	465	475	440	460
Turbidity (NTU)	44	44	41	43

TDS (mg/l)	1300	1200	1100	1200
TSS (mg/l)	750	750	900	800

Declaration

I, the undersigned, declare that this thesis entitled “**Textile Wastewater Treatment by Using Moringa seed as a Natural Coagulant**” is my original work, and has not been presented for a degree in any University, and that all sources of materials used for the thesis has been duly acknowledged.

Declared by:

Name: Fetiya Hamid

Signature: -----

June 2015