ADDIS ABABA UNIVERSITY
ADDIS ABABA INSTITUTE OF TECHNOLOGY
SCHOOL OF CHEMICAL & BIO-ENGINEERING

INVESTIGATION OF REMOVAL EFFICIENCY OF BLENDED CACTUS MUCILAGE AND ALUM COAGULANTS IN TEXTILE WASTEWATER TREATMENT

A thesis submitted to the School of Chemical and Bioengineering presented in partial Fulfillment of the requirements of the Degree of Masters of Science in Chemical Engineering (Environmental engineering stream)

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June, 2019

Addis Ababa, Ethiopia

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

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Acknowledgement

First and foremost, I am thankful for His love and kindness of the Almighty God in giving me health, strength, patience and protection throughout my entire life. I would like to show my sincere gratitude to my advisor Dr. Solomon kiros for his valuable guidance and suggestion. Without his instruction, impressive kindness and patience I could not have accomplished my study. I would also like to show appreciation to all my teachers who have helped me to develop the fundamental and critical academic competence. I shall extend my thanks to the laboratory workers of AAIT School of Chemical and Bio-Engineering for their support during my research works, APF(Addis pharmuctical industry) of Adigrat branch, Arbaminch Textile employers especially Mr. Binyam the manager of the company, and Almeda textile factory for allowing me to use the facilities for my experimental works. Last but not least, I would like to say thanks to my Loving Parents especially my father and brother for their efforts, prayers and motivation throughout my life.

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List of Abbreviations

AAiT	Addis Ababa institute of Technology
ANOVA	Analysis of variance
BOD	Biological Oxygen Demand
BBD	Box-Behneken design
CI	Confidence Interval
COD	Chemical Oxygen Demand
DO	Dissolved Oxygen
EEPA	Ethiopian Environmental Protection Authority
EPA	Environmental Protection Authority
FTIR	Fourier transforms infrared spectroscopy
Mg/L	Milligram per liter
NTU	Nephelometric Turbidity Unit
Pt-Co	Platinum Cobalt Unit
PH	potential of Hydrogen
RPM	Revolution per Minute
RSM	Response surface methodology
TDS	Total dissolved solids
TSS	Total Suspended solids
TS	Total solids
WTP	Water Treatment Plants

Abstract

Textile wastewater treatment is one the most difficult environmental issues because it contains high color, high suspended solids, chemical oxygen demand (COD), pH, temperature, turbidity and toxic chemicals. The direct discharge of this wastewater without previous or proper treatment into the water bodies, like lakes, rivers, etc. pollutes the water affecting directly and indirectly the water. This indicated urgent need to develop cost effective and efficient treatment method. In this connection, coagulation is one of the most effective and simple to operate treatment methods to remove colloidal and suspended solid 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 overall objective of this study was to investigate the removal efficiencies of blended inorganic and natural coagulants namely alum and cactus mucilage. The evaluation of removal efficiency of the blended coagulants was done for color, COD, TSS, and TDS before and after treatment. The raw textile wastewater was collected from Arbaminch textile Investment group. The treatment process was carried out using a jar test at different coagulant dose, pH and mixing time values for constant other parameters. The coagulant dose, pH and mixing time were the three factors considered in the treatment process. The dose of the coagulants were 0.5-3 g per 500ml textile waste water whereas pH was set at 3-11 and mixing time (flocculation time) 20-60 minutes. The pH was adjusted using hydrochloric acid and sodium hydroxide (1M). The maximum removal efficiency of the blended coagulant was obtained at coagulant dose of 1.75g, pH 7 and mixing time of 40minuties for Color, TSS and TDS removal but 3g, pH 3, 40minutes for COD removal and the removal efficiency was 74.3%, 73.9%, 62.3, and 74.5 % respectively and from the observed results the optimum conditions were at 2.194g of coagulant dose, pH 3.840 and 38.992 minutes of mixing time with rapid mixing 100 rpm for 2 minutes and slow mixing 40 rpm for 20-60 minute and 60 minutes of settling time. At these optimum conditions 74.3 % color reduction, 71.177% COD reduction, 73.094 % TSS and 62.930 % TDS reduction was obtained.

1. Introduction

1.1. Background

Textile industries are one of the most common and essential economic sectors in the world. On the other hand, high volume of water consumption and varying wastewater characteristics due to many inputs and products, such as dyes, biocides, carriers, detergents, etc. used in the process are the factors that have caused a continuous effort to find appropriate technologies to treat textile industry wastewater(Patel & Vashi, 2012).

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 (Development,Ministry of finance and economic 2010). However, there is a practice of direct discharge of the untreated or partially treated effluent into environment. This is not a good international practice, since textile effluents are highly colored and saline as well as contain non-biodegradable compounds. On the other hand, high volume of water consumption and varying wastewater characteristics are the factors that have caused a sustained effort to find appropriate technologies to treat textile industry wastewater (Ghebremichael, 2004). Therefore, the problem associated with textile wastewater discharge in Ethiopia need immediate solution.

Textile industries are one of the largest of water users and polluters (Nemerow, 1978). Major pollutants in textile wastewaters are high suspended solids, chemical oxygen demand, heat, heavy metals, color, acidity, and other soluble substances (Adel et al., 2005). The waste water generated during the processes is discharged into the water streams. So, the toxic pollutants which are present in that effluent gets mixed up with the water streams and pose threat to both environment and human beings mainly through food web. Therefore, need arises to focus our attention on treating the textile effluent before its disposal to the environment.

The removal of dyes, high suspended solids, chemical oxygen demand, heat, heavy metals, color, and acidity from textile effluent can be carried out through several chemical, physical and biological methods. One of the most popular processes in effluent treatment is coagulation. The uses of synthetic coagulants are not considered as suitable due to health and economic considerations. Aluminum sulfate (e.g. Al_2 $(SO_4)_3$ $.18H_2O$) and iron salts are mostly used as coagulant reagents. Recent study done by (Vashi, 2013) indicated a number of serious drawbacks linked to the use of aluminum salts such as Alzheimer's disease associated with high aluminum residuals in treated waste water, excessive sludge production during waste water treatment and considerable changes in water chemistry due to reactions with the OH^- and alkalinity of water.

In addition, the use of alum salts is inappropriate in some developing countries because of the high costs of imported chemicals and low availability of chemical coagulants (Arnoldsson, Bergman, Engineering, & Environment, 2008). Chemical coagulation with the help of above discussed coagulants may be the method of choice for removal of textile wastewater before being fed to the biological treatment, if necessary. However, it has also some drawbacks as the efficiency of the treatment strongly depends on pH. Moreover, the coagulation process is not always efficient enough because at different environmental conditions such as at extreme pH and at very low or very high temperature, it may produce very sensitive, fragile flocs, which result in poor sedimentation. These flocs may rupture under any type of physical forces. To improve the efficiency of coagulation process, number of high molecular weight compounds such as polymers from synthetic or natural origin may be recommended(Verma, Dash, & Bhunia, 2012). A number of studies have pointed out that the introduction of natural coagulants as a substitute for metal salts may ease the problems associated with chemical coagulants (Marey, 2019).

Natural macromolecular coagulants are promising and have attracted the attention of many researchers because of their abundant source, low price, non-toxicity, multi-functional and biodegradability (Hemapriya, et al.2015). A natural derived coagulant offers several advantages over conventional coagulants such as aluminum sulphate: for example the activity is maintained over a wide range of influent pH values means no pH correction required, natural alkalinity of the raw water is unchanged following coagulation means no addition of alkalinity required and sludge production is greatly reduced and is essentially organic in nature with no aluminum residuals means sludge volumes are reduced by a factor of up to 5 (Ndabigengesere A., 1998). Among the natural coagulants Cactus (*Opuntia.ficus-indica*) is one of the plants that acts as a coagulation in wastewater clarification due to the presence of a soluble, high molecular weight cationic protein which can reduce the ,COD, turbidity, color of untreated water (Ghebremichael, 2005). This paper concentrates on removal efficiency and assesses its effect on the treatment of textile waste water by combined the natural coagulant (Cactus mucilage) and synthetic coagulant (alum). Alum can be bought in liquid form with a concentration of 8.3%, or in dry form with a concentration of 17%.

Thus as a solution can be minimize to the problem that has been stated in the above by using the combined Cactus (Opuntia.ficus-indica) and alum can be used as an alternative to replace synthetic aluminum to reduce the turbidity, Color, pH, TSS, TDS, COD, heavy metals and heat of the water and make it more suitable and safe to be consumed by textile industry and the coagulation/flocculation technique followed by sedimentation is used worldwide in the treatment of residual waste waters. This treatment consists in the quick dispersion of the coagulating agent in the water to be treated, followed by intense agitation, which is commonly defined as quick mixing. Coagulants are added to effluents to destabilize the colloidal material and aggregate the small particles to form greater species, which are more easily removed as flocs (Metcalf, Eddy, 2003). In this paper, the potential removal efficiency of combined Cactus (*Opuntia.ficus-indica*) and aluminum sulphate for textile wastewater treatment was evaluated, its limits analyzed, and the optimum use and dosage assessment was evaluated.

1.2. Statement of the Problem

Environmental pollution is one of the greatest challenges human beings face. Water quality change due to industrial pollution is one of the major environmental concerns throughout developing countries including Ethiopia. Textile sector is one of highly water polluted industries because as several processes produce multi-component wastewater. Textile dyeing and finishing processes are among the most environmentally unfriendly industrial processes, because they produce colored wastewaters that are heavily polluted with dyes, dissolved solids, textile auxiliaries and chemicals (Roussy, J., M. Van Vooren, 2005). Wastewater discharge could be a common environmental issue for all textile industries. In downstream and near residential areas, these effluents cause pollution across the rivers and conjointly produce different environmental stresses.

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 and there are many chemical coagulants like aluminum salt, ferric salt and synthetic polymers that are widely used in water purification and treatment since ancient times. But these chemical coagulants release harmful substances to the environment which have detrimental effects on the human health. Besides, they are ineffective in low temperature water, are relatively costly, produce large sludge volumes, and significantly affect pH value of the treated water. They also cause diseases like "*Alzheimer*" in human beings (Beyene, et al., 2017). In order to reduce these problems, it is necessary to search other alternative coagulants. Nowadays, attentions are focused on searching renewable resources. This study is carried out to develop a new coagulant by blending alum and cactus mucilage coagulants. There is a potential to use locally available cactus mucilage as a coagulant combining with alum.

1.3. Objective

1.3.1. General objective

The general objective of this study is to investigate the performance of combined organic coagulants (cactus mucilage) with inorganic coagulant (alum) for textile waste water treatment.

1.3.2. Specific objective

The specific objectives of this thesis work are:

- To characterize the physiochemical properties of raw textile waste water including Color, TSS, TDS, pH, Turbidity, Temperature and COD
- To characterize the treated textile waste water (Color, TDS, TSS and COD)
- Determine the effect of operating parameters, namely coagulant dose, contact time, and pH on the performance of coagulation process of the textile waste water
- Determine the optimum value of these operating parameters
- To analysis the FTIR (Fourier Transform Infrared Spectroscopy) spectrum to identify functional groups of surface and localize the different absorption bands characteristic of cactus mucilage

1.4. Significance of the Study

This study was introducing the application of cactus as coagulant which is natural material and will initiate the country to reduce importing chemical-based coagulants via replacing them with renewable-based coagulant which has higher attribute than the current one which will in turn decrease value of importing and transportation. In this way the country will become self-sufficient. In addition it brings the county to green economy system. Generally once the research is done successfully and implemented, it is expected that to have the following significances.

- ✓ Reduce cost of chemical coagulant for the textile waste water treatment
- ✓ Farmers who cultivate the plant will be benefited financially
- ✓ Create employment for the local people

Therefore, as stated above this thesis work will possess significance on the society, environment and country.

2. Literature Review

2.1. Introduction

The degradation of surface and groundwater quality due to industrial and urban waste has been recognized for a long time. The rivers and stream are the common recipients of industrial effluent all over the world. The deterioration in water quality has an adverse effect on human beings as well as aquatic ecosystem directly or indirectly (Ugochukwu, 2004). The current practice of any industrial unit is to discharge wastewater into local environment without any treatment. The untreated or partially treated effluent on entering a water body either gets dissolved or lie suspended on river bed, thereby causing the pollution of water body. Industries are tempted to assume that they cannot avoid large volumes of wastewater produced during major industrial operations and therefore, they become lax in pollution prevention and textile industry is one example.

Textile and clothing industry is one of the largest and oldest industries present globally. The type of dyes and chemicals used in the textile industry are found to be differing depending on the fabrics manufactured. Textile industry is very water demanding, it uses fresh water for various activities in manufacturing process. Textile industries are one of the most polluting sources in terms of the volume and complexity of its waste water discharge. The waste water generated from textile industry is generally warm, Alkaline, Strong smelling and colored by chemicals used in dyeing processes.

In general, textile industries typically generate 200–350 L of wastewater per 1 kg of finished product (Gozálvez-Zafrilla et al., 2008) resulting in an average pollution of 100 kg chemical oxygen demand (COD) per ton of fabric (Jekel, 1998).

Wastewater generated from textile industry consists of various types of dyes, synthetic chemicals used in manufacturing processes, these dyes has high molecular weight and complex structure. Also it has high solubility in water makes a major impact of environment when it is discharged to water sources. More over a typical textile waste water is known to have various pH solutions, high temperature, high chemical oxygen demand(COD),high biochemical oxygen demand(BOD), high suspended solids (SS) and also characterized by high level color imparted by residual dyes. As industrialization develops, wastewater treatment has become a major issue in recent years and wastewater is not only the main maker of environmental imbalance but also furnish to the decrease of fresh water source of earth. Presently, there is a demand for new and environmental friendly wastewater treatment technology as the fresh water is polluting. Eventually, issues like the major process of textile industries, the sources of wastewater, characteristics of wastewater, and its environmental problems associated with industry wastewater, and the treatment options used in the treatment of textile wastewater was discussed. Lastly, special attention was given to coagulation process using combined (cactus and alum) as a coagulant for textile wastewater treatment and factor affecting the process.

2.2. The Production Process of Textile Industry and Sources of Wastewater

2.2.1. Production Process in Textile Industry (Textile Processing)

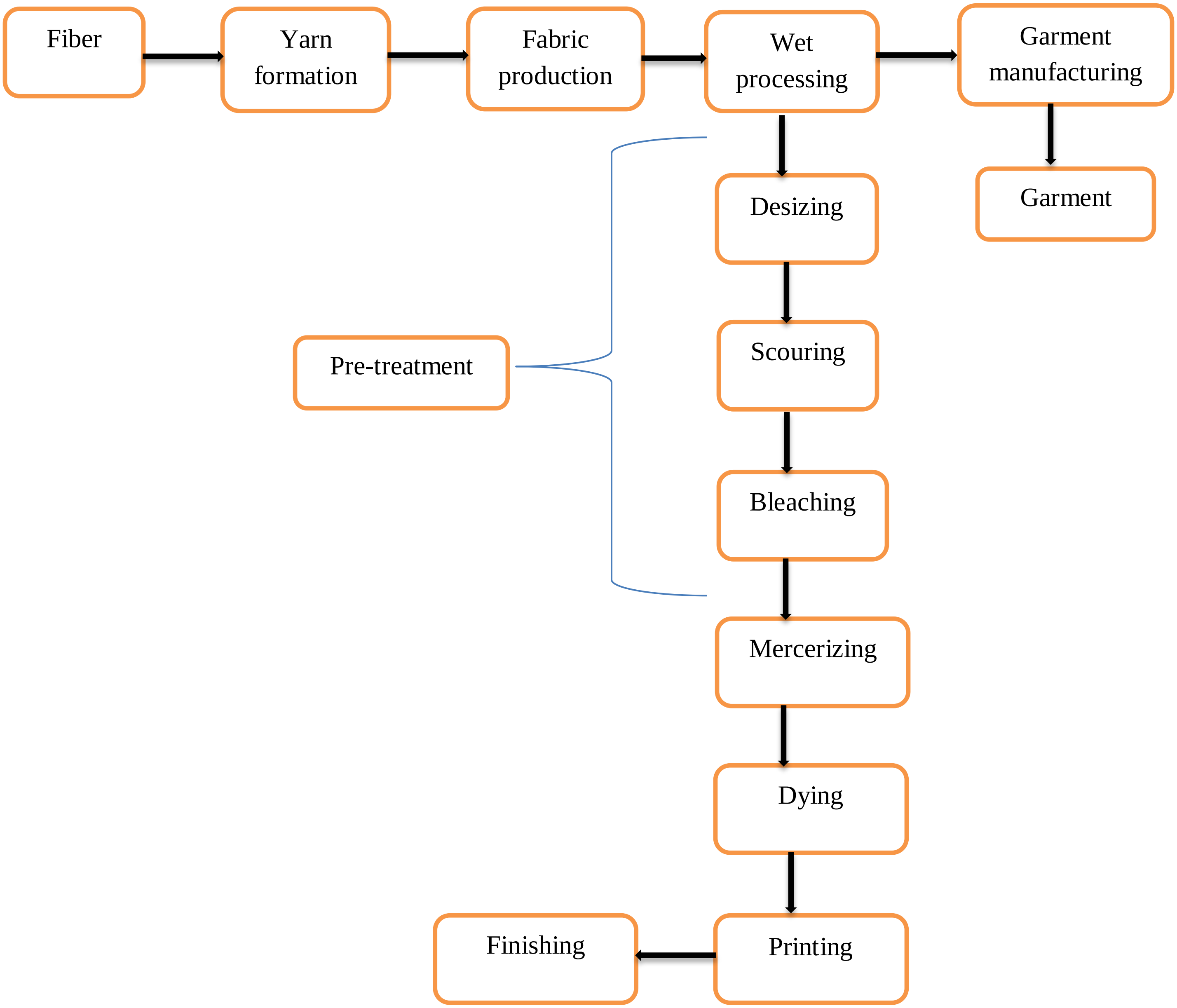

Figure 2. 1: Process flow diagram of diverse stages involved in textile processing

The textile industry comprises a varied cluster of facilities that fabricate and/or process textile-associated commodities (e.g., fibers, yarn, and fabric) for additional processing into clothing, household products, and industrial products. Textile industry facilities variously, collect and arrange fibers; alter fibers into yarn, thread, or webbing; transform the yarn into fabric or allied commodities; and dye and finish these commodities during the diverse steps involved in textile manufacturing (Ghosh, P and Gangopadhyay, 2000).

The following are the four stages by which the production of textile occurs.

1. Yarn formation,
2. Fabric formation
3. Fabric processing (wet processing), and
4. Textile fabrication.

2.2.1.1.Textile wet processing

Textile wet processing is a key stage in textile fabrication as it contributes the highest worth to the textiles by bestowing upon it its elegant and practical properties. Textile wet processing requires the use of huge quantities of water as an intermediate. This water is contaminated by unfixed dyes, chemicals, and auxiliaries, and it is released as effluent at the final stage (see the tables 2.1 and 2.2). Textiles effluents are difficult to treat because they contain a cocktail of chemicals (Saxena, et al., 2017).

Table 2. 1 : Wet textile processing and waste water generation

S.no	Process	Waste water contaminants
1	Desizing	BOD from water-soluble sizes; synthetic sizes ; lubricants
2	Scouring	NaOH disinfectant and insecticide residue; detergents; fats; oils; wax; lubricants
3	Bleaching	Hydrogen peroxide; sodium silicate; organic stabilizer
4	Mercerizing	High pH; NaOH
5	Dying	Heavy metals; salts; surfactants; BOD; Color
6	Printing	Suspended solids; urea; solvent; color; metals; BOD
7	Finishing	BOD; COD; Suspended solids; spent solvents

(Adopted and modified from (Babu BR, Parande SR, 2007)

2.2.1.2. Sizing and Desizing

Sizing is carried out for the purpose of removing the loose hairy fibers from the surface of the cloth, thereby giving it a smooth, even and clean looking face. In the sizing process, the yarn is coated for ease of use in weaving, knitting, and for tufting. Sizing is performed by applying chemicals such as polyvinyl alcohol, carboxymethyl cellulose, and polycyclic acids. It is estimated that about 750 kilograms (kg) of sizing material is present in the effluent discharged from an average mill that produces about 60,000 meters of fabric(Council, 1995). The presence of sizing elements in the yarn hampers processes such as dyeing, printing, and finishing. For example, the existence of starch can inhibit the dispersion of dye into the fibers.

Desizing is the process of removing sizing materials from the fabric, which is applied in order to increase the strength of the yarn. Fabric which has not been desized, is very stiff and causes difficulty in its treatment in subsequent processes. During the desizing process, starch is detached or altered into a water-soluble condition, either by hydrolysis or by oxidation (Batra, n.d, 2017). The desizing process contributes approximately 50% to the total amount of wastewater, which contains high levels of biological oxygen demand (BOD). The application of enzymes that can degrade starch into simple ethanol instead of complex anhydroglucose can resolve the problem. An oxidative arrangement such as hydrogen peroxide (H_2O_2) can also be utilized to complete the dissolute of starch to carbon dioxide (CO_2) and water (H_2O) (Babu BR, Parande SR, 2007)

Table 2. 2 : Ecological friendly unconventional chemicals for textile wet processing

s.no	Purpose	Convectional chemical	Alternative chemical
1	Sizing	Starch	Water-soluble polyvinyl alchol
2	Desizing	Hydrochloric acid	Amylase
3	Scouring of cotton	Sodium hydroxide	Pecrinase
4	Bleaching	Hypochloride	Hydrogen peroxide
5	Oxidation of vat and sulfur dyes	Potassium dichromate	Hydrogen peroxide
6	Thickener	Kerosene	Water-based polyacrylate copolymer
7	Hydrotropic agent	Urea	Dicyanamide(Partially)
8	Water repellent	C8 fluorocarbons	C6 fluorocarbons
9	Wetting agent	Alkyl phenol ethoxylates	Fatty alchol phenol ethoxylates
10	Neutralization agent	Acetic acid	Formic acid
11	Mercerization	Sodium hydroxide	Liquid ammonia
12	Reducing agent	Sodium sulifide	Glucose, acetyl acetone
13	Dyeing	The powder form of sulfur dyes	Per- reduced dyes

(Adopted and modified from (Arputharaj et al., 2016))

2.2.1.3.Scouring

Scouring is a chemical washing process carried out on cotton fabric to remove natural wax and non-fibrous impurities (e.g. the remains of seed fragments) from the fibers and any added soiling or dirt. The fabric is boiled in an alkali solution which forms soap with free fatty acids. Cotton wax and other noncellulosic integrant of cotton are separated by hot alkali, detergent, or soap solutions, such as glycerol, ethers, and scouring solvents in this process (Madhu, A., & Chakraborty, 2017). Scouring can be performed in batch or continuous processes, which contributes major organic loads to textile effluents that are accumulated from the use of sodium hydroxide (NaOH), disinfectants, detergents, insecticide residues, pectin, fats, oils, and wax, spin finishes, knitting lubricants, and spent solvents. The pH of these effluents is highly alkaline, ranging from 10 to 11 (S. K. and S. S, 2012).

2.2.1.4.Bleaching

Bleaching improves whiteness by removing natural coloration and the remaining trace impurities from the cotton. The natural color substance in yarn imparts a creamy appearance to the fabric. It is essential to decolorize the yarn through bleaching to acquire white yarn that will allow for the production of pale and bright shades (Madhu, A., & Chakraborty, 2017). Hypochlorite ranks as one of the oldest industrial bleaching agents. However, nowadays, absorbable organohalides are also used as bleaching agents. The production of very toxic chlorinated organic by-products in the bleaching process can be diminished by using adsorbable organically bound halogen (AOx). Peracetic acid (CH_3CO_3H) is also a safe alternative to hypochlorite. Higher brightness values with less fiber damage can be obtained by using CH_3CO_3H. In recent times, single-step preliminary processes for desizing, scouring, and bleaching have helped in minimizing the amount and toxicity of the effluent (Slokar, Y. M., & Le Marechal, 1998).

2.2.1.5.Mercerization

Mercerizing is a treatment for cotton fabric and thread that gives fabric or yarn a lustrous appearance and strength. The process is applied to cellulosic materials like cotton. During mercerizing, the fabric is treated with sodium hydroxide solution to cause swelling of the fibers. This results in improved strength and dye affinity. Fabric and fibers of cotton are mercerized in the gray form after bleaching in order to provide luster, increase strength, and enhance dye uptake. The process of mercerization involves treating cotton materials with a strong caustic soda solution (about 18–24%) for 1 to 3 minutes and subsequently washing-off the caustic solution. Longitudinal shrinkage of cotton may occur when it is treated with this solution. Hence, during this process, the fibers/fabrics are held under tension to avoid shrinkage. The fabric and fibers obtain the required characteristics of luster, improved strength, dye intake potential, and amplified absorbency. Membrane techniques may be used for recovery of NaOH present in the wash water. Use of zinc chloride ($ZnCl_2$) is another way to increase the fabric's weight, accelerate dye intake, and permit the easy recovery of NaOH. This practice does not need neutralization, and thus, it is environment-friendly (Karim, M et al., 2006)

2.2.1.6.Dyeing

Dyeing is the process of adding color to textile products like fibers and yarns. Dyeing is normally done in a special solution containing dyes and particular chemical. After dyeing, the dye molecules have un-dissociated chemical bond with fiber molecules. The temperature and time are the two key controlling factors in this process. During dyeing, chemical pigments (dyes) are applied to fibers or fabric to provide colors. In the textile industry, synthetic dyes, which typically originate from coal tar and intermediates of petroleum industry, are most often used. In synthetic dyes chromospheres atoms are accountable for the color and auxochromes, an electron withdrawing or donating substituent, are responsible for the exaggeration of the color. Numerous chemicals, such as organic processing assistants, formaldehyde, heavy metals, surfactants, salts, and sulfide may be added to dyes to enhance dye adsorption onto the fibers, and these chemicals are key contaminants in the wastewater from the dyeing process (Sarayu & Sandhya, 2012). The auxochrome and chromophore groups of dyes are also responsible for the contamination of effluents (Szymczyk, et al., 2007).

In the dyeing process, water is applied to carry dyes in steam form to warm up the treatment baths. The dyeing of cotton fiber requires huge quantities of water for wet operations. For example, 0.6 to 0.8 kg of sodium chloride (NaCl), 30 to 60 grams of dyestuff and 70 to 150 liters (L) of water are required for dyeing 1 kg of cotton with reactive dyes (Chakraborty et al., 2005). Once the dyeing procedure is complete, a variety of colored dye baths containing elevated amounts of salts and organic substances are released. In general, dye baths are highly contaminated. For example, wastewater formed by reactive dyeing comprises hydrolyzed reactive dyes not fixed on the textile materials accounting for 20 to 30% of the reactive dyes. These unused dyes account for the different colors of the effluents from the dyeing operations. Organic substances or dyeing auxiliaries cannot be recycled. Hence, their addition in effluents results in the high BOD and chemical oxygen demand (COD) of the effluents (Babu, 2007).

2.2.1.7.Printing

Printing is usually interpreted as “localized dyeing”; a division of dyeing. In printing, dyes are applied to selected sections of the fabric that constitute the design. Necessary reactions involved in printing are similar to dyeing. In dyeing, color is used in solution form, while in printing, color is applied as a thick paste of the dye. Urea is the most frequently used chemical reactive dye in printing and leads to a high pollution load (Babu et al., 2007). PVC and phthalates in plastisol printing pastes add another level of anxiety, as their usage is unsafe to the workers (Saxena et al., 2017). Recently, screen-free printing techniques that are more ecofriendly than is the case with these other techniques, such as electrostatic printing and ink jet printing, have evolved (Lukanova, V., 2005)

2.2.1.8.Finishing

Finishing is a series of processes to which all bleached, dyed, printed and certain grey fabrics are subjected before sending to the market. The object of textile finishing is to render the goods fit for their purpose or end-use and/or improves serviceability of the fabric. Fabric may receive considerable added value by applying one or more finishing processes. A range of finishing operations is required in the processing of both natural and artificial fiber textiles. Finishing is done to develop precise characteristics in the refined fabric and involves the use of a large number of finishing agents designed for cross-linking, softening, and waterproofing. Biocidal finishing agents are used to provide certain antimicrobial characteristics to the finished textile fabric and also to shield the end commodities from microbial degradation (United States Environmental Protection Agency, 1997). Principally, biocides are inorganic or organo-synthetic chemicals used to disinfect, sanitize, or sterilize substances to preserve materials or to protect them from microbiological degradation. Textile biocides are mainly linked with toxicity in dye house effluent. Similarly, dye carriers also contribute toxicity to textile effluents (Arslan-Alaton, 2007)

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2.2.2. 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(J., 1984). 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 (ISPCH, 1995)

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 (Delee, 1998). 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 (A.M., 1993).

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 (T., 2014).

2.3. 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. 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 (Ghaly AE et.al, 2004). Generally, the effluent from a textile industry is characterized by extreme fluctuations in many parameters such as COD, BOD, pH, strong color and salinity.

The composition of the wastewater will depend on the different organic-based compounds, chemicals and dyes used in the dry and wet-processing steps. Recalcitrant organic, colored, and toxicant, surfactant, chlorinated compounds and salts are the main pollutants in textile effluents (Venceslau M.C., 1994).

2.4. Environmental Impact of Textile Wastewater

The characteristics of textile effluents vary and depend on the type of textile manufactured and the chemicals used. The textile wastewater effluent contains high amounts of agents causing damage to the environment and human health including suspended and dissolved solids, biological oxygen demand (BOD), chemical oxygen demand (COD), chemicals, odor and color. Most of the BOD/COD ratios are found to be around 1:4, indicating the presence of non-biodegradable substances(Ghaly, Ananthashankar, Alhattab, & Ramakrishnan, 2014). Textile processing is extremely chemical-intensive and involves the use of numerous environmentally unfriendly, non-biodegradable chemicals. In addition, large quantities of unused chemicals are left after the final stage of processing along with the process water. The toxic, non-recyclable chemicals utilized in the various processes are challenging to eliminate from bilge waters, as they require tertiary and additional treatments to address their various properties; however, neglecting to treat these wastewaters leads to environmental degradation when they are released to the environment. Textile processing is extremely chemical-intensive and involves the use of numerous environmentally unfriendly, non-biodegradable chemicals.

As the majority of the textile wet processing units are situated in developing or underdeveloped nations in which processing is conducted by small facilities that lack the financial resources to purchase and operate costly machinery for appropriate handling of wastewaters, these effluents are instead being released into the surrounding environment without adequate treatment to make them safe. This has resulted in severe ecological harm in the vicinity of many of these facilities (Saxena et al., 2017).

A massive amount of fresh water is required, as it is used in multiple processes, as a medium, a washing-off agent, a solvent for the dissolution of chemicals, and in the production of vapor for heating the process baths. The precise quantity of water used is based on the nature of substance being processed, the types of dyes and finishing agents, and the kind of handling technology engaged. About 50 to 100 L water is obligatory for the handling of 1 kg of textile materials (Uqaili & Harijan, 2011). Water is released in wet processing along with the unfixed dyes, chemicals, and auxiliaries in the form of textile effluents. Textile effluents are combinations of both organic and inorganic chemicals differing in amounts and features. Metals are necessary for many dyes, as they are responsible for imparting color. Copper, zinc, chromium, lead, cobalt, nickel, and manganese are common heavy metals associated with different dyes. The release of these heavy metals into water bodies and soil can be dangerous to human health and the environment (Hameed & El-Khaiary, 2008; Pang & Abdullah, 2013). The possible pollutant and the nature of effluents released from each step of the wet process are listed in Table 2.3 below.

Table 2. 3 : Sources of water pollution at various stages of processing

Process	Possible Pollutants	Nature of Effluent
Desizing	Starch, glucose, PVA, resins, fats and waxes do not exert a high BOD.	Very small volume, high BOD (30-50% of total), PVA.
Kiering	Caustic soda, waxes, soda ash, sodium silicate and fragments of cloth.	Very small, strongly alkaline, dark color, high BOD values (30% of total)
Bleaching	Hypochlorite, chlorine, caustic soda, hydrogen peroxide, acids	Small volume, strongly alkaline, low BOD (5% of total)
Mercerizing	Caustic soda	Small volume, strongly alkaline, low BOD (Less than 1% of total)
Dyeing	Dye stuff, mordant and reducing agents like sulphides, acetic acids and soap	Large volume, strongly coloured, fairly high BOD (6% of total)
Printing	Dye, starch, gum oil, china clay, mordents, acids and metallic salts	Very small volume, oily appearances, fairly high BOD.
Finishing	Traces of starch, tallow, salts, special fishes, etc.	Very small volume, less alkaline, low BOD.

(Adopted and modified Ghaly et al., 2014)

The textile effluents contain trace metals like Cr, As, Cu and Zn, which are capable of harming the environment (Ghaly et al., 2014). Dyes in water give out a bad color and can cause diseases like haemorrhage, ulceration of skin, nausea, severe irritation of skin and dermatitis. 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 re-oxygenation process and hence hamper the growth of photoautotrophic organisms. 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(Ghaly et al., 2014).

Textile industry emits large quantities of pollutants in form of liquid discharge, solid wastes and air pollutants to the environment. The industry has been always regarded as a water-intensive sector; the main environmental concern is the amount of large amount of effluent discharged and the chemicals within that effluent. Energy consumption and air emissions are another two important issues as well (IPPC 2001). The industry requires as many as 2,000 different chemicals from dyes to transfer agents, which are used at various steps of the process. Additionally, large amounts of water are used to convey these chemical and wash them out. The output of these processes is expelled wastewaters full of chemical which sink into the environment (HSRC 2006). Textile mills discharge millions of gallons of effluent each year. The effluent contains natural impurities extracted from the fibers and a mixture of process chemicals such as inorganic salts, dyes and heavy metals. The effluent is usually high in both temperature and pH because it’s saturated with dyes, and many chemicals used during the process. In general, the effluent is highly-colored, high in BOD and COD, has a high conductivity and is alkaline in nature. This effluent represents a threat to the aquatic life if it is not properly treated before disposal (Lawrence 1996, HSRC 2006 and Badani et al. 2005).

Chemicals that evaporate into the air became air pollutants. Some of them may be breathed or absorbed through the skin. Others are carcinogenic, they may cause harm to children or may trigger allergic reactions in some people. These air pollutants can fall out to become surface water or groundwater pollutants and water pollutants can infiltrate into the ground or volatilize into the air (Woodard 2001, HSRC 2006, Lo et al., 2012).

Hazardous solid wastes from the industry and disposed in the ground can influence the quality of groundwater and surface waters by way of leachate entering the groundwater and traveling with it through the ground (Lo et al. 2012 and Woodard 2001). .

Waste treatment processes can also transfer substances from one of the three waste categories to one or both of the others. Waste treatment or disposal systems themselves can directly impact the quality of air, water or ground. The total spectrum of industrial wastes must be managed as substances resulting from a system of interrelated activities (Woodard 2001).

One of the most critical problems of developing countries is improper management of vast amount of wastes generated by various anthropogenic activities. More challenging is the unsafe disposal of these wastes into the ambient environment (Kanu et al. 2011). The key environmental issues associated with textile industry are water use, treatment, and disposal of aqueous effluent (Odjegba and Bamgbose, 2012). Water scarcity is the most important sustainability issue facing the textile industry. The environmental risk is a function of environmental exposure (concentration and duration) and polluting potential (hazard characteristics or toxicity). Hence, reducing the emissions into the various environmental pathways can reduce the environmental risk (Shaikh 2009).

Textile wastewaters generated from different stages of textile processing contain huge amounts of pollutants that are very harmful to the environment if released without proper treatment (Verma et al. 2012). The extent of environmental pollution due to dye bath water is very high (Selvakumar et al. 2010). Environmental pollution caused by the release of a wide range of dyes through industrial wastewater is a serious problem in the present day (Mahmood et al. 2011). There are large numbers of mechanical and chemical processes involved in the textile industry and each process has a different impact on the environment. The presence of sulfur, naphtha, vat dyes, nitrates, acetic acid, soaps, chromium compounds, heavy metals like copper, arsenic, lead, cadmium, mercury, nickel, and cobalt, and certain auxiliary chemicals all collectively makes the effluent highly toxic. The mill effluent is also often of a high temperature and pH, both of which are extremely damaging (Kant 2012). Also, the accumulation of color hinders sunlight penetration, disturbing the ecosystem of the receiving water (Georgiou et al. 2003; Merzouk et al. 2010).

2.5. 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. These dynamic structures affect the treat ability 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. 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 (Pala A, 2001).

2.5.1. 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. (Pala, A., 2001). Biological process consisting mainly conventional, and extended activated sludge system. 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. 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 (Arshad Husain et,al, 2013). 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 (Arshad Husain et,al, 2013). Biological treatment of dyeing wastewater is cheaper than other methods, but it is less efficient for color removal due to toxicity of the wastewater and the need for an aeration system (Sapci Z and Ustun B, 2003)

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.

2.5.2. 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. 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. 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. 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. 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(Verma et al., 2012).

2.5.3. 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. 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. Coagulation is an essential process in the treatment of industrial wastewater. Chemical oxidation uses strong oxidizing agents such as hydrogen peroxides, chlorine and others to force degradation of resistant organic pollutant. Chemical oxidation is the most commonly used method of color removal 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(Verma et al., 2012).

2.6. Coagulation Process

In industrial wastewater different compounds are present like suspended, colloidal and dissolved solids. Suspended solids have a diameter larger than 10^{-6} mm, colloidal solids between 10^{-9} mm and 10^{-6} mm and dissolved solids smaller than 10^{-9}mm. This material must be removed prior to discharge (Metcalf, Eddy, 2003). 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. Coagulation is the one of the most popular unit operations in water and wastewater treatment. This process is physic-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. 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. Coagulation, flocculation and clarification, followed by filtration, which are the key steps in wastewater treatment systems(Karoliny, Souza, & Almeida, 2018). 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.

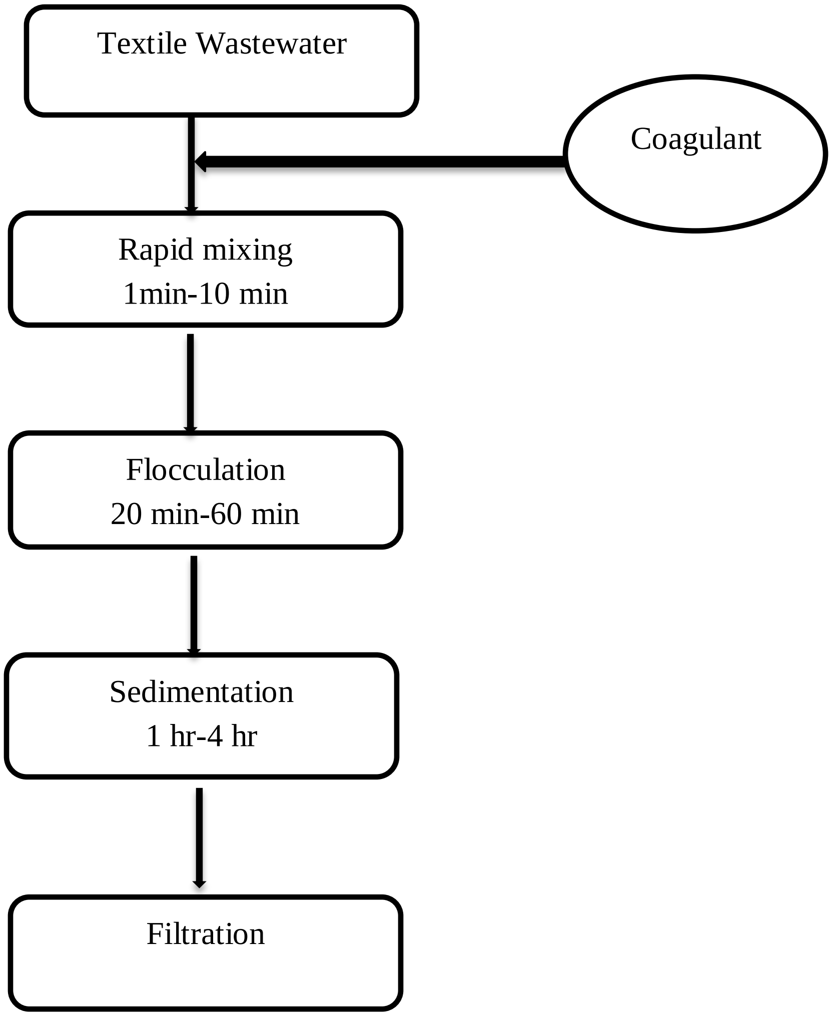

Figure 2. 2 : Steps of coagulation process

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 (Metcalf, Eddy, 2003). 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 and 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. 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 treatment methods that can be employed to reduce color effluent and also to reduce the total load of pollutants. The main advantage of these methods is cost effective, simple to operate and energy saving treatment alternative (Metcalf, Eddy, Inc., 2003). 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 (Metcalf, Eddy, Inc., 2003).For this reason, alternative natural materials have been considered for environmental applications. In this study the term was used coagulation, in line with the common trend, to represent the whole process starting from adding of coagulant in wastewater up to their settlement.

2.6.1. 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 (Mohd Ariffin, A. H.; Tan, P. L.; Zainura, 2008). 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. 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 inadequate to destabilize the particles. Above this range, the coagulant essentially serves as a chemical coating which re-stabilizes the particle (Patel H and Vashi R.T, 2013). 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 was 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(Verma et al., 2012). 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 (Safiullah S. et.al. 2012).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. 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. The next phase is flocculation time, it is characterized by slow mixing and allows destabilized particles to agglomerate together into larger particles (S. S. et al., 2012).

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 disagglomeration of particles. In general, optimization of coagulant dose, mixing time, and pH is significantly increases the effectiveness and efficiency of coagulation.

2.6.2. Types of Coagulants

A viable pre-treatment technology for reducing the pollution problems is the physic-chemical treatment using coagulants and this has been successfully applied to different wastewaters (M. R. Et.al., 2010).The coagulants used play a major role in the treatment of wastewater and disposal of sludge. 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) (Sunita Singh and Sonal Choubey., 2014). 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. There are two types of chemicals coagulants used in coagulation process. These include; primary coagulants and coagulant aids (Metcalf, Eddy, Inc., 2003). 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 (M. C. A. et al., 2002). 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 cactus etc. (O.P. Sahu and P.K. Chaudhari., 2013). 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. 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 assesil dementia and with Alzheimer's disease (G. et.al.,2013). 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 (Milind. R.et.al. 2010).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 (Milind. R.et.al. 2010).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. 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 synthetic polymers cannot be recycled, or cannot be used as fertilizers or animal feed (O.P. Sahu and P.K. Chaudhari., 2013). 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 (G. V. et al., 2008). Moringa seed, Nirmali, Okra, Beans, cactus, 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 (Mohd Ariffin Abu Hassan, et,al, 2008). 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 (G. V. et al., 2008).In coagulation cactus has been recognized to remove industrial pollutant namely from textile, dairy, brewery, and tannery (Gobinath.R et.al, 2013). Table 2.4 explains the gap of the study with comparing different coagulants with removal efficiency.

Table 2. 4 : Gap of the study with comparing different coagulant

Coagulant	Dose g/l	Removal efficiency (%)				Reference
		color	COD	TSS	TDS	
Alum	3	49.8	7.4	30.7	17.5	Radin M. et.al, 2014
	5	75	25	40.5	37.3	
	7	85.4	32	47	29.4	
Moringa	0.3	86	20.3	41.8	32	Khanittha Charoenlarp et.al ,2012
	0.7	72.6	31	33.4	36.7	
	1	75	28	29.3	38.4	
Ferric sulfate	1	53.8	5.7	10.4	18.7	Radin M. et.al, 2014
	4	62	22	19	22.6	
	7	59.3	24.4	28.5	27.4	
Blended Cactus mucilage and alum	1	Gap of this study				This study
	3.5					
	6					

Advantage of natural coagulant over chemical coagulants

a. Sustainability refers it is natural, abundant, renewable source, more eco-friendly and reduce chemical dependency.
b. Sludge refers in terms of reduce sludge volume, biodegradable, higher nutritional sludge value and disposing no treatment
c. Cost was explained in terms of lower sludge handling and treatment cost, local materials and local labor, no pH and alkalinity adjustment, lower procurement cost, lower coagulant dose and lower cost.
d. Nature of the coagulant explains in terms of non-corrosive, non-toxic, highly biodegradable and safe.

2.7. Application of Cactus as Coagulant

Cactus pear (*Opunta ficus-indica*) or prickly pear, a member of the Cactaceae family, is widely distributed in Mexico and in all American hemispheres and grows in many other parts of the world, such as Africa, Australia, Europe and the Mediterranean basin. There are several species belonging to the genus *Opuntia* (Family Cactaceae). This includes *O. Ficus-indica*, *O. dillenii*, *O. robusta*, *O. Tormentosa* and *O. tuna*. Of these, *O. ficus-indica* is the most common species that is grown in Ethiopia. Cactus pear (*O. ficus-indica*) is commonly known in Ethiopia as “Beles”. Cactus pears were largely ignored by the scientific community until the beginning of the 1980s when several studies and reports were published on their biological functions (Society, 2016). Previous studies by Zhang et al.2005 stated that the potential of cactus as a coagulant is due to its contents in terms of nutrition and medicinal components such as proteins, amylase, malic acid, resin, vitamins and cellulose.

The main factor behind the possibility of using cactus as material for waste water treatment is its biochemical composition without any toxic effects. It was reported by many studies that cactus is composed of low protein and lipid contents and polysaccharide is considered as the main ingredient. Interestingly, cactus species are known by the polysaccharic mucilage production. The presence of minerals, such as Ca^{2+} and K^{+}, is necessary for the gelatinous properties of mucilage. It was reported that calcium ion exists in the cactus mucilage as calcium-oxalate and this has strong effect in molecular conformation of the mucilage increasing its water holding capacity. Moreover, the rate of mucilage viscosity was observed to be enough to show industrially desired consistency to liquid formulations, especially in suspension preparations, suggesting the possible use of this product as natural thickeners. The high flocculation/coagulation capacity of cactus may related to its polysaccharide structure that composed of various carbohydrates, such as l-arainose, d-galactose, l-rhamnose, d-xylose and galacturonic acid. In this context, it was reported that galacturonic acid is significantly implicated as the main active coagulant agent, based on its polymeric structure. This polymeric structure provides a bridge for particles to adsorb. Moreover, the functional groups of cactus polysacharides included carboxyl (-COOH), hydroxyl (–OH) and amino or amine ($–NH_2$) groups, as well as hydrogen bonds. These functional groups considered as preferred groups for the coagulation-flocculation process (Rebah & Siddeeg, 2017).

The presence of polysaccharide as cactus main compound may play an important role in the thermal stability of the cactus based biofloculant. It is also very important to point out the presence of two factors making cactus an attractive candidate for the treatment of wastewaters from various origins. The first is the flocculation stability of cactus based flocculant at wide range of pH (3 - 12) and the second is the enhancement of the flocculation activity in the presence of divalent and trivalent cations. These proprieties (moisture, pH, the sugar composition, etc.) make the mucilage as a suitable media for microbial growth. This fact is in favors with the use of cactus as packed material for biofilter. Moreover, this composition make cactuses as a natural biosorbent material that well fit with the definition of sustainability, making them appropriate for regions of the world, where cactus is available. Generally For many accessibility criteria, cactus based materials are very attractive for wastewater treatment. Cactus plants are renewable, abundant, environmentally friendly, adaptable and biodegradable. The capability of various cactus preparations for pollutant removal was also observed. The cactus can be used as coagulant/flocculant, as biosorbent and as packed material for biofilter. Interestingly, the presence of cactus enzymatic system useful for the transformation of toxic textile dyes makes it as potential material for textile wastewater treatment (Rebah & Siddeeg, 2017). *Cactus Opuntia* contains carbohydrates such as d-galactose, d-xylose and galacturonic acid. Galacturonic acid is the active ingredient that affords coagulation capability of *cactus Opuntia*. It is highly possible that Galacturonic acid exist predominantly in polymeric form that provides a bridge for particles to adsorb on. The polygalacturonic acid structure evidently indicates that it is anionic due to partial deprotonating of carboxylic functional group in aqueous solution.((K. G. B. S, 2017)).

Cactus pear is adapted to many parts of Northern Ethiopia. Farmers maintain cactus backyards but most of the fruit harvest comes from the wildly growing cactus plantation (Tesfay, B. Mulugeta, G. and Tadesse, 2011). Although it is an important fruit and forage plant, there is no clear cut information about production and managements of cactus pear in Tigray. This means that the production would be high if appropriate management and production strategies are given to this crucial plant. Currently cactus pear is widely spread throughout the region and is believed to cover more than 379,338 hectares of land, i.e., 7.4% of the total land of the Tigray region (SAERT 1994; Nefzaoui et al., 2010) and becomes the integral part of the people’s economy. (Tesfay, B. Mulugeta, G. and Tadesse, 2011) also reported that in Tigray region alone, uncultivated cactus covered about 32,000 ha of land. A powdered cactus mucilage formed large flocs with impurities in the sample which facilitated setting and cleared supernatant was produced(Belayneh, n.d 2015.)

Figure 2. 3: Photographic representation Cactus pear (O. ficus-indica)

3. Materials and Methods

3.1. Materials

3.1.1. Equipment and Materials

The equipment and tools that were used throughout the experiments to achieve the objective of the study were: jar test apparatus, refrigerator, oven, measuring cylinder, beakers, magnetic stirrer, weight balance, pipette, crucible, domestic mill, sieve, filter paper, pH meter, HACH turbid meter (model 2100N), COD reactor, Spectrophotometer (HACH model), FTIR, glove, cactus, textile waste water, polyethylene bottle etc.

3.1.2. Chemicals and reagents

The following chemicals were also used during the experimental works such as:

- 1 M HCl & Na (OH) was used to adjust pH during the experiment
- Distilled water for washing the raw material
- Prepared reagents were used for turbidity and COD
- Detergent for cleaning
- Alum and cactus as raw material
- KBr for FTIR analysis
- Concentrate nitric acid was added to the effluent to prevent the growth of microbial bacteria and was sealed to prevent air oxidation.

All these chemicals were available in AAiT environmental engineering laboratory except alum was collected from Neway plc located in Arada sub city of Addis Ababa

3.2. Methods

3.2.1. Sample Collection and Study Area

In this study, the wastewater sample was collected from the textile factory of Arbaminch (figure 3.1), located around Sebeta town, 28 km southwest of Addis Ababa. The industry has a wastewater treatment plant that has the potential (capacity) to treat 3500 m^3 of wastewater / day. Wastewater samples were collected, by using grab sampling technique, from the inlet of the treatment plant. Clean Plastic containers (polyethylene terephthalate (PET) were used for sample collection after cleaned with detergent, rinsed with tap water, soaked in 10% of concentrate nitric acid and rinsed with deionized water. Finally the collected sample 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 analysis were done.

Figure 3. 1: Wastewater of the sampling area in the Arbaminch Textile Factory

3.2.2. Characterization of Textile Wastewater Composition

Before conducting experimental treatments, it is necessary to identify the composition and concentration of physical-chemical parameters. Thus, an initial experimental analysis was conducted and determined the physic-chemical characteristics of textile wastewater in terms of parameters, namely Color, COD, TSS, TDS, Temperature, Turbidity and pH. This experiment was conducted partially in School of chemical and Bio- engineering laboratory of AAiT based on standard procedures.

3.2.3. Coagulant Preparation

For the purpose of this study mature healthy and fresh leaves of the cactus plant (*Opuntia.ficus-indica*) was collected from Adigrat surrounding area, which located in the eastern zone of Tigray, around 900 km far from Addis Ababa, where it is abundantly available, low cost, safe for human health and biodegradable, offering various options (coagulant/flocculent, biosorbent, etc.) for the treatment of wastewater. The cactus samples were washed with distilled water thoroughly and the outer covering along with the thorns were removed and put in clean fresh polyethylene plastic bags. The collected cactus mucilage (inner part of cactus) was dried in oven at 65 ∘C temperature range, if it is above this range there is an opportunity to denature the composition of the mucilage and stored for 24 hours. Then dried cactus material were powdered using mortar and pestle and sieved to get particles of a size $\leq$ 300 µm and later used for coagulation purposes (Shilpaa et al. 2012 &Hedaoo, 2018) and stored at room temperature until final analysis.

The commercially available laboratory graded chemical coagulant Alum was collected from Neway plc located in Arada sub city of Addis Ababa. Figure 3.2 below shows the commercially available Alum and cactus mucilage prepared in powder form.

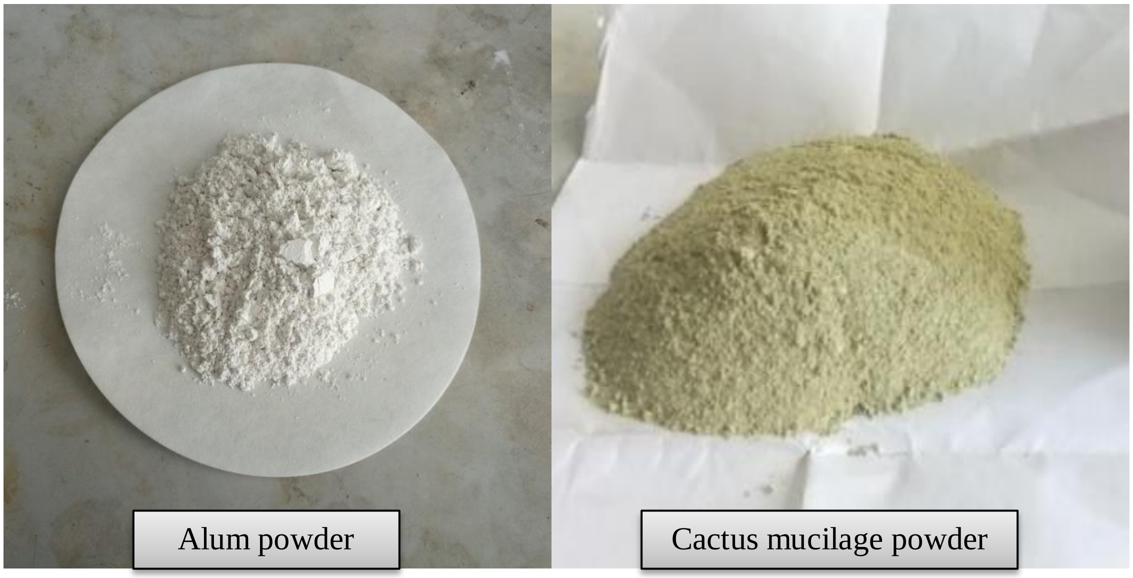

Figure 3. 2: Photographic representation of alum powder and cactus mucilage powder

3.2.4. FTIR analysis of Cactus mucilage

FTIR characterization was performed in order to identify the functional groups existing on the coagulant that might be involved in the textile waste water uptake process. FTIR analysis was conducted on cactus mucilage powder using Perkin Elmer spectrum FTIR spectrometer in average wave number from 4000 to 400cm^{-1} to observe the functional groups. The prepared cactus mucilage samples for the analyses were first milled using mortar and pestle to powdery conditions and the powder was mixed with KBr particles(1 mg of dried sample and 100 mg of dried KBr) to make it suitable to infrared analysis. The mixture was then pressed to a small thickness to make the sample a pellet that required for FTIR study. Then the coagulant was scanned in the spectrum range of 4000-400 cm^{-1}, a graph which comprises percentage of transmittance or reflectance along Y- axis and wavelength along X- axis. The type of the functional group present with in the coagulant can be predicted by the help of available table by studying the peak between particular wave numbers. Figure 3.3 shows the equipment of FTIR spectrophotometer to determine the functional group existing on the coagulant.

Figure 3. 3: Perkin Elmer Spectrum FTIR spectrophotometer (IR prestige 21 model)

3.2.5. Experimental Procedure: Jar test

- ✓ Took 500ml of the textile waste water
- ✓ Adjusted the textile waste water at required pH (3, 7 and 11 were used)
- ✓ Adjusted mixing time (flocculation time) of the textile waste water (20, 40 and 60 minutes)
- ✓ Weigh the Cactus mucilage and alum dose (0.5, 1.75 and 3g/0.5 L were used)
- ✓ Blended the natural coagulant cactus mucilage with alum. The blending ratio was 50% cactus mucilage *to* 50% alum
- ✓ Added the blended coagulant to the waste water
- ✓ Rapid mixing at 100 rpm for 2 minute was carried out followed by slow mixing (floc formation) at 40 rpm for 20, 40 and 60 minute
- ✓ Allowed the sample in the beakers for settling for 1 hour
- ✓ Filtered the supernatant after settling using whatman filter paper
- ✓ Analyzed the filtrate

Figure 3.4 shows as the batch experimental determination of the percentage removal efficiency of the color, COD, TSS and TDS.

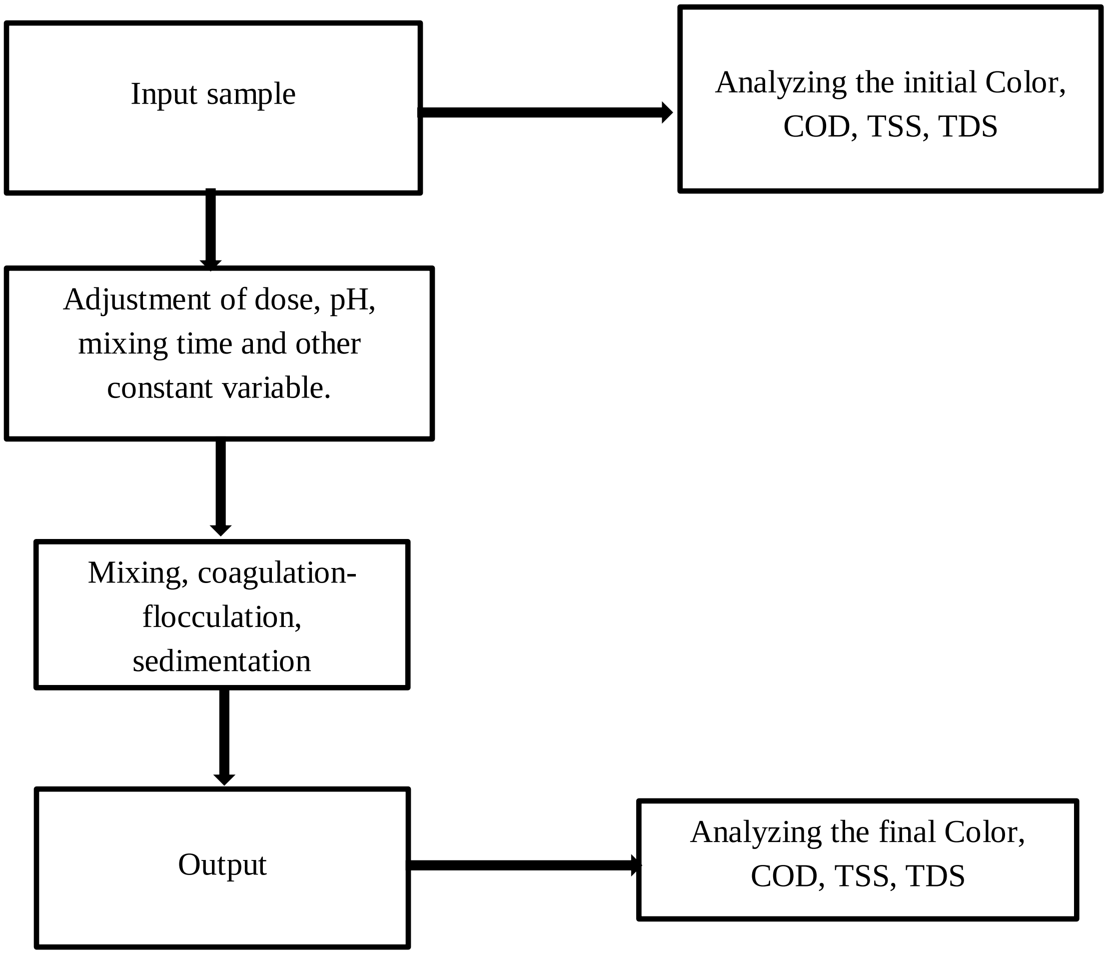

Figure 3. 4: The batch experiment for determination of Color, COD, TSS and TDS

3.2.6. Coagulation Experiments

This research was used the jar test apparatus consists of the batch test, accommodating a series of four beakers together with four spindle steel paddles as shown in figure 3.5. Before operating the jar test the wastewater samples were mixed homogenously and their initial pH were adjusted by adding either 1M strong acid (HCl) or strong base (NaOH). Then the sample of 500 ml was poured in to each beaker.

After the different dose (1-6g/l) of the blended(cactus and alum) coagulants were 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 20, 40 and 60 minutes (Beyene et al., 2017).

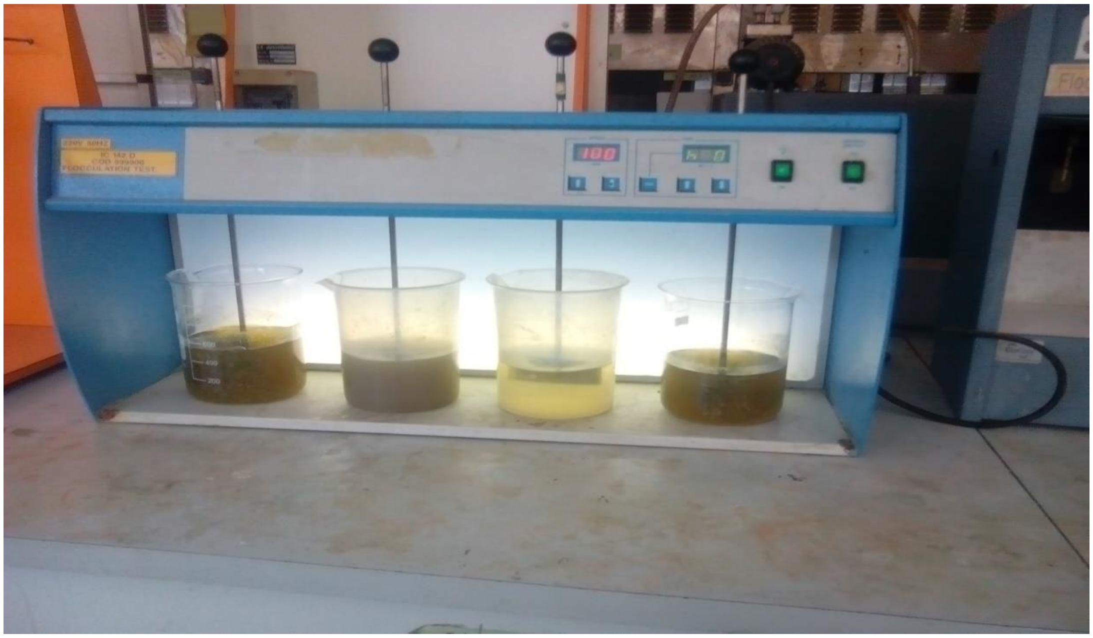

Figure 3. 5: Photographic representation of experimental set up of Jar test apparatus

The rapid mixing disperses the coagulant uniformly throughout the beakers whereas the slow mixing promotes the formation floc by enhancing collisions of particle that led to larger flocs. After the agitation being stopped, the suspension was allowed to settle for 60 minutes. After settlement the supernatant was filtered using filter papers. Then the samples of supernatant taken from each beaker were tested for color, TSS, TDS, and COD.

The results of the final concentrations were compared with results of the initial concentrations of wastewater sample. In this study the experimental set up were conducted by varying some operating parameters like coagulant dose, pH and mixing time with constant other parameters in order to study their effect in coagulation-flocculation process, which also helped as to obtain the optimum condition of parameters.

3.2.7. Analysis Method

3.2.7.1. pH Measurements

The pH measurement of the samples were carried out using pH meter (Model: JENWAY; 3505 pH meter). The wastewaters samples were investigated directly before the treatment and HCl & Na (OH) were used to adjust pH during the experiment by varying the value 3, 7 and 11 in order to see effect on the responses. But before starting the experiment pH meter was calibrated using three buffer solutions (pH = 3, pH = 7 and pH = 11). After calibrated the pH meter was inserted in to samples and read the value. The pH used in the experimental work was represented in appendix B of D.

3.2.7.2. Temperature Measurement

The temperature was measured using Digital Thermometer tp101 stainless, which is a reliable way to ensure that waste water has read the proper temperature. Before measuring the temperature, the thermometer was calibrated correctly. The thermometer used in the experiment work represented in appendix B of E.

3.2.7.3. Turbidity Measurements

The primary purpose of the coagulation/flocculation process is the removal of turbidity from the wastewater. 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. Turbidity measurements were conducted by using a HACH turbid meter (Model 2100N). First the turbid meter was calibrated with StablCal stabilized formazin primary standards of 0.01, 20, 100, 200 and 1000 NTU before starting the analysis. The sample was filled into a sample cell (test tube) and put into the cell holder for measurement and turbidity of the samples was displayed on the LCD panel of the instrument in NTU. HACH turbid meter (model 2100N) used for turbidity measurement was shown in appendix B of F.

3.2.7.4. Color Measurements

The following are the experimental procedures that were followed to determine the color of the wastewater before and after treatments. A HACH spectrophotometer (Model DR 2500) was used for the color measurements in Pt-Co unit. The experimental procedures followed to determine the color of the wastewater after treatment were: Wipe the blank (deionized water) and place it into the cell holder and touch zero, the display was show 0 Pt-Co and, then Wipe the prepared 10ml wastewater sample and place it into the cell holder and touch read, the result was appeared in Pt-Co unit. Appendix B of G or J shows HACH spectrophotometer (model DR 2500) used for color measurement.

3.2.7.5. Total Solids Analysis (TS)

Total solids content defined as the residue remaining after a wastewater sample has been evaporated and dried at a temperature of 103^{o}C to 105^{o}C. Total solids are a measure of the suspended and dissolved solids in water. Matter suspended or dissolved in wastewater is considered as solids. Appendix B of H shows wastewater samples in the oven for TS and TDS analysis.

Experimental procedure:-

i. Heat clean dish at a temperature of 103 – 105^{0}C for 1 hour.
ii. Store and cool dish in desiccators at room temperature until needed.
iii. Weigh the dish immediately before use.
iv. Add a measured volume of 20 ml well mixed sample into the dish.
v. Cool dish in desiccators to balance temperature and weigh it immediately.
vi. The amounts of total solid in the sample can be computed using equation given below

$$Total\ solid\left(\frac{mg}{l}\right) = \left(\frac{A\ -\ B}{sample\ volume, ml}\right) *\ 1000,$$

$$where\ A = \ weight\ of\ dried\ residue\ +\ dish, mg, and\ B = \ weight\ of\ dish, mg$$

3.2.7.6. Total Dissolved Solid analysis (TDS)

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 iv to v outlined in total solid procedure above(session 3.2.7.5)

iii. The amounts of total dissolved solid in the sample can be computed using equation below

$$Total\ dissolved\ solid, mg/l\ =\ ((A\ -\ B)/(sample\ volume, ml) * 1000,$$

$$where\ A = \ weight\ of\ dried\ residue\ +\ dish, mg, and\ B =\ weight\ of\ dish, mg$$

3.2.7.7. Total Suspended Solid Analysis (TSS)

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

$$Total\ suspended\ solid\ (mg/l)\ =\ Total\ solid\ (mg/l) - Total\ dissolved\ solid\ (mg/l)$$

3.2.7.8. Chemical oxygen demand Analysis (COD)

A DR2800 spectrophotometer was used to measure the COD value. About 0.2 mL of the water sample prior to and after treatment was pipetted into COD reagent vials, then inverting each vial up and down a couple of times, the reagent was mixed with the sample and inserted into a COD reactor. A blank were prepared based on our COD range by mixing equal amount of distilled water with the respective reagent. In the meantime the HANNA reactor was warmed up until it reaches up to 150^{o}C with strong oxidizing agent, potassium dichromate solution, which oxidizes organic matter chemically. Then the sample and the blank was fed in to the reactor and stayed there for two hours at that temperature. After two hours the reactor was off and waiting for some time, until the system cools down to reasonable temperature, the sample and the blank was taken out and put in to a rack until it cools down to room temperature. Finally a bench photo meter was calibrated using the blank and after that the sample was inserted in the photo meter for reading. The HANNA COD reactor or digester was shown in Appendix B of I.

3.2.8. Design experiments and optimization of process variables

So as to determine the effect of main parameter on the removal efficiency as well as to optimize all the affecting parameters collectively by statistical experimental design, Response surface methodology was used. Box–Behnken statistical experiment design (BBD) of the RSM, consisting of a three-factor and three-level pattern was used to conduct the experiments. The result of the experimental design were studied and interpreted by design.Expert.11.1.0.1 statistical software to estimate the response of the dependent variable.

i. Selection of response variable

% color removal, % COD removal, % TSS removal, and % TDS removal

ii. Choice of factors, levels and range

Factors: The potential design factors that have prime effect on the coagulation of textile waste water are:-

a. Coagulant dose

b. pH

c. Mixing time (flocculation time)

The variables and their levels in coagulation process were depicted in Table 3.1.

Table 3. 1 : The variables and their levels in coagulation process

No	Parameters	Units	Range and level		
1	Factors	-	-1	0	+1
	Coagulant dose	g/0.5L	0.5	1.75	3
	Ph	-	3	7	11
	Mixing time	Min	20	40	60

The collected data from the treatment of textile wastewater at different dose, pH and contact time value were analyzed using the Design-expert® version 11 portable software by the Response surface (Box-Behnken) design with factors of coagulant dose, pH and contact time with three levels of each factor was selected and replication of five times at the center point to see each treatment effect on the response (Color, TDS, TSS and COD) was recorded and analysis of variance (ANOVA) at confidence interval of 95% was used for statistical significance of the experimental data.

Table 3. 2 : Three- Factor, Three level Box-Behenken statistical experimental design

Std	Run	Factor 1	Factor 2	Factor 3	Response 1	Response 2	Response 3	Response 4
		A:Dose	B:pH	C:Mixing time	Color removal	COD removal	TSS removal	TDS removal
		G	NG	Min	%	%	%	%
14	1	1.75	7	40				
3	2	0.5	11	40				
13	3	1.75	7	40				
17	4	1.75	7	40				
16	5	1.75	7	40				
11	6	1.75	3	60				
15	7	1.75	7	40				
9	8	1.75	3	20				
2	9	3	3	40				
1	10	0.5	3	40				
5	11	0.5	7	20				
4	12	3	11	40				
7	13	0.5	7	60				
10	14	1.75	11	20				
8	15	3	7	60				
6	16	3	7	20				
12	17	1.75	11	60				

The coagulant dose, pH and mixing time were based on most literature values. Design comprised of 17 runs in random order; all points in coded factor levels. The center point (0, 0, 0) was replicated five times. The combination of the three factors (coagulant dose, PH & Mixing time) studied in the response surface experiment and optimization was based on the +1 and -1 variable levels of the experimental design.

The optimization of these factors may significantly increase the process efficiency. In conventional multifactor experiments, optimization is usually carried out by varying a single factor while keeping all other factors fixed at a specific set of conditions. It is not only time-consuming, but also usually incapable of reaching the true optimum due to ignoring the interactions among variables. Response surface methodology (RSM) has been proposed to determine the influences of individual factors and their interactions. RSM is a statistical technique for designing experiments, building models, evaluating the effects of several factors, and searching optimum conditions for desirable responses. With RSM, the interactions of possible influencing factors on treatment efficiency can be evaluated with a limited number of planned experiments (Imen, Lamia, Asma, Neacute ji, & Radhouane, 2013).

4. Result and Discussion

4.1. FTIR analysis of Cactus mucilage

FTIR characterization was performed in order to investigate the functional group of the coagulant that might be involved in the textile waste water coagulation process. The strong stretch at around 3,390 cm^{-1} was due to polymeric OH stretching vibration of water and stretching vibration of amine (figure 4.1). Peaks at around 2920 cm^{-1}, was due to the presence of C=C–H group, which indicates aromatic ring, and carboxylic acid as well as OH in weak broad appearance. Sharp peak at 1735 cm^{-1} confirmed the presence of C=O Stretch, C-C=C symmetric stretch, amine group, benzene group and carboxylic acid salt. Several peaks were formed from 750 to 500 cm^{-1}, which further confirms the presence of aromatic group. From the Peaks of FTIR spectrum, it could be evidently investigated that the major involvements of functional groups like –NH, –OH, C-O, C-H, –CH, aromatic and –COOH present on the cactus mucilage. Generally, the cactus represent the characteristic frequencies of the functional groups associated with mucilage, such as, carboxylic acid, carboxylate, ether, amine and alcohol groups. This results agree with the study done by Madera-santana et al., 2018 and other literatures that corroborating the presence of carbohydrates in the mucilage. The principal IR absorptions for certain functional groups used as reference representation in Appendix C.

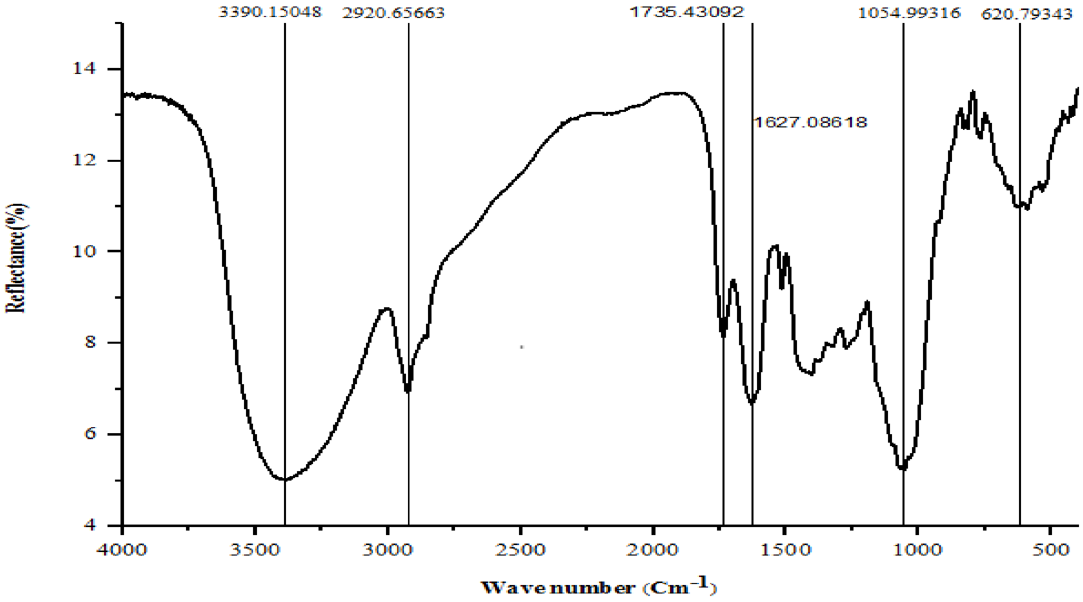

Figure 4. 1: FTIR spectrum of Cactus opuntia (ficus-indica) mucilage

4.2. Raw Textile Effluent Characterization

The experimental analysis undertaken to examine the initial character of the textile wastewater samples were demonstrated in table 4.1. Accordingly, the collected samples had a red and dark blue color as shown in appendix B of A and the measurement average result was 2300 Pt-Co and indicated as highly colored. The pH of the effluent was found average value 10.2. 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 average result of 1130 mg/l and 545 mg/l, respectively and this tell as contains high suspended solids. 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 average value of 1675 mg/l of total solids. Finally, COD average values were 1170 mg/l and almost similar result had also reported by N. Muralimohan and T. Palanisamy who have conducted study on textile effluents and the result shows it contains high chemical oxygen demand. The preliminary characters of average untreated textile wastewater sample were summarized under Table 4.1.

Table 4. 1 : Characteristics of Arbaminch untreated (raw) textile wastewater

Parameters	Value ± SD
PH	10.2±0.2
Temperature(^{o}c)	38 ± 1
Turbidity(NTU)	160 ± 5.1
Color(Pt-Co)	2300 ± 200
Chemical oxygen demand(COD)(mg /l)	1170 ± 30
Total solids(TS)(mg/l)	1675 ± 25
Total suspended solids (TSS)(mg/l)	545 ± 27
Total dissolved solids(TDS)(mg/l)	1130 ± 23

4.3. Statistical Analysis of the Experimental Results

The process consists of three main factors: coagulant dose, pH and mixing time with different responses. After following the above series of procedure, the experimental outcomes of those particular results were measured for Color, COD, TSS, and TDS. The result obtained from the experiment was indicated in table 4.2

Table 4. 2 : Result obtained from the experiment

Std	Run	Factor 1	Factor 2	Factor 3	Response 1	Response 2	Response 3	Response 4
		A:Dose G	B:pH	C:Mixing time Min	Color removal %	COD removal %	TSS removal %	TDS removal %
14	1	1.75	7	40	72.5	65.7	73.5	60.9
3	2	0.5	11	40	63.2	58.2	57.7	50.8
13	3	1.75	7	40	71.4	66.2	72.2	59.9
17	4	1.75	7	40	74.3	67.2	73.9	61.5
16	5	1.75	7	40	73.4	66.7	73.2	61.2
11	6	1.75	3	60	70.4	66.3	63.6	53.2
15	7	1.75	7	40	72.7	67.9	72.9	62.3
9	8	1.75	3	20	72.1	65	67.4	60.5
2	9	3	3	40	68	74.5	67.6	60.8
1	10	0.5	3	40	73.8	63.7	61.6	54.6
5	11	0.5	7	20	62.5	60.3	57.9	49.7
4	12	3	11	40	62.5	59.3	59.2	52.9
7	13	0.5	7	60	64	56.8	57.2	48.2
10	14	1.75	11	20	61.4	57	59.9	53.8
8	15	3	7	60	60.2	65.9	58.6	50.2
6	16	3	7	20	62.4	64.4	61.6	56.4
12	17	1.75	11	60	63.7	53.4	57.7	49.4

The resulting data obtained on Table 4.2, were analyzed using Design expert® 11.1.0.1 software to decide the effects of operating parameters; coagulant dose, pH and mixing time. The dependent variable used as a response parameter was the percentage of removal. All experiments were carried out in a randomized order to minimize the effect of unexpected variability in the observed response due to extraneous factors. Design Summary for percentage removal with three levels and three factors. The design model of the experiments are quadratic polynomial and the center point per blocks is five using Design expert® 11 software.

Table 4. 3 : Design Summary of factorial designs

Design Summary of Design expert® 11 software			
File Version	11.1.0.1		
Study Type	Response Surface	Subtype	Randomized
Design Type	Box-Behnken	Runs	17
Design Model	Quadratic	Blocks	No Blocks

To determine whether or not the quadratic model is significant, it was crucial to perform analysis of variance (ANOVA), table 4.3. The probability (P-values) values were used as a device to check the significance of each coefficient, which also showed the interaction strength of each parameter. The smaller the P-values are, the bigger the significance of the corresponding coefficient.

4.3.1. Effects of Experimental Variables for percentage removal of Color (%)

Table 4. 4 : Analysis of variance (ANOVA) for the percentage removal of color (%)

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	423.52	9	47.06	55.65	< 0.0001	Significant
A-Dose	13.52	1	13.52	15.99	0.0052	
B-PH	140.28	1	140.28	165.89	< 0.0001	
C-Mixing time	0.0013	1	0.0013	0.0015	0.9704	
AB	6.50	1	6.50	7.69	0.0276	
AC	3.42	1	3.42	4.05	0.0841	
BC	4.00	1	4.00	4.73	0.0661	
A^2	118.50	1	118.50	140.13	< 0.0001	
B^2	1.95	1	1.95	2.30	0.1730	
C^2	117.38	1	117.38	138.81	< 0.0001	
ABC	0.0000	0				
Residual	5.92	7	0.8456			
Lack of Fit	1.27	3	0.4225	0.3633	0.7841	not significant
Pure Error	4.65	4	1.16			
Cor Total	429.44	16				

F- Value is a test for comparing model variance with residual (error) variance. If the variances are close to the same, the ratio will be close to one and it is less likely that any of the factors have a significant effect on the response. It is calculated by Model Mean Square divided by Residual Mean Square. Here the Model F-value of 55.65 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to personal error or disturbance. Probability values and/ or "Prob > F" values less than 0.0500 indicate model terms are significant. In this case A (coagulant dose), B (pH), AB (coagulant dose &pH), A^2 (coagulant dose), C^2 (mixing time) are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

The lack of fit F-value of 0.36 implies the lack of fit is not significant relative to the pure error. There is a 78.41% chance that a lack of fit F-value this large could occur due to noise. Non-significant lack of fit is good so we want the model to fit.

Coefficient of variation, the standard deviation expressed as a percentage of the mean, predicted residual error, sum of Squares, which is a measure of how the model fits each point in the design; the R-Squared, measure of the amount of variation around the mean explained by the model; Adj R-Squared that is a measure of the amount of variation around the mean explained by the model, adjusted for the number of terms in the model. Pred R-Squared a measure of the amount of variation in new data explained by the model, and Adequate Precision, this is a signal to disturbance ratio due to random error, presented in the Table 4.5, below, are used to decide whether the model can be used or not.

Table 4. 5 : Model adequacy measures for color removal

Std. Dev.	0.9196		R^2	0.9862
Mean	67.56		Adjusted R^2	0.9685
C.V. %	1.36		Predicted R^2	0.9358
			Adeq Precision	19.2828

The "Pred R-Squared" of 0.9358 is a reasonable agreement as close to the "Adj R-Squared" of 0.9685 in less than 0.2 difference as one might expect. "Adeq Precision" measures the signal to disturbance ratio due to random error. A ratio greater than 4 is desirable. Here ratio of 19.283 indicates an adequate signal. Therefore, this model can be used to navigate the design space. By the designed experimental data from table 4.3, the quadratic polynomial model for percentage removal of color was retreated and shown as below:

Final Equation in Terms of Coded Factors

Color removal (%) = $+72.86 - 1.30*A - 4.19*B - 0.0125*C +1.28*AB-0.9250*AC+BC-5.31*A^2-0.6800*B^2-5.28*C^2$

Where, A-coagulant dose, B- pH, C-mixing time

The coefficient of factors shows as weather it is affected negatively if it is negative sign or positively if the coefficient is positive for the percentage removal of color.

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

Final Equation in Terms of Actual Factors

Color removal (%) = +52.46657+10.53820*Dose - 1.39813*pH+1.03263*Mixing time+0.255000*Dose *pH-0.037000*Dose * Mixing time+0.012500*pH * Mixing time-3.39520*Dose^2-0.042500*pH^2-0.013200*Mixing time^2

The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor.

The actual versus predicted values using model in the above equation

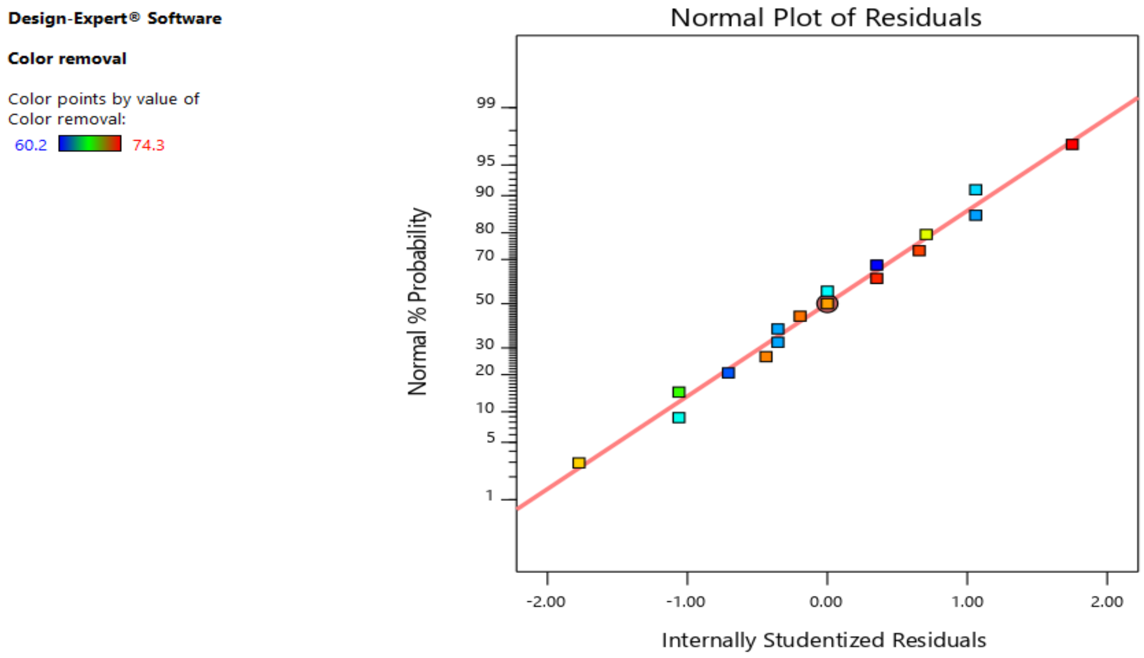

Figure 4. 2: Normal plots of residuals for color removal

From the plot as shown above, the normal probability plot indicates the residuals following a normal distribution, in the case of this experiment the points in the plots shows fit to a straight line in the figure, this shows that the quadratic polynomial model satisfies the assumptions analysis of variance (ANOVA) i.e. the error distribution is approximately normal.

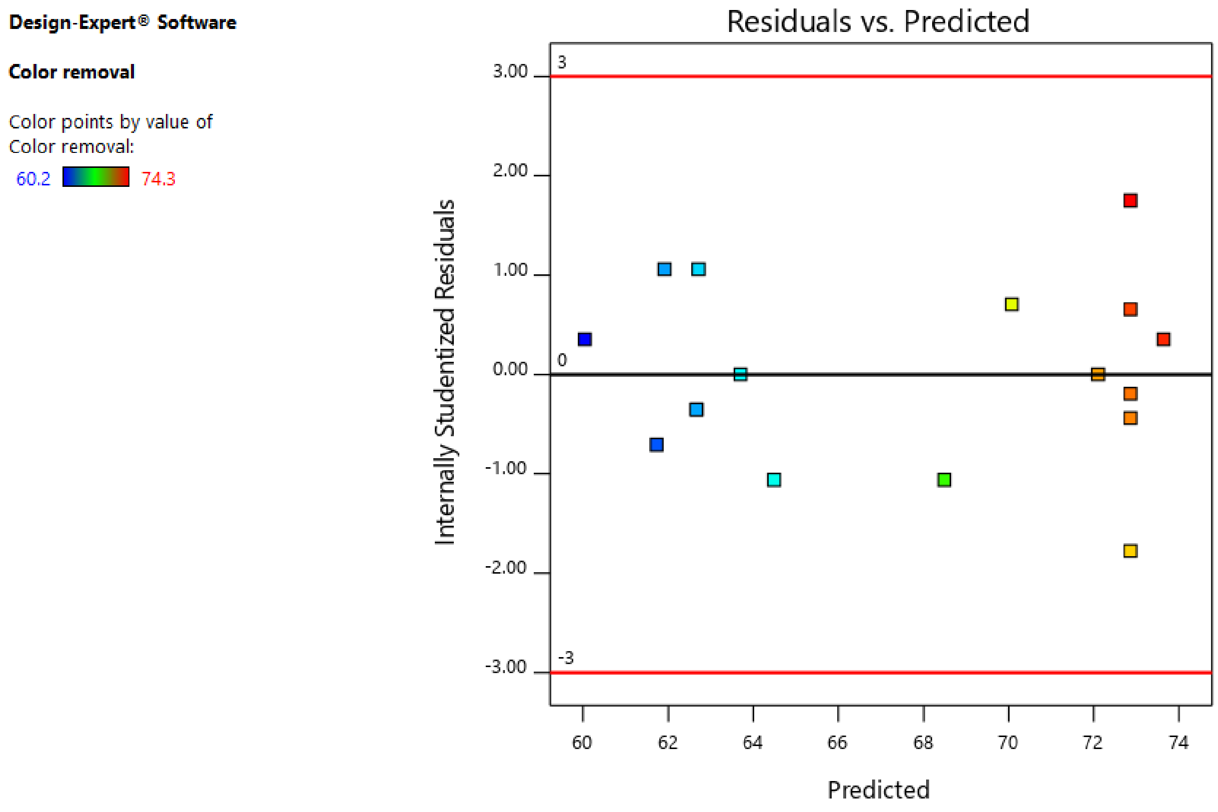

Figure 4. 3 : Residual versus predicted values for color removal

If the model is correct and the assumptions are satisfied, the residuals should be structure less; in particular, they should be unrelated to any other variable including the predicted response. A simple check is to plot the residuals versus the fitted (predicted) values. A plot of the residuals versus the rising predicted response values tests the assumption of constant variance. The plot shows random scatter which justifying no need for an alteration to minimize personal error.

The effects of the parameters for the percentage of color removal are to generate one factor response and surface plots of the equation. The one factor effect on response were plotted in figure (4.4), (4.5),(4.6) and three dimensional i.e. Interactions can be contour or 3D response surfaces effect were plotted in figures (4.7) below as a function of the interactions of any two of the variables by holding the other value of the variable at middle.

4.3.1.1. Effect of coagulant dose on percentage of color removal

Figure 4.4 shows the effect of coagulant dose on percentage removal of color, so coagulant dose is one of the most important parameter that has been considered to determine the performance of coagulants in coagulation and flocculation. 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 0.5 – 3g, while pH 7 and mixing time of 40 minute consisting of rapid mixing 100 rpm for 2 minutes and slow mixing 40 rpm and 60 minute settling time. Accordingly, there was continuous removal of color with increases in coagulant doses up to 1.75g and then decline from 1.75-3g because the removal rate of color was low when the amount of dose was below 1.75g, because the amount of coagulant is inadequately to destabilize the particles. On the other hand, the removal of color is lower when the coagulant dose is greater than 1.75g, because the aggregated particles redisperse & disturb particle settling due to excess amount of the coagulant added. The minimum removal efficiency of color was observed at coagulant dose of 3g which is around 66 % and the maximum was observed at around 1.75g which is 74%.

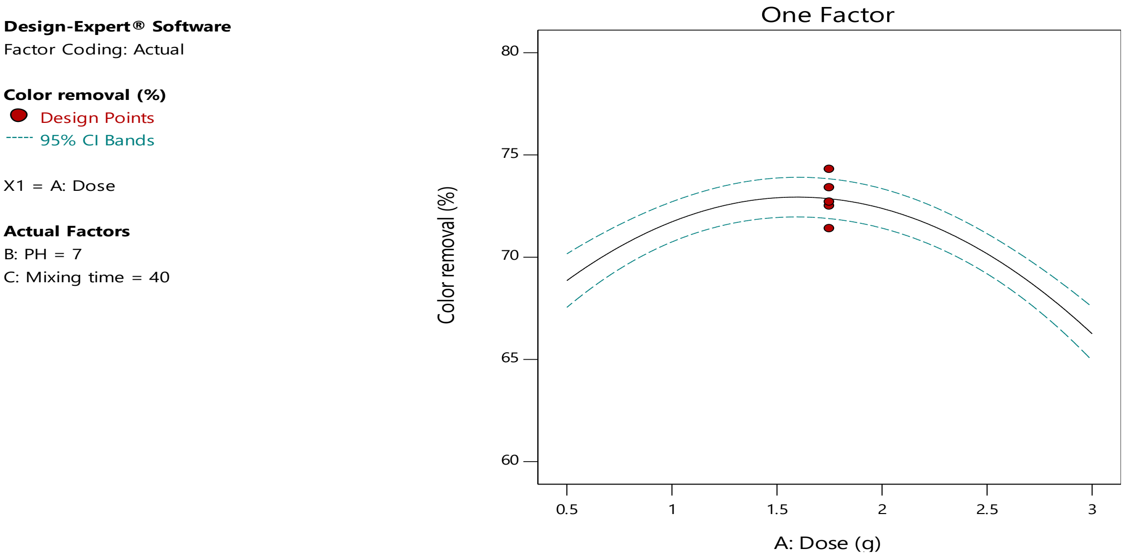

Figure 4. 4 : Effect of coagulant dose on percentage of color removal

4.3.1.2. Effect of pH on percentage removal of color

The effect of pH was analyzed by varying dose from 1.75g and mixing time is 40 minute which consisting the other parameters constant for a range of pH which varied from 3 to 11 and the coagulation experiment clearly affirmed that the percentage removal to be good for color at acidic condition than basic particularly at pH 3. The minimum removal efficiency is at pH of 11, which is around 65% and maximum removal was observed at pH of 3 which is around 74%. At some point particles surface charge were reduced or neutralized as well as destabilized the suspension. The removal capacity has gradually declined as pH increase beyond 3 and as pH approaches basic condition it is proved to decrease the solubility of blended coagulant. Because, as pH increased beyond the optimum positive charges of cactus surface decreased significantly. Consequently, the contribution charge neutralization roles of the coagulant to destabilize the particles become less. Moreover, at pH 3 the result showed that flocs produced by blended coagulant were rapid and caused large size of flocs formation which was important for easiness

of the settlement.

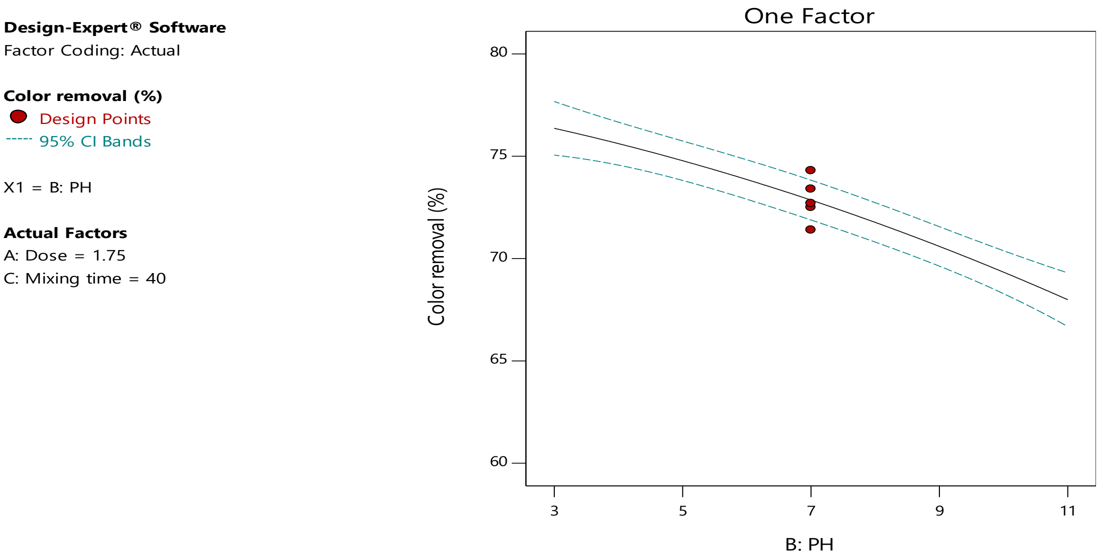

Figure 4. 5: Effect of pH on percentage of color removal

4.3.1.3. Effect of mixing time on percentage removal of color

The time of macrofloc formation (flocculation time) is one of the operating parameters that is given great consideration in any water treatment plant that involves coagulation–flocculation operations. Figure 4.6 represents the effect of flocculation time for removal of color was tested by varying 1.75g coagulant dose, and at pH 7, which consists of rapid mixing 100 rpm for 2 minute and slow mixing time from 20 to 60 minutes with 60 minutes of settling time. The experimental result shows that there was a continuous removal of color, while increasing the mixing time from 20 to 40 minutes. When the mixing time is short (<40 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 (>40 min) it would lead to an increase in flocs breakage & limit the size of the floc formed. It is a great importance to optimize the mixing time so as to increase removal performance of the coagulation.

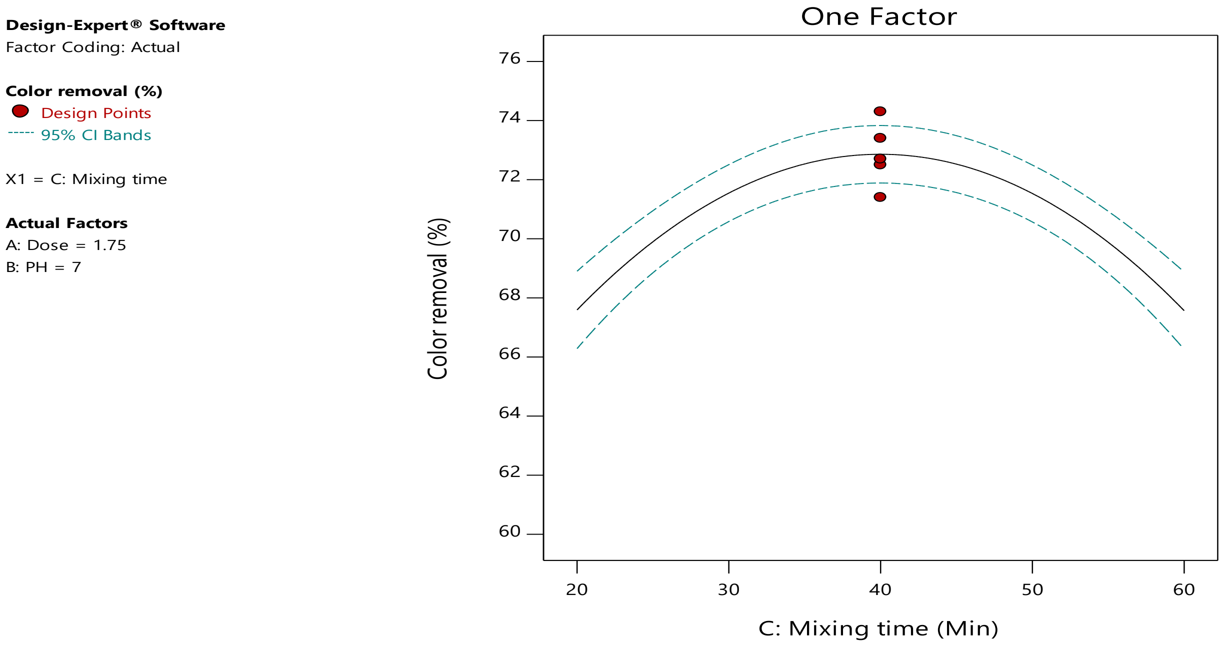

Figure 4. 6: Effect of mixing time on color removal

4.3.1.4. Interaction effect on percentage removal of color

The possible interactions that can affect the percentage removal of color was only pH and coagulant dose. Figures 4.7 and Tables 4.4 from analysis of variance, demonstrate the significant interaction effects that exist between the processing variables. As observed from the Figures and Tables, the only significant interaction that affects percentage removal of color is between pH and coagulant dose. According to the study, interaction between pH and coagulant dose, have increased the percentage removal of the color.

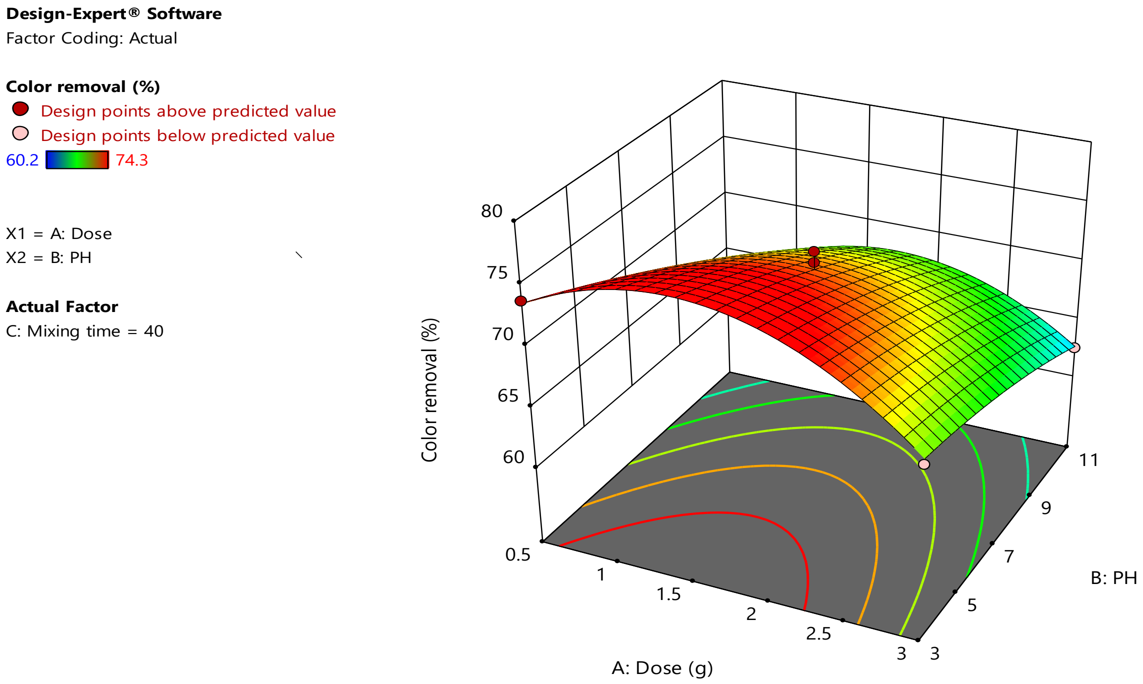

Figure 4. 7: 3D plot of the interaction effect of dose and pH on Color removal

4.3.2. Effects of Experimental Variables for percentage removal of COD (%)

Table 4. 6 : Analysis of variance (ANOVA) for the percentage removal of COD (%)

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	435.02	9	48.34	107.09	< 0.0001	Significant
A-Dose	78.75	1	78.75	174.48	< 0.0001	
B-PH	216.32	1	216.32	479.27	< 0.0001	
C-Mixing time	2.31	1	2.31	5.12	0.0581	
AB	23.52	1	23.52	52.12	0.0002	
AC	6.25	1	6.25	13.85	0.0074	
BC	6.00	1	6.00	13.30	0.0082	
A^2	2.03	1	2.03	4.51	0.0714	
B^2	18.92	1	18.92	41.93	0.0003	
C^2	74.10	1	74.10	164.16	< 0.0001	
Residual	3.16	7	0.4514			
Lack of Fit	0.2275	3	0.0758	0.1035	0.9538	not significant
Pure Error	2.93	4	0.7330			
Cor Total	438.18	16				

F- Value is a test for comparing model variance with residual (error) variance. If the variances are close to the same, the ratio will be close to one and it is less likely that any of the factors have a significant effect on the response. It is calculated by Model Mean Square divided by Residual Mean Square. Here the Model F-value of 107.09 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to personal error or disturbance. Probability values and/ or "Prob > F" values less than 0.0500 indicate model terms are significant. In this case A (coagulant dose), B (pH), AB (coagulant dose &pH), AC (coagulant dose & mixing time), BC (coagulant dose & mixing time) B^2 (pH), C^2 (mixing time) are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

The lack of fit F-value of 0.10 implies the lack of fit is not significant relative to the pure error. There is a 95.38% chance that a lack of fit F-value this large could occur due to noise. Non-significant lack of fit is good so we want the model to fit.

Table 4. 7 : Model adequacy measures for COD removal

Std. Dev.	0.6718	R^2	0.9928
Mean	63.44	Adjusted R^2	0.9835
C.V. %	1.06	Predicted R^2	0.9812
		Adeq Precision	41.1919

The "Pred R-Squared" of 0.9812 is reasonable agreement as close to the "Adj R-Squared" of 0.9835 in less than 0.2 difference as one might expect. "Adeq Precision" measures the signal to disturbance ratio due to random error. A ratio greater than 4 is desirable. Here ratio of 41.1919 indicates an adequate signal. Therefore, this model can be used to navigate the design space. The quadratic polynomial model for percentage removal of COD was retreated and shown as below: Final Equation in Terms of Coded Factors

COD removal (%) = $+66.74+3.14*A-5.20*B-0.5375*C-2.43*AB+1.25*AC-1.22*BC-0.6950*A^2$
$-2.12*B^2-4.19*C^2$
Where A- Coagulant dose, B- pH and C- mixing time

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

Final Equation in Terms of Actual Factors
COD removal (%) = +41.15905+5.46180*Dose+2.01625*pH+0.831812*Mixing time -
0.485000*Dose * pH+0.050000*Dose * Mixing time-0.015312*pH * Mixing time-
0.444800*Dose^2-0.132500*pH^2 0.010488*Mixing time^2

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

Figure 4.8 the plot as shown below indicates, the normal probability plot indicates the residuals following a normal distribution, in the case of this experiment the points in the plots shows fit to a straight line in the figure, this shows that the quadratic polynomial model satisfies the assumptions analysis of variance (ANOVA) i.e. the error distribution is approximately normal.

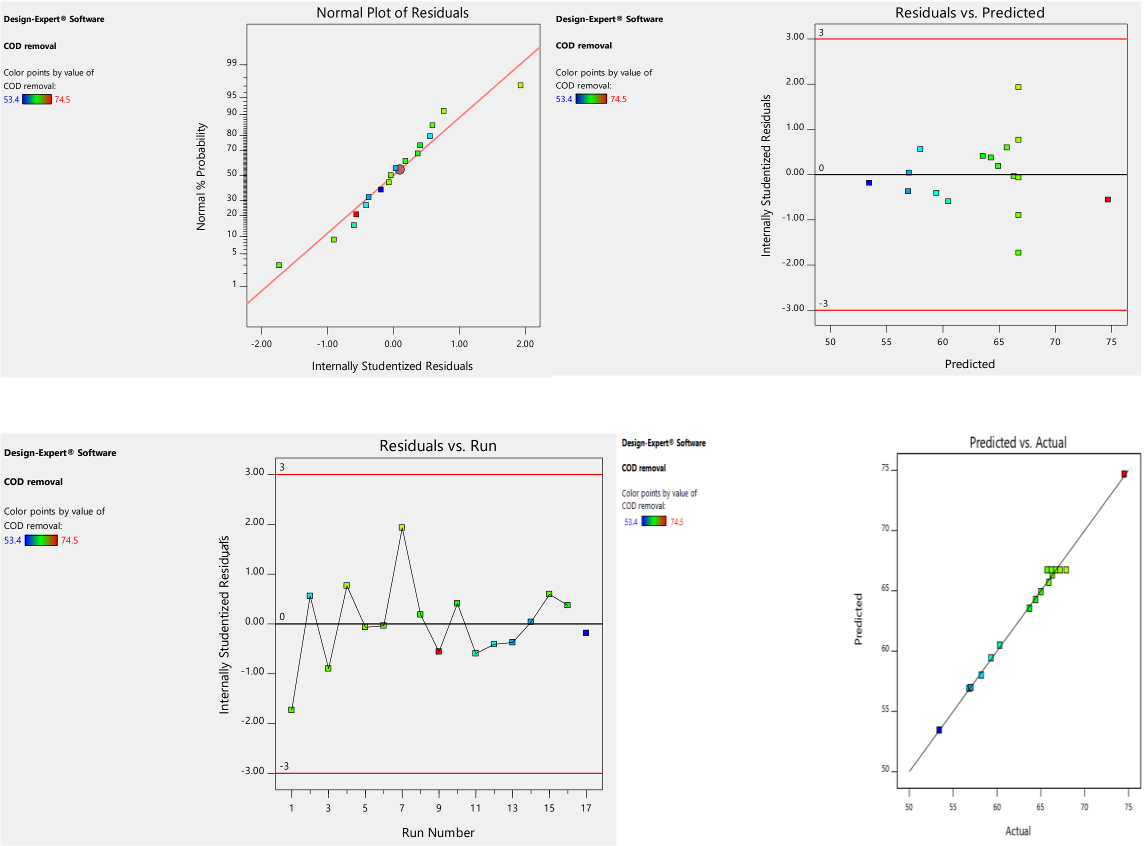

Figure 4. 8: Diagnostics graphs in terms of coded factors

4.3.2.1. Effect of coagulant dose on percentage removal of COD

Figure 4.9 shows the effect of coagulant dose on percentage removal of color, so coagulant dose is one of the most important parameter that has been considered to determine the performance of coagulants in coagulation and flocculation. 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 0.5 – 3g, while pH 7 and mixing time of 40 minute consisting of rapid mixing 100 rpm for 2 minutes and slow mixing 40 rpm and 60 minute settling time. Accordingly, there was continuous removal of COD with increases in coagulant doses up to 3g, when the amount of dose was below 3g the amount of coagulant is inadequately to destabilize the particles. The minimum removal efficiency of COD was observed at coagulant dose of 0.5g which is around 63% and the maximum was observed at around 3g which is 70%.

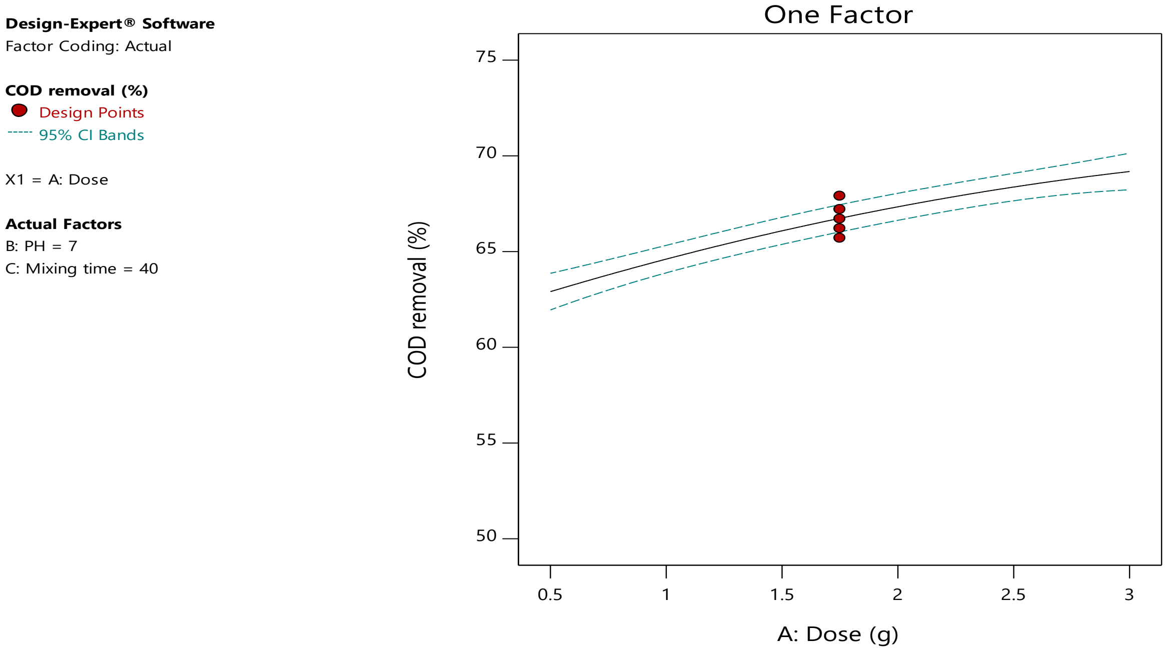

Figure 4. 9: Effect of dose on COD removal

4.3.2.2. Effect of pH on percentage removal of COD

The effect of pH was analyzed by varying pH from 3-11 and constant mixing time as well as dose which is 40 minute, 1.75g respectively, which consisting of rapid mixing 100 rpm for 2 minutes and slow mixing 40 rpm and 60 minute setting time and the coagulation experiment clearly affirmed that the percentage removal to be good for COD at acidic condition than basic, particularly at pH 3. The minimum removal efficiency is at pH of 11, which is around 59% and maximum removal was observed at pH of 3 which is around 69%. At some point particles surface charge were reduced or neutralized as well as destabilized the suspension. The removal capacity has gradually declined as pH increase beyond 3 and as pH approaches basic condition it is proved to decrease the solubility of blended coagulant. Because, as pH increased beyond the optimum positive charges of cactus surface decreased significantly. Consequently, the contribution charge neutralization roles of the coagulant to destabilize the particles become less. Moreover, at pH 3 the result showed that flocs produced by blended coagulant were rapid and caused large size of flocs formation which was important for easiness of the settlement. Therefore, the blended coagulant is effective for the treatment of textile wastewater in acid condition.

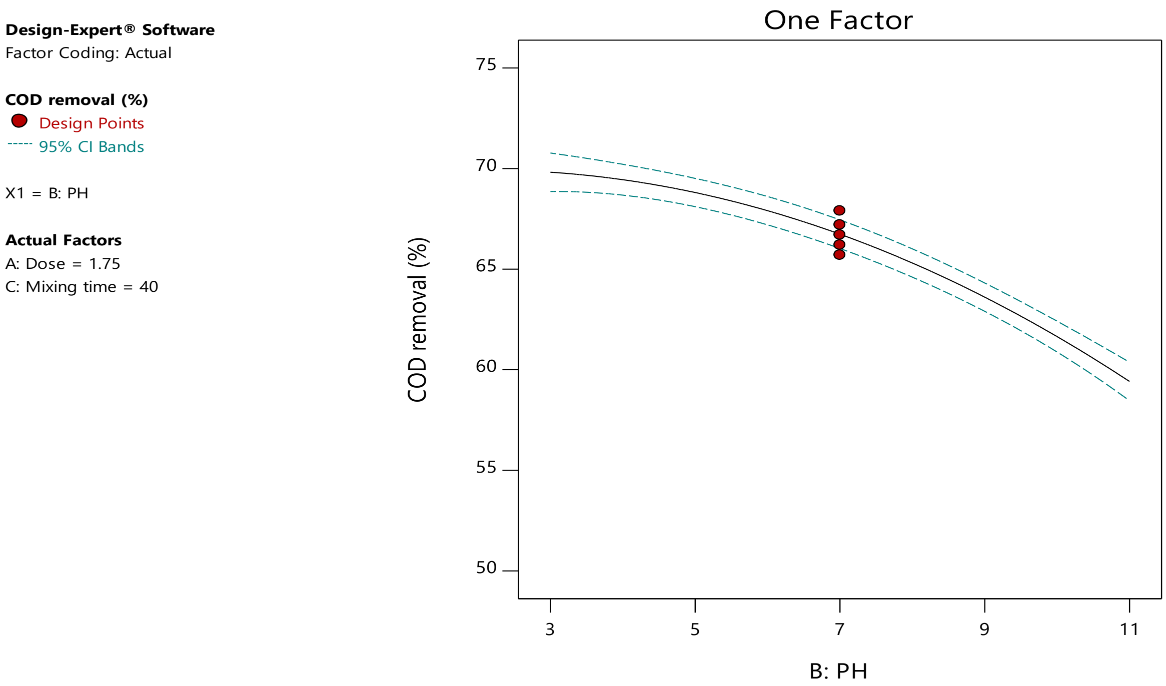

Figure 4. 10: Effect of pH on COD removal

4.3.2.3. Interaction effect on percentage removal of COD

The possible interactions that can affect the percentage removal of COD analyzed using ANOVA. Figures 4.11, 4.12 and Tables 4.6 from analysis of variance, demonstrate the significant interaction effects that exist between the processing variables. As observed from the Figures and Tables, the only significant interaction that affects percentage removal of COD is between pH and coagulant dose, pH and mixing time as well as coagulant dose and mixing time. According to the study, interactions between the operating variables have increased the percentage removal of the COD.

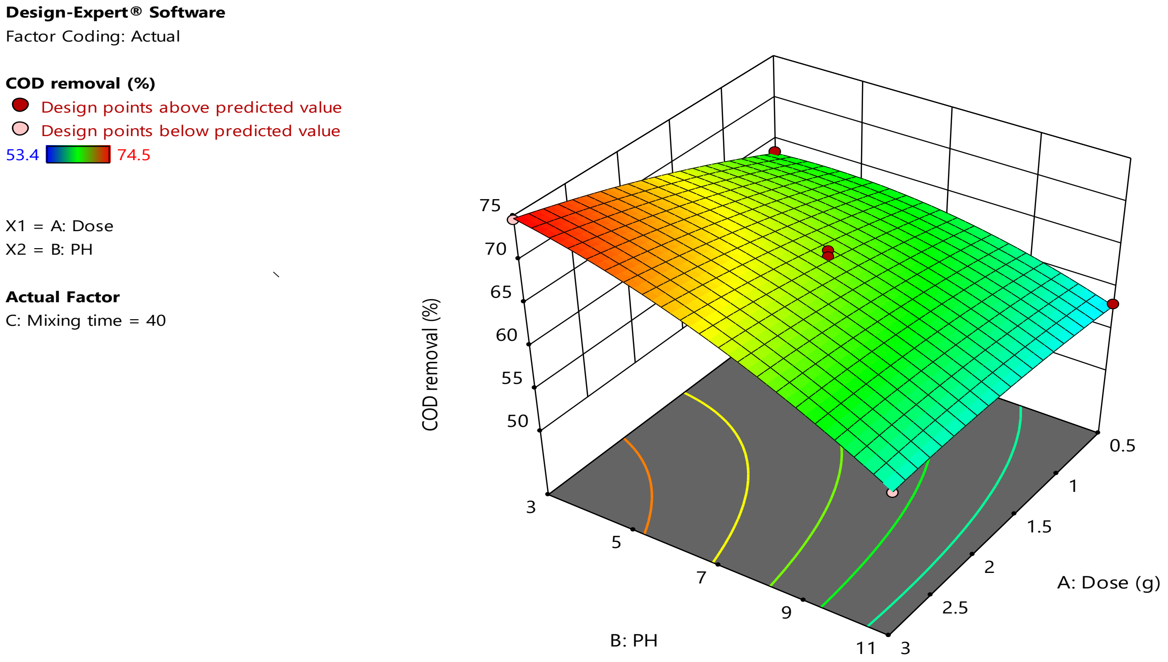

Figure 4. 11: 3D plot of the interaction of dose and pH on COD removal

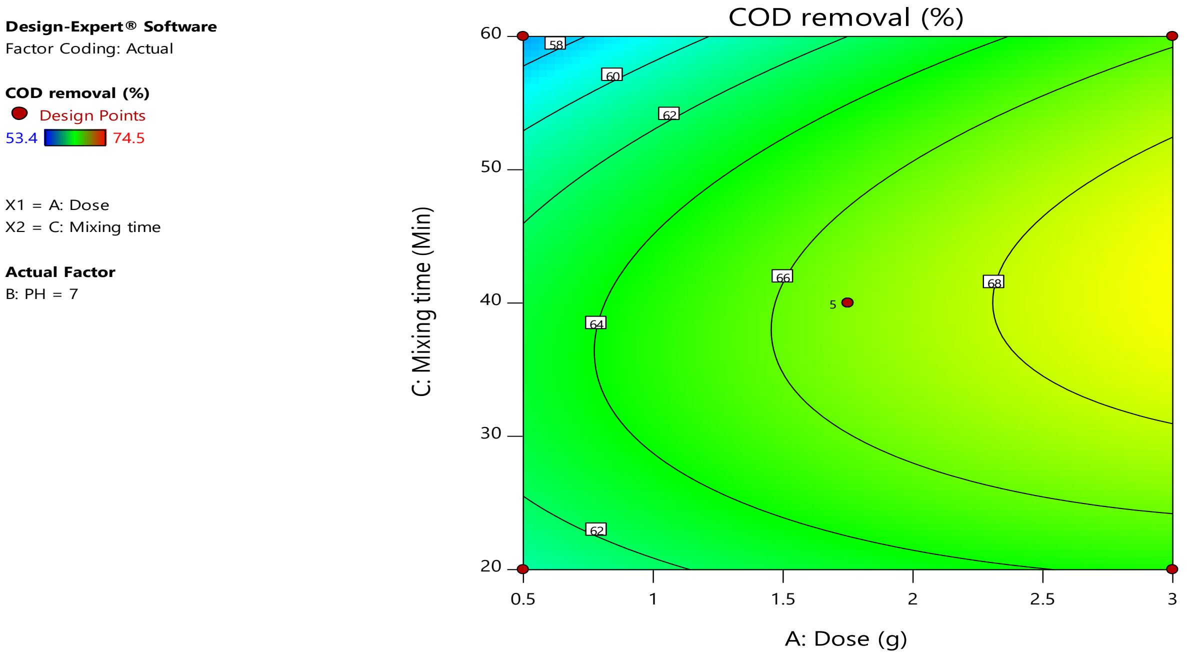

Figure 4. 12: Contour plot of the interaction of dose and mixing time on COD removal

4.3.3. Effects of Experimental Variables for percentage removal of TSS (%)

Table 4. 8 : Analysis of variance (ANOVA) for the percentage removal of TSS (%)

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	680.72	9	75.64	166.26	< 0.0001	Significant
A-Dose	19.85	1	19.85	43.62	0.0003	
B-PH	82.56	1	82.56	181.48	< 0.0001	
C-Mixing time	11.76	1	11.76	25.85	0.0014	
AB	5.06	1	5.06	11.13	0.0125	
AC	1.32	1	1.32	2.91	0.1320	
BC	0.6400	1	0.6400	1.41	0.2743	
A^2	234.95	1	234.95	516.46	< 0.0001	
B^2	72.34	1	72.34	159.02	< 0.0001	
C^2	197.28	1	197.28	433.65	< 0.0001	
Residual	3.18	7	0.4549			
Lack of Fit	1.53	3	0.5108	1.24	0.4064	not significant
Pure Error	1.65	4	0.4130			
Cor Total	683.90	16				

F- Value is a test for comparing model variance with residual (error) variance. If the variances are close to the same, the ratio will be close to one and it is less likely that any of the factors have a significant effect on the response. It is calculated by Model Mean Square divided by Residual Mean Square. Here the Model F-value of 166.26 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to personal error or disturbance. Probability values and/ or "Prob > F" values less than 0.0500 indicate model terms are significant. In this case A (coagulant dose), B (pH), C (mixing time), AB (coagulant dose &pH), A^2 (coagulant dose), B^2 (pH), C^2 (mixing time) are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

The lack of fit F-value of 1.24 implies the lack of fit is not significant relative to the pure error. There is a 40.64% chance that a lack of fit F-value this large could occur due to noise. Non-significant lack of fit is good.

Coefficient of Variation, the standard deviation expressed as a percentage of the mean; Predicted Residual Error, Sum of Squares, which is a measure of how the model fits each point in the design; the R-Squared, measure of the amount of variation around the mean explained by the model; Adj R-Squared that is a measure of the amount of variation around the mean explained by the model, Pred R-Squared a measure of the amount of variation in new data explained by the model, and Adequate Precision, this is a signal to disturbance ratio due to random error, presented in the table 4.11, below, are used to decide whether the model can be used or not.

Table 4. 9 : Model adequacy measures for TSS removal

Std. Dev.	0.6745	R^2	0.9953
Mean	64.45	Adjusted R^2	0.9894
C.V. %	1.05	Predicted R^2	0.9604
		Adeq Precision	31.9492

The "Pred R-Squared" of 0.9604 is reasonable agreement as close to the "Adj R-Squared" of 0.9894 in less than 0.2 difference as one might expect. "Adeq Precision" measures the signal to disturbance ratio due to random error. A ratio greater than 4 is desirable. Here ratio of 31.9492 indicates an adequate signal. Therefore, this model can be used to navigate the design space. The quadratic polynomial model for percentage removal of TSS was retreated and shown as below:

Final Equation in Terms of Coded Factors

$$\text{TSS removal (\%)} = +73.14+1.58*A-3.21*B-1.21*C-1.13*AB-0.5750*AC+0.4000*BC-7.47*A^2 -4.15*B^2-6.84*C^2$$

Where, A-coagulant dose, B- pH, C-mixing time

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

Final Equation in Terms of Actual Factors

TSS removal (%) = 21.3004 + 20.4878 * Dose + 3.0175 * pH + 1.31363 * Mixing time -0.225 * Dose * pH-0.023 * Dose * Mixing time + 0.005 * pH * Mixing time -4.7808 * Dose^2 + -0.259063 * pH^2 -0.0171125 * Mixing time^2

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

From the plot as shown in figure 4.13, the normal probability plot indicates the residuals following a normal distribution, in the case of this experiment the points in the plots shows fit to a straight line in the figure, this shows that the quadratic polynomial model satisfies the assumptions analysis of variance (ANOVA) i.e. the error distribution is approximately normal.

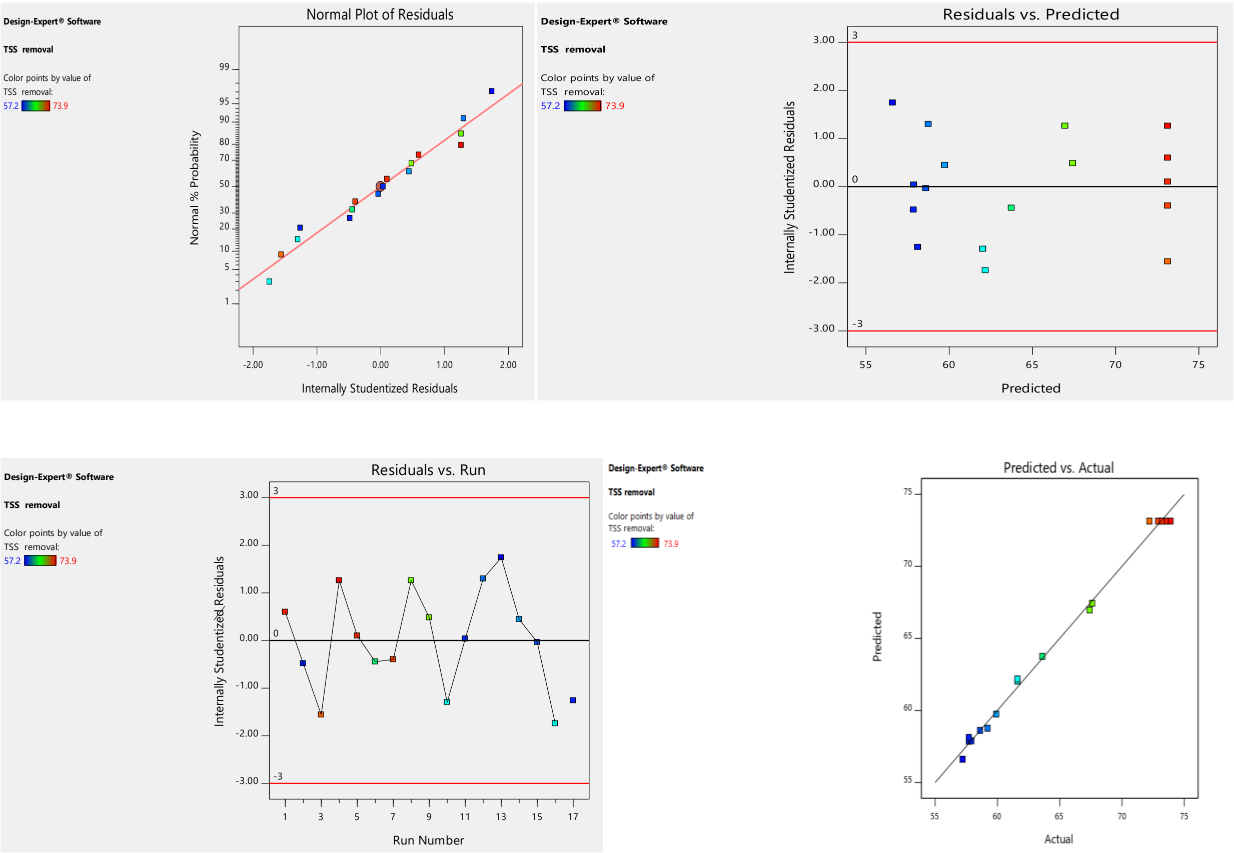

Figure 4. 13: Diagnostics graphs in terms of coded factors

4.3.3.1. Effect of coagulant dose on percentage removal of TSS

The total suspended solids removal efficiency of blended coagulant was determined by adding different doses of cactus and alum powder. As shown in Figure 4.14 the percentage removal of TSS from the colloidal and suspended wastewater sample was increased from 63% to 74% with the increment of coagulant dose from 0.50 to 1.75 g. This increment in removal of TSS is due to increment of active site of the coagulant. Similarly, the percentage removal of TSS from colloidal and suspended wastewater is decreased from 74%% to 66% as the dose of coagulant increased from 1.75 g to 3g because as dose is high chemical coating of the particles happen that is way the determination of optimum point of the variables was needed.

The suspended solid of the treated textile waste water was decreased highly with an increase of the dose of blended coagulant. This is because, when the negatively charged suspended particle of textile waste water mix with a sufficient amount of blended coagulant (positively charged ions), the suspended particles present in the untreated textile waste water becomes neutralized, (Okuda *et al*., 1999). Once the repulsive charges of the suspended particles has been neutralized since opposite charges attract each other, the van der waals force causes the particles to agglomerate and form floc which in turn enable them to settle down.

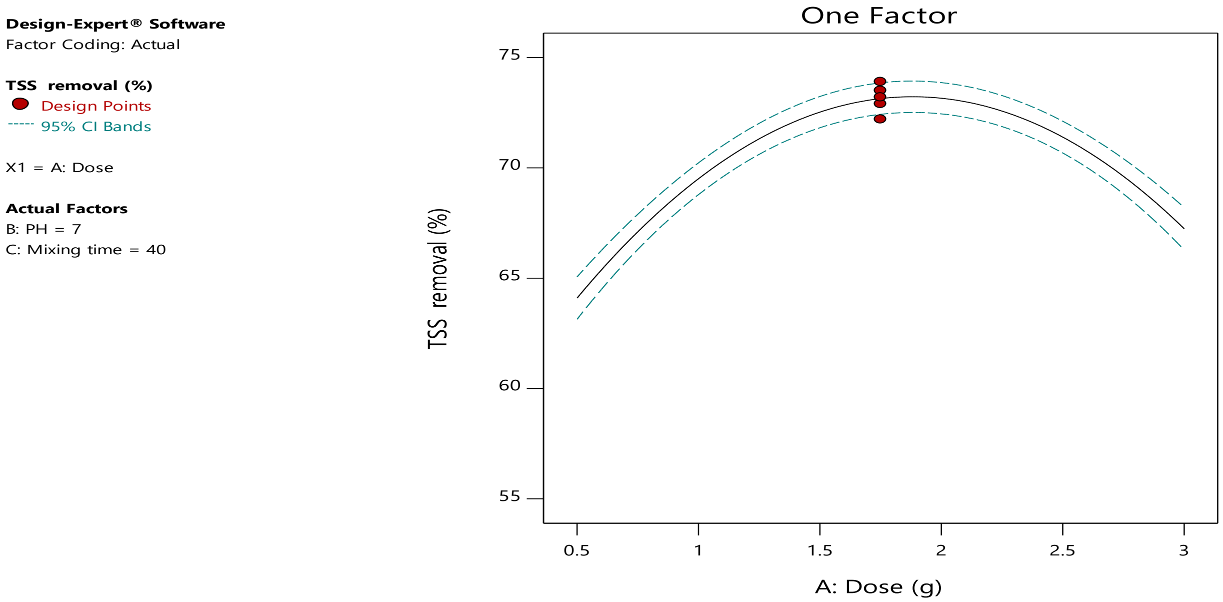

Figure 4. 14: Effect of dose on TSS removal

4.3.3.2. Effect of pH on percentage removal of TSS

The effect of pH was analyzed by varying pH from 3-11 and constant mixing time as well as dose which is 40 minute, 1.75g respectively, which consisting of rapid mixing 100 rpm for 2 minutes and slow mixing 40 rpm and 60 minute setting time and the coagulation experiment clearly affirmed that the percentage removal to be good for TSS at acidic condition than basic particularly at pH 6. The minimum removal efficiency is at pH of 11, which is around 65% and maximum removal was observed at pH of 6 which is around 73%. At some point particles surface charge were reduced or neutralized as well as destabilized the suspension. The removal capacity has gradually declined as pH increase beyond 6 and as pH approaches basic condition it is proved to decrease the solubility of blended coagulant. Because, as pH increased beyond the optimum positive charges of cactus surface decreased significantly. Consequently, the contribution charge neutralization roles of the coagulant to destabilize the particles become less. Moreover, at pH around 6 the result showed that flocs produced by blended coagulant were rapid and caused large size of flocs formation which was important for easiness of the settlement. Therefore, the blended coagulant is effective for the treatment of textile wastewater in acid condition.

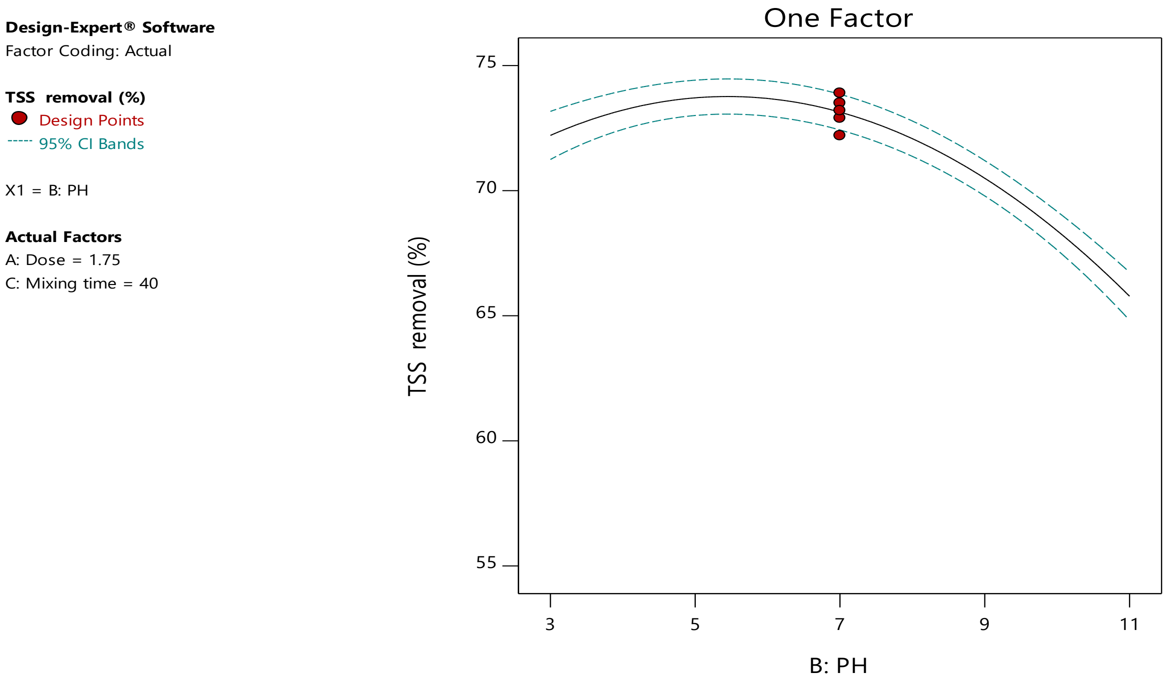

Figure 4. 15: Effect of pH on TSS removal

4.3.3.3. Effect of mixing time on percentage removal of TSS

Figure 4.16 indicates as the effect of mixing time on removal of TSS was tested by varying 0.5-3g and constant coagulant dose 1.75g as well as pH of 7, which consists of rapid mixing 100 rpm for 2 minute and slow mixing time from 20 to 60 minutes with 60 minutes of settling time. The experimental result shows that there was a continuous removal of color, while increasing the mixing time from 20 to 40 minutes. When the mixing time is short (<40 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 (>40 min) it would lead to an increase in flocs breakage & limit the size of the floc formed. It is a great importance to optimize the mixing time so as to increase removal performance of the coagulation. The minimum percentage removal was observed at mixing time of 60 minutes which is around 65% and maximum percentage removal was observed at 40 minutes which is around 73% that is way the optimum point of the variables is needed.

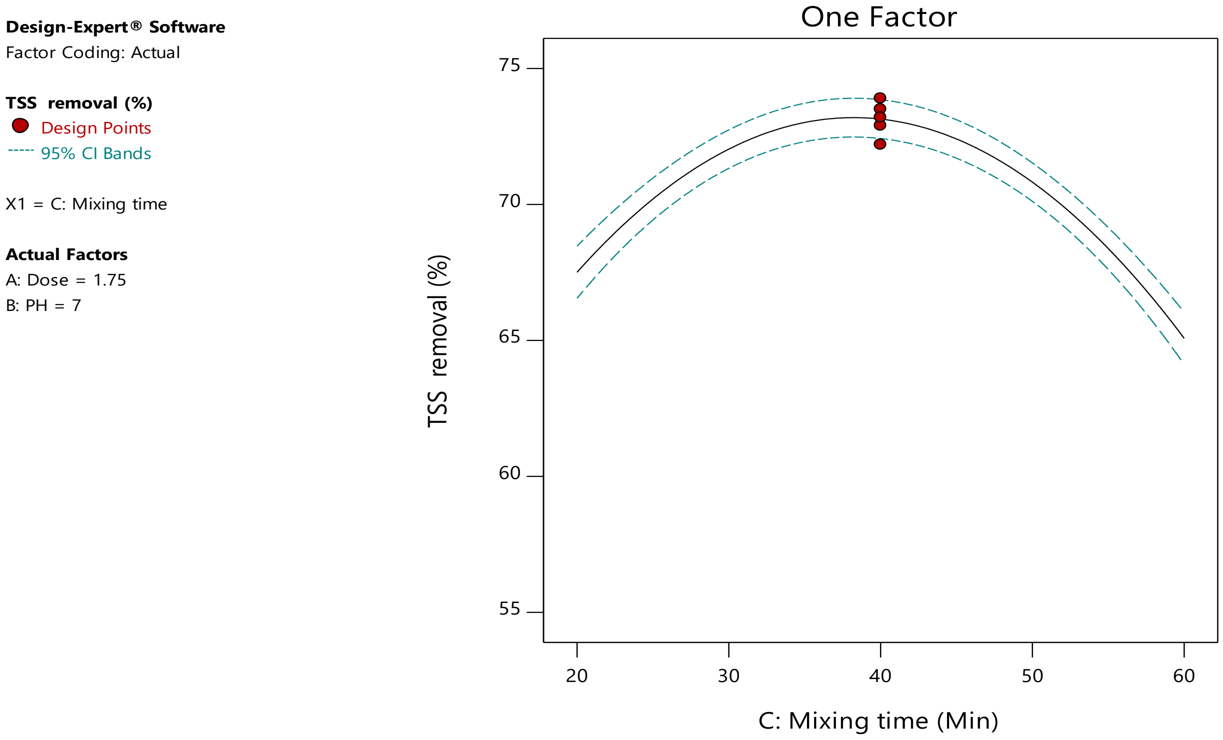

Figure 4. 16: Effect of mixing time on TSS removal

4.3.3.4. Interaction effect on percentage removal of TSS

The possible interactions that can affect the percentage removal of TSS were pH and coagulant dose. Figures 4.17 and Tables 4.8 from analysis of variance, demonstrate the significant interaction effects that exist between the processing variables. As observed from the Figures and Tables, the only significant interaction that affects percentage removal of TSS is between pH and coagulant dose. According to the study, interaction between pH and coagulant dose, have increased the percentage removal of the TSS.

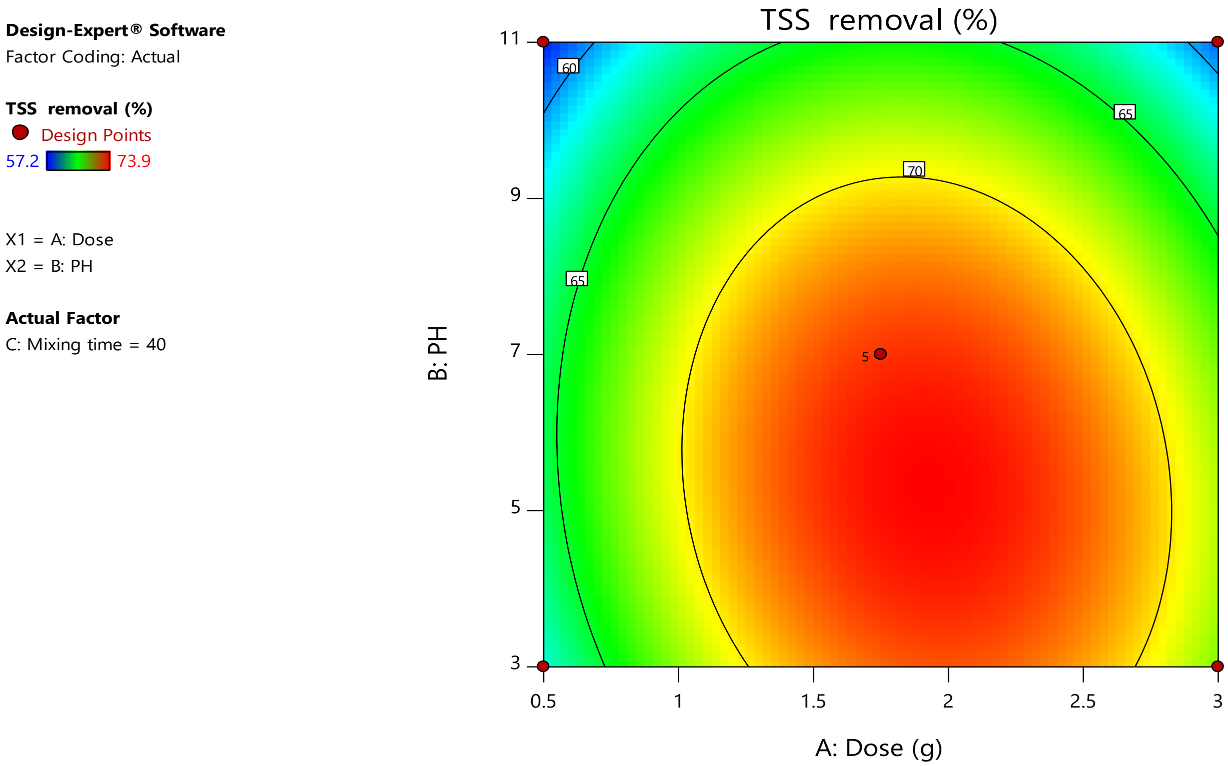

Figure 4. 17: Contour plot of the interaction effect of dose and pH on TSS removal

4.3.4. Effects of Experimental Variables for percentage removal of TDS (%)

Table 4. 10 : Analysis of variance (ANOVA) for the percentage removal of TDS (%)

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	401.49	9	44.61	59.23	< 0.0001	significant
A-Dose	36.12	1	36.12	47.97	0.0002	
B-PH	61.61	1	61.61	81.80	< 0.0001	
C-Mixing time	47.04	1	47.04	62.46	< 0.0001	
AB	4.20	1	4.20	5.58	0.0502	
AC	5.52	1	5.52	7.33	0.0303	
BC	2.10	1	2.10	2.79	0.1387	
A^2	94.70	1	94.70	125.74	< 0.0001	
B^2	11.36	1	11.36	15.08	0.0060	
C^2	117.94	1	117.94	156.60	< 0.0001	
Residual	5.27	7	0.7531			
Lack of Fit	2.20	3	0.7333	0.9549	0.4948	not significant
Pure Error	3.07	4	0.7680			
Cor Total	406.76	16				

F- Value is a test for comparing model variance with residual (error) variance. If the variances are close to the same, the ratio will be close to one and it is less likely that any of the factors have a significant effect on the response. It is calculated by Model Mean Square divided by Residual Mean Square. Here the Model F-value of 59.23 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to personal error or disturbance. Probability values and/ or "Prob > F" values less than 0.0500 indicate model terms are significant. In this case A (coagulant dose), B (pH), C (mixing time), AC (coagulant dose &mixing time), A^2 (coagulant dose), B^2 (pH), C^2 (mixing time) are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

The lack of fit F-value of 0.95 implies the lack of fit is not significant relative to the pure error. There is a 49.48% chance that a lack of fit F-value this large could occur due to noise. Non-significant lack of fit is good so we want the model to fit.

Coefficient of Variation, the standard deviation expressed as a percentage of the mean; Predicted Residual Error, Sum of Squares, which is a measure of how the model fits each point in the design; the R-Squared, measure of the amount of variation around the mean explained by the model; Adj R-Squared that is a measure of the amount of variation around the mean explained by the model, Pred R-Squared a measure of the amount of variation in new data explained by the model, and Adequate Precision, this is a signal to disturbance ratio due to random error, presented in the table 4.14, below, are used to decide whether the model can be used or not.

Table 4. 11 : Model adequacy measures for TDS removal

Std. Dev.	0.8678	R^2	0.9870
Mean	55.66	Adjusted R^2	0.9704
C.V. %	1.56	Predicted R^2	0.9017
		Adeq Precision	20.1472

The "Pred R-Squared" of 0.9017 is reasonable agreement as close to the "Adj R-Squared" of 0.9704 in less than 0.2 difference as one might expect. "Adeq Precision" measures the signal to disturbance ratio due to random error. A ratio greater than 4 is desirable. Here ratio of 20.1472 indicates an adequate signal. Therefore, this model can be used to navigate the design space. The quadratic polynomial model for percentage removal of TDS was retreated and shown as below:

Final Equation in Terms of Coded Factors

TDS removal (%) = $61.16 + 2.125 * A-2.775 * B-2.425 * C-1.025 * AB-1.175 * AC + 0.725 * BC-4.7425 * A^2-1.6425 * B^2-5.2925 * C^2$

Where, A-coagulant dose, B- pH, C-mixing time

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

Final Equation in Terms of Actual Factors

TDS removal (%) = 29.132 + 15.6382 * Dose + 0.739687 * pH + 0.956063 * Mixing time - 0.205 * Dose * pH-0.047 * Dose * Mixing time + 0.0090625 * pH * Mixing time-3.0352 * Dose^{2}-0.102656 * pH^{2}-0.0132313 * Mixing time^{2}

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

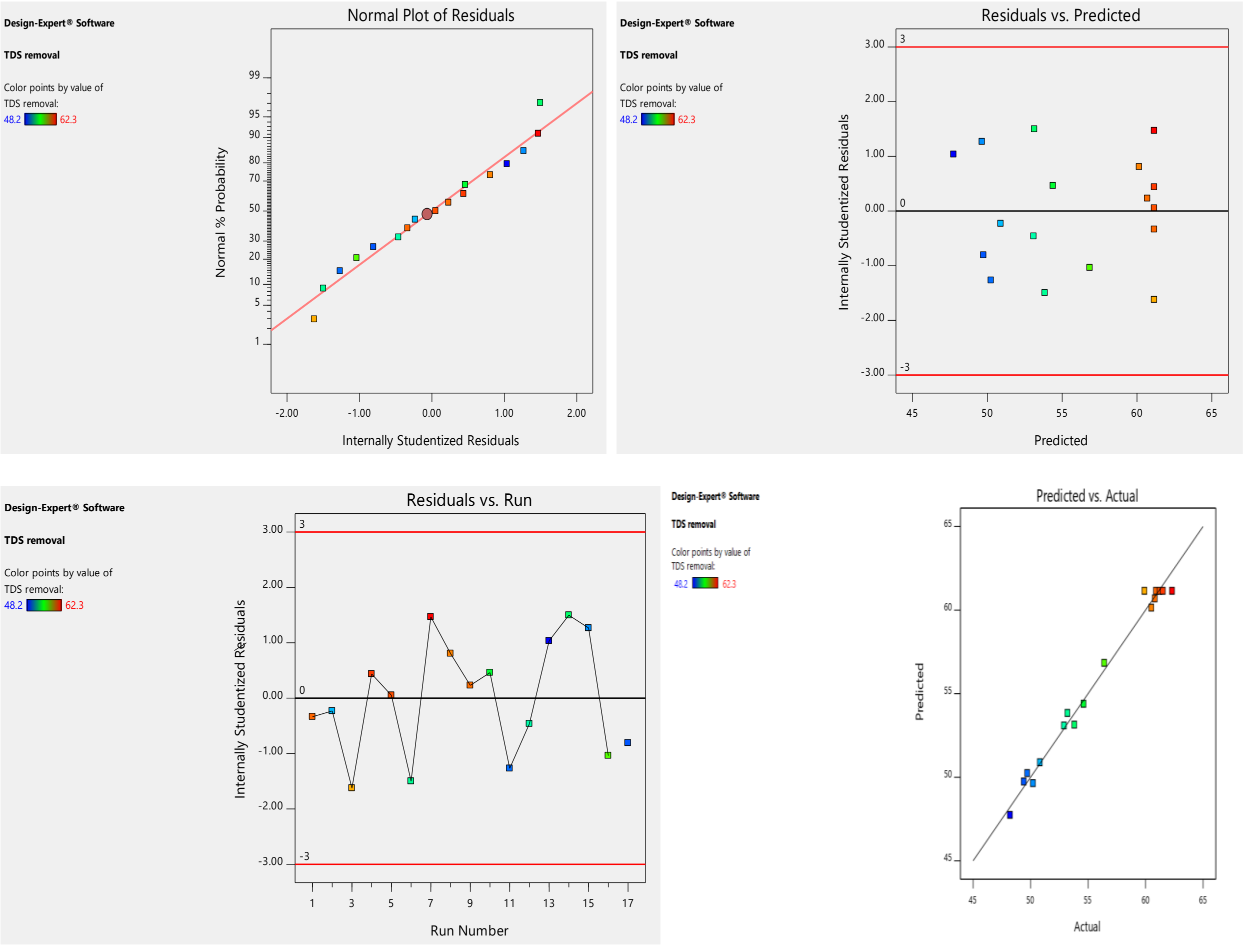

Figure 4. 18: Diagnostics graphs in terms of coded factors

4.3.4.1. Effect of coagulant dose on percentage removal of TDS

TDS is mostly used to express the concentration of dissolved minerals in a given wastewater sample. It contains carbonates, chlorides, sulphates, phosphates, and nitrates of calcium, magnesium, sodium, and potassium. It increases the conductivities of water due to the presence of those dissolved impurities. The principal anions contributing to the TDS value include carbonate, bicarbonate, chloride, sulphate and nitrates, and cations: calcium, magnesium, potassium, and sodium. The presence of those dissolved solids in the wastewater sample can comprise inorganic salt and organic matter.

As shown in Figure 4.19, percentage removals of TDS are increased from 54% to 62% as the dose of coagulant increased from 0.5 g to 1.75 g. In the same way, percentage removals of TDS are decreased from 62% to 59% as the dose of coagulant is increased from 1.75 g to 3 g that is way the optimum points of the variables was needed.

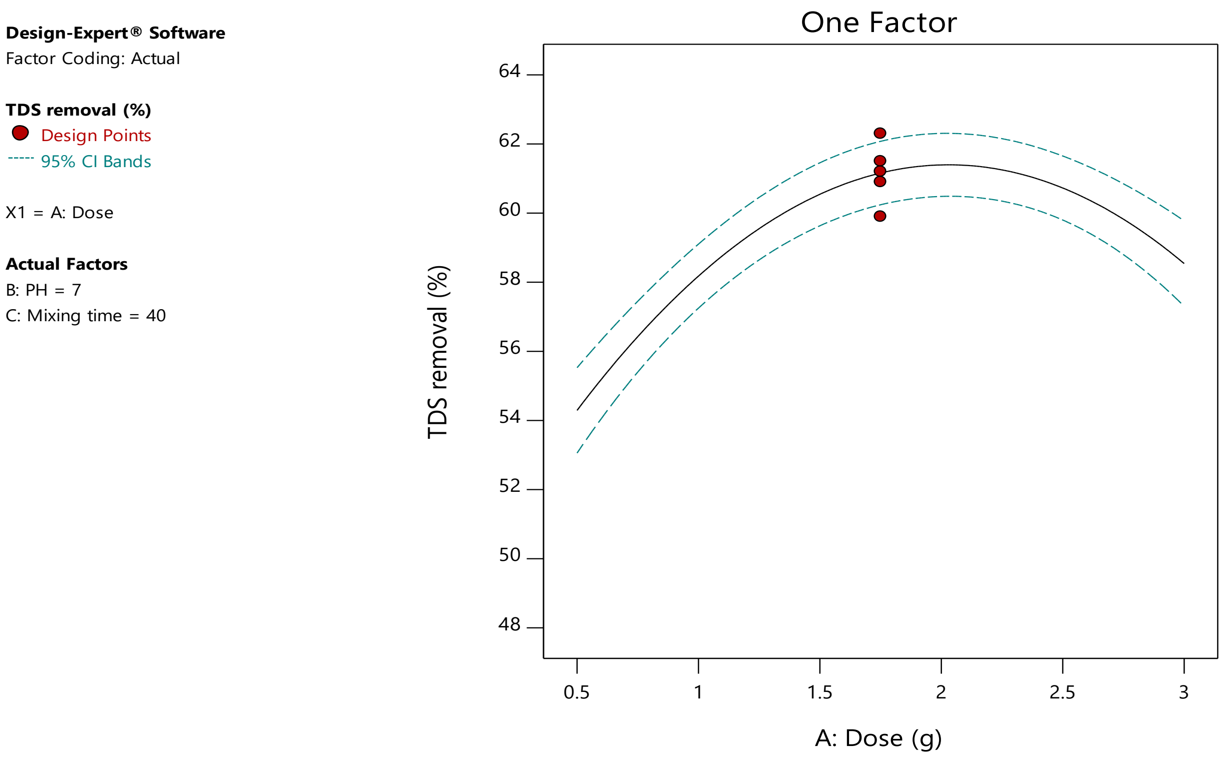

Figure 4. 19: Effect of dose on TDS removal

4.3.4.2. Effect of pH on percentage removal of TDS

The effect of pH was analyzed by varying pH from 3-11 and constant mixing time as well as dose which is 40 minute, 1.75g respectively, which consisting of rapid mixing 100 rpm for 2 minutes and slow mixing 40 rpm and 60 minute setting time and the coagulation experiment clearly affirmed that the percentage removal to be good for TDS at acidic condition than basic particularly at pH 3. The minimum removal efficiency is at pH of 11, which is around 57% and maximum removal was observed at pH of 3 which is around 62%. At some point particles surface charge were reduced or neutralized as well as destabilized the suspension. The removal capacity has gradually declined as pH increase beyond 3 and as pH approaches basic condition it is proved to decrease the solubility of blended coagulant. Because, as pH increased beyond the optimum positive charges of cactus surface decreased significantly. Consequently, the contribution charge neutralization roles of the coagulant to destabilize the particles become less. Moreover, at pH 3 the result showed that flocs produced by blended coagulant were rapid and caused large size of flocs formation which was important for easiness of the settlement. Therefore, the blended coagulant is effective for the treatment of textile wastewater in acid condition.

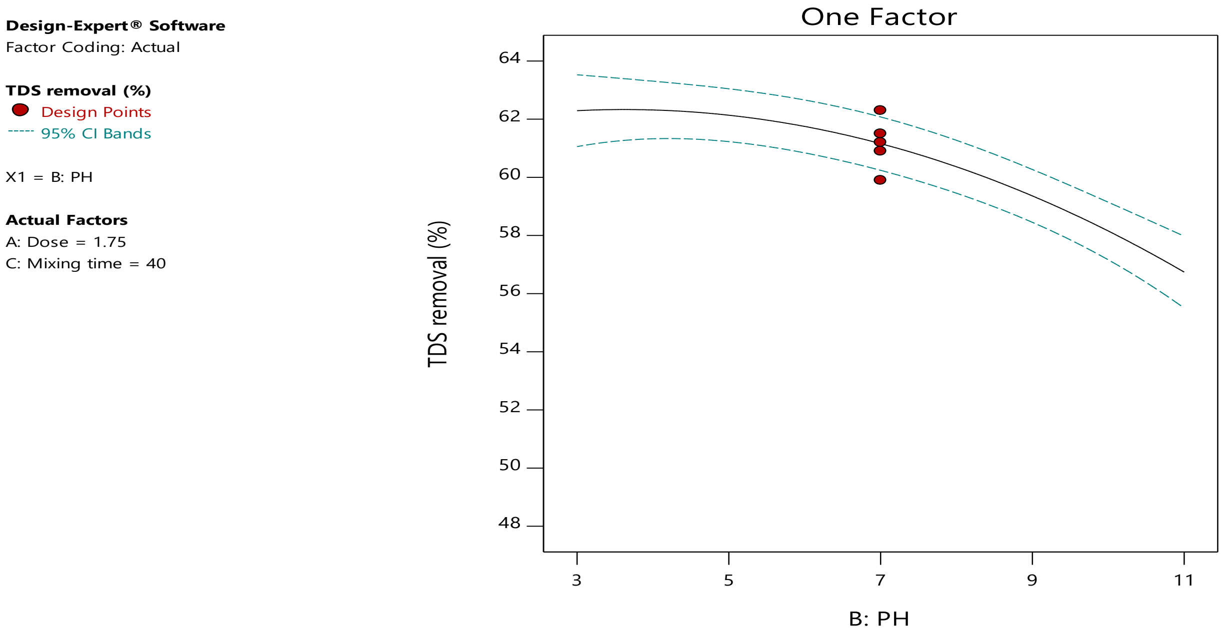

Figure 4. 20: Effect of pH on TDS removal

4.3.4.3. Effect of mixing time on percentage removal of TDS

Figure 4.21 indicates as the effect of mixing time on removal of TDS was tested by varying 20-60 minutes mixing time and constant pH of 7 as well as coagulant dose which is 1.75g and consists of rapid mixing 100 rpm for 2 minute and slow mixing speed 40 rpm with 60 minutes of settling time. The experimental result shows that there was a continuous removal of TDS, while increasing the mixing time from 20 to 40 minutes. When the mixing time is short (<40 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 (>40 min) it would lead to an increase in flocs breakage & limit the size of the floc formed. It is a great importance to optimize the mixing time so as to increase removal performance of the coagulation.

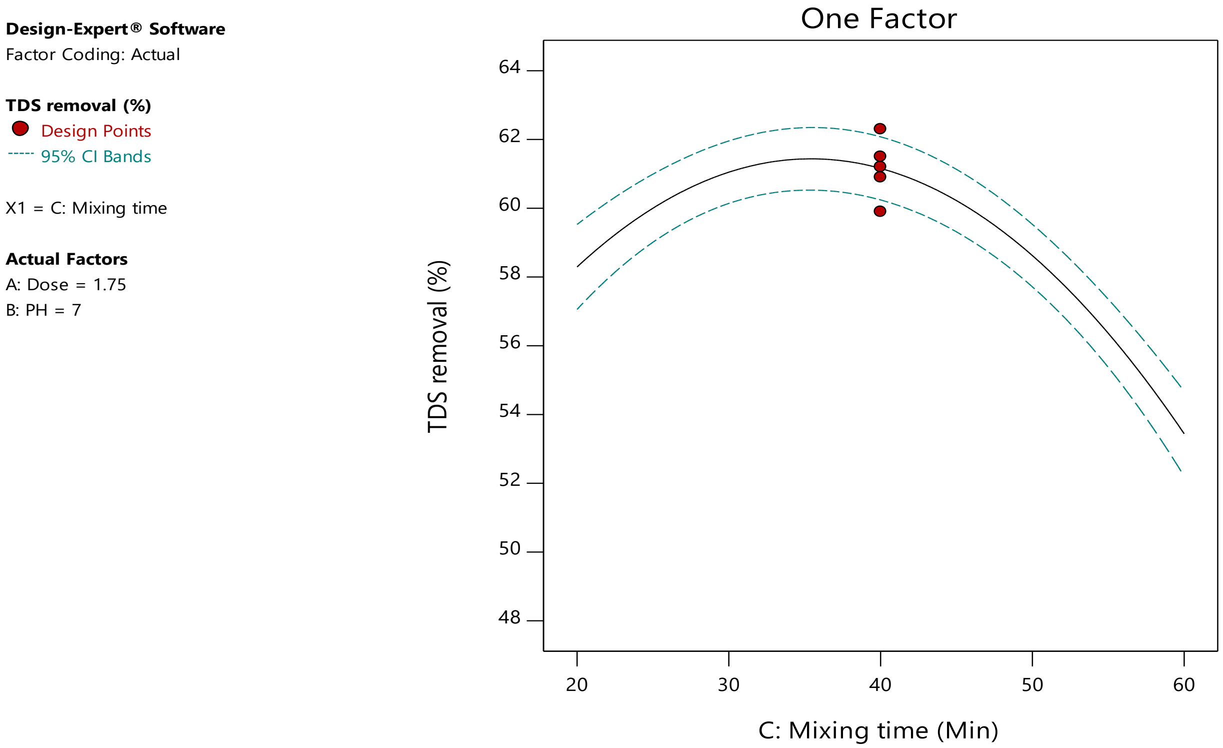

Figure 4. 21: Effect of mixing time on TDS removal

4.3.4.4. Interaction effects on percentage removal of TDS

The possible interactions that can affect the percentage removal of TDS were pH and coagulant dose. Figures 4.22 and Tables 4.10 from analysis of variance, demonstrate the significant interaction effects that exist between the processing variables. As observed from the Figures and Tables, the only significant interaction that affects percentage removal of TDS is between mixing time and coagulant dose. According to the study, interaction between mixing time and coagulant dose, have increased the percentage removal of the TDS.

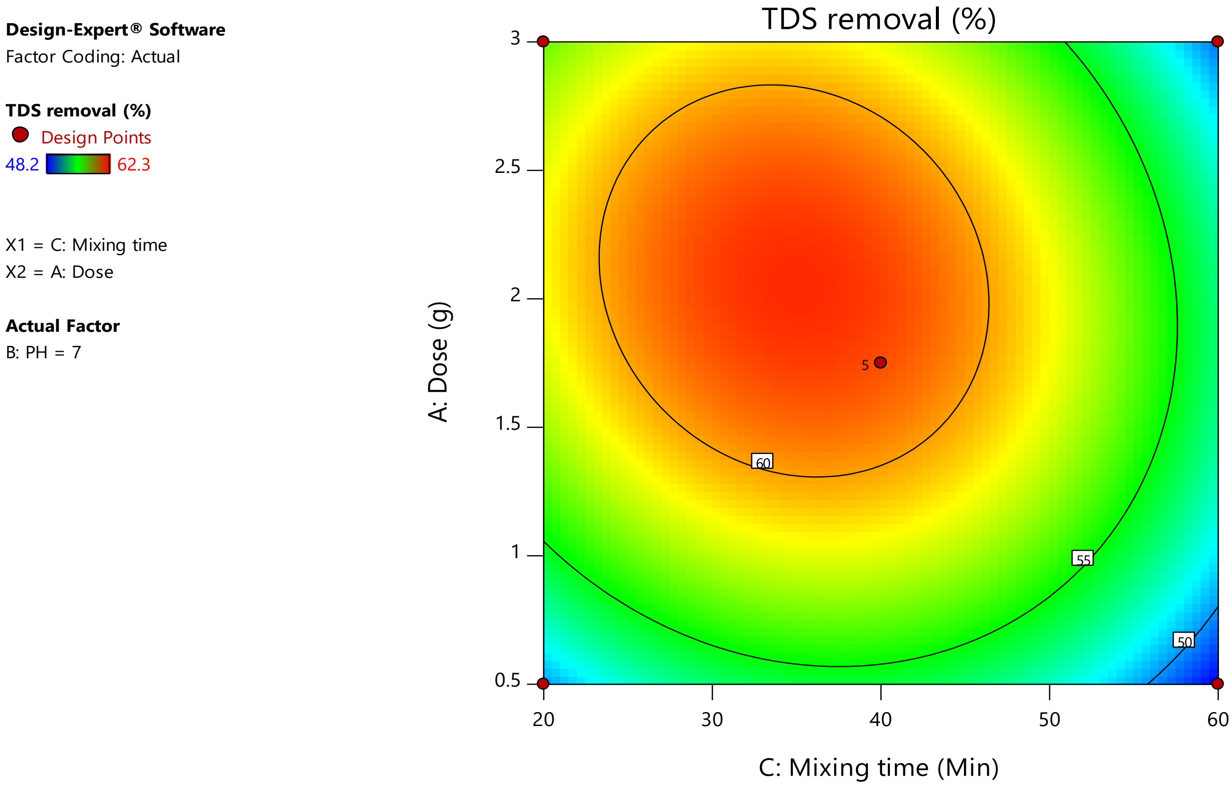

Figure 4. 22: Contour plot of the interaction effect of dose and mixing time on TDS removal

4.4. Optimization

Process optimization is the discipline of adjusting a process so as to optimize (make the best or most effective use of) some specified set of parameters without violating some constraint. The most common goals are minimizing cost and maximizing throughput and/or efficiency. Depending on the parameters by compromising percentage removal, economy and energy carrying, the best solution was selected and summarized as follows:

Table 4. 12 : Goal of the optimization and its range

Parameters	Purpose(goal)	Unit	Minimum	Maximum
Coagulant dose	In range	G	0.5	3
pH	In range	-	3	11
Mixing time	In range	Min	20	60
% Color removal	Maximized	%	60.2	74.3
% COD removal	Maximized	%	53.4	74.5
% TSS removal	Maximized	%	57.2	73.9
% TDS removal	Maximized	%	48.2	62.3

Using design expert version@11.1.01 two optimum Solutions was found for all responses

Table 4. 13 : Optimum points with its desirability for the combined responses

Number	Dose g	pH	Mixing time	Color removal	COD removal	TSS removal	TDS removal	Desirability	
1	2.194	3.840	38.992	74.300	71.177	73.094	62.930	0.946	Selected
2	2.181	3.916	39.212	74.301	71.094	73.164	62.890	0.946	

Desirability range from zero to one for any given response, the program combines individual desirability into a single number and then searches for the greatest overall desirability. A value of one represents the ideal case. A zero indicates that one or more responses fall outside desirable limits. In this numerical optimization is 0.946 which is nearer to the ideal case &indicates the responses falls in the desirable limit.

Based on the optimum the desirability plots as shown below figure

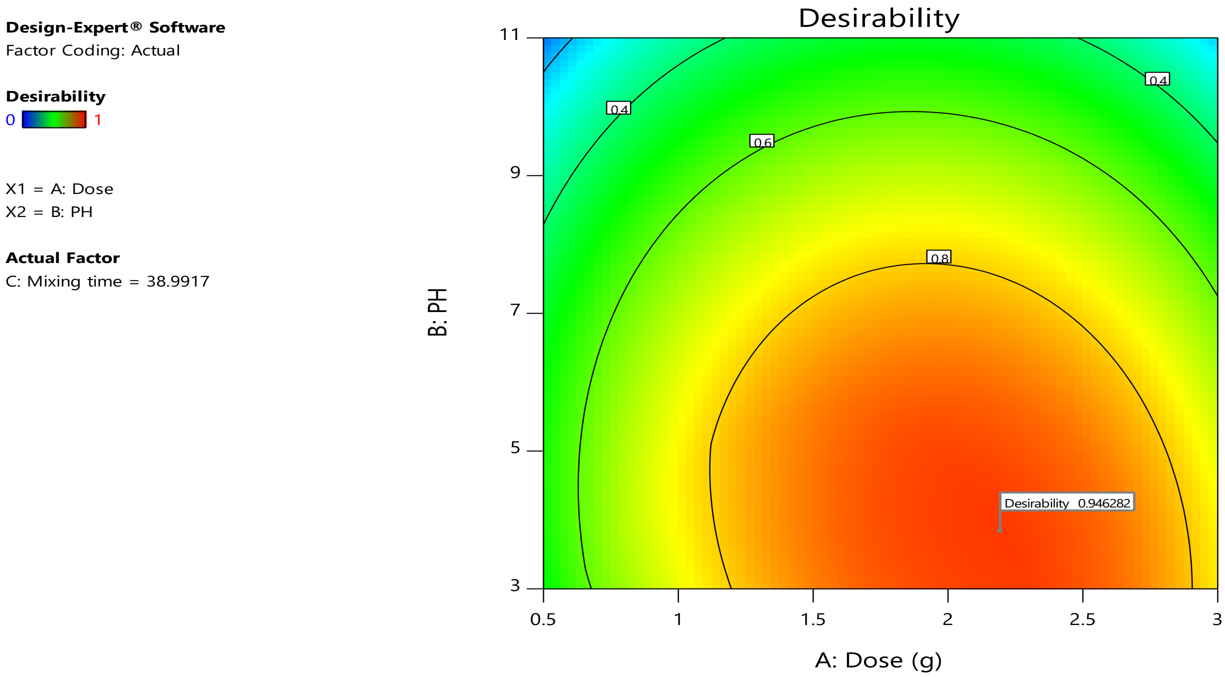

Figure 4. 23: Desirability of the interaction effect based on the optimum solution

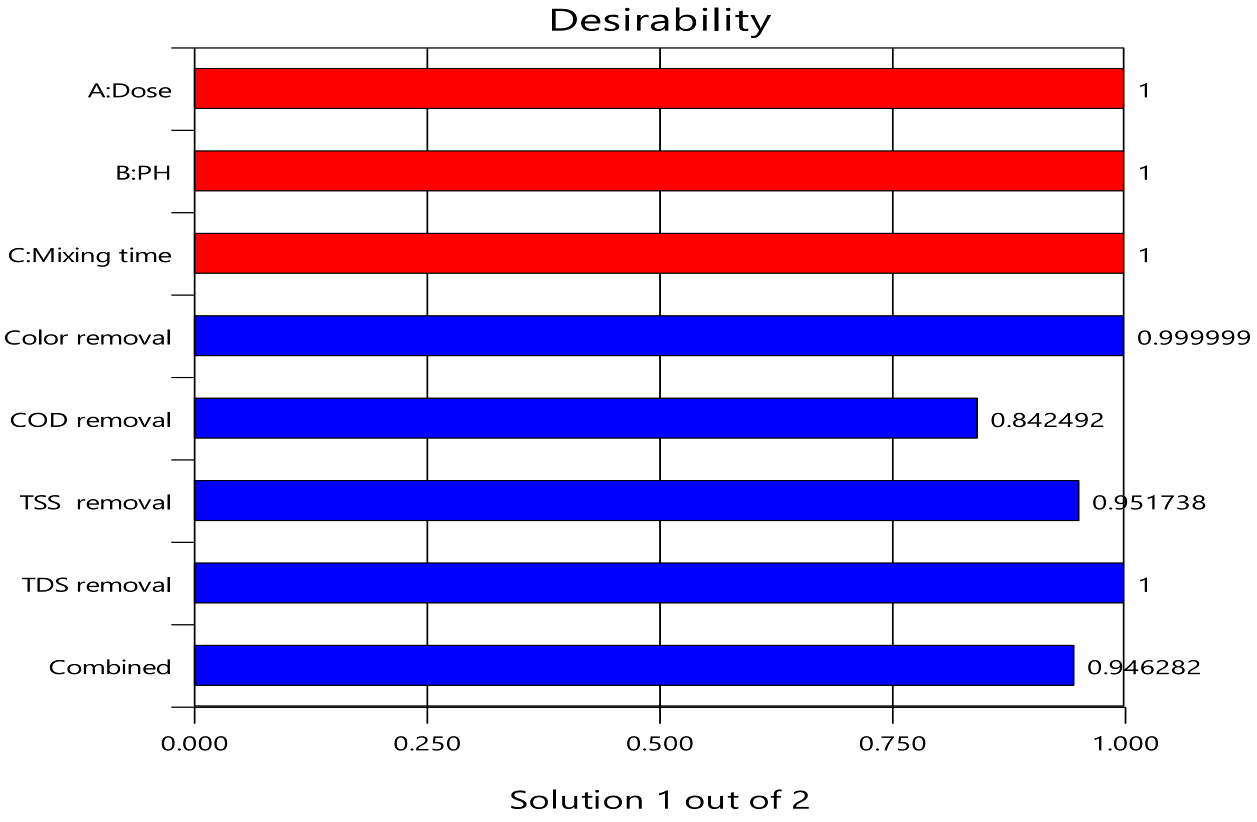

Figure 4. 24: Bar graph that shows Desirability on response based on the optimum solution

4.4.1. Validation of the model

In order to verify this prediction, the experiment was conducted and the results were computable with prediction, as a result, the model was considered to be accurate and reliable for predicting the percentage removal of textile waste water using combined cactus and alum as coagulant. Based on the second-order models, numerical optimization was carried out to maximize the removal efficiency using the response optimizer in Design expert®11.1.0.1 The optimal values of test variables were calculated as 2.19g dose , 3.84 pH and 38.99 minutes mixing time . The analyses show that the average removal efficiency of color, COD, TSS and TDS was 74.3%, 71.177%, 73.094% and 62.930% respectively with its desirability 0.946.

5. Conclusion and Recommendations

5.1. Conclusion

A combined coagulant (alum and cactus mucilage) was used for the treatment of textile waste water in order to remove color, COD, TDS, and TSS. This study concluded that the treatment of textile effluent before disposal is important to ensure the safety of our environment. 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 five specific objectives of the study. The initial character of the textile wastewater sample showed pH -10.2, color – 2300 Pt-Co, COD – 1170 mg/l, TS – 1675 mg/l, TDS – 1130 mg/l, TSS – 545 mg/l, and Turbidity is 160 NTU. This shows that the textile waste water was highly polluted, high suspended solid, highly colored and alkaline in pH.

The coagulation experiment proved coagulant dose, pH, and mixing time for being important operating parameters for the removal of color, COD, TDS and TSS from textile wastewater using blended of alum and cactus as a coagulant and their optimum conditions were at 2.194g of coagulant dose, pH 3.840, & 38.992 minutes of mixing time. At these optimal conditions the removal efficiency of color, COD, TSS, and TDS was 74.3 %, 71.177 %, 73.094%, and 62.930% respectively. The FTIR study also confirmed the presence of various functional groups which are responsible for the coagulation process.

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

Finally from this study we can concluded that, removal efficiency of the blended coagulant of alum and cactus mucilage was very good in the treatment of textile waste water for color, COD, total dissolved solid, and suspended solid..

5.2. Recommendations

- ✓ It is better to study the removal efficiency of the blended coagulant on textile waste water by varying the other operational variables like blending ratio, settling time, particle size and temperature in detail to improve the removal efficiency of the system.
- ✓ It is also better to study the environmental impact assessment of the textile waste water on the workers, inland water, animals, plants and soil fertility found in the textile industry in special and the society near by the textile industry in diverse.
- ✓ It is recommended to study the other responses like turbidity, BOD, heavy metals, pH, temperature etc.
- ✓ Future research should be done using this technology in combinational with other cost effective treatment methods for further treatment to improve the removal efficiency of the system.
- ✓ In Ethiopia textile industry is among the major manufacturing industries, therefore the government should give thoughtful attention so as to minimize disposal of untreated wastewater containing dye effluents as well as other synthetic chemicals to the environment.
- ✓ It is essential to create awareness to farmers to plant and engage on cactus pad development all over the countries and creating market to local people to sell the plant.

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Appendices

Appendix A: Textile Wastewater Disposal Standards (Environmental Protection Authority)

THE MANUFACTURE AND FINISHING OF TEXTILES

Limit Values for Discharges to Water

Parameter	**Limit Values**
Temperature	40 °C
pH	6 – 9
BOD_5 at 20^0C	50 mg/l
Total nitrogen (as N)	40 mg/l
COD (mg O_2 /l)	150 mg/l
Total phosphorus (as P)	10 mg/l
Suspended solids	30 mg/l
Total ammonia (as N)	20 mg/l
Oils, fats & grease	20 mg/l
Phenols	1 mg/l
Mercury (as Hg)	0.001 mg/l
Nickel (as Ni)	2 mg/l
Cobalt (as Co)	1 mg/l
Lead (as Pb)	0.5 mg/l
Antimony (as Sb)	2 mg/l
Tin (as Sn)	5 mg/l
Chromium (as Cr VI)	0.1 mg/l
Chromium (as total Cr)	1 mg/l
Arsenic (as As)	0.25 mg/l
Cadmium (as Cd)	1 mg/l
Zinc (as Zn)	5 mg/l
Copper (as Cu)	2 mg/l
Mineral oils (Interceptors)	20 mg/l

Benzene, toluene & xylene (combined)	1 mg/l
Mineral oils (Biological Treatment)	5 mg/l
Organochlorine pesticides (as Cl)	0.03 mg/l
Mothproofing agents (as Cl)	0.003 mg/l
Organophosphorus pesticides (as P)	0.003 mg/l
Adsorbable organic halogen compounds (AOX)	5 mg/l
Sulphide (as S)	2 mg/l

Limit Values for Emissions to Air

Parameter	**Limit value (mg/Nm^3)**
Particulate matter	50
Volatile organic carbons (as C) (excluding formaldehyde)	50
Formaldehyde	20
Isocyanates (as NCO)	0.1

Appendix B: Photographic Representation of laboratory work

A. Photographic representation of before and after treatment of textile wastewater

B. Electronic balance

C. Cactus mucilage before drying

D. pH meter (model: JENWAY; 3505 pH meter)

E. Digital Thermometer tp101 stainless F. HACH turbid meter (Model 2100N)

G. Spectrophotometer (HACH model) H. oven for TS, TSS and TDS analysis

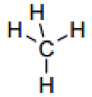
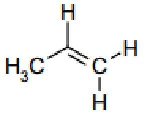
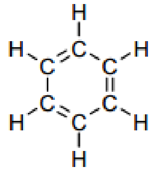
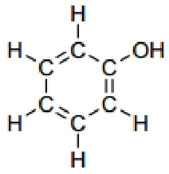
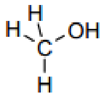
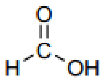
I. HANNA

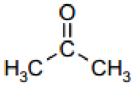
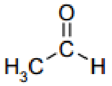
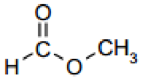
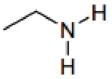
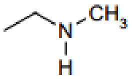
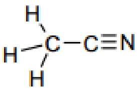
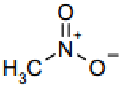
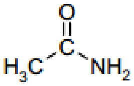
COD reactor

J. Spectro uv-vis Double beam Pc (UVD 3200 model) for color measurement

K. Sieve analyzer for particle size of cactus mucilage

Appendix C: Principal IR Absorptions for Certain Functional Groups

Functional Group Names **&** **Example compounds**	**Absorption Ranges(cm^{-1})** [Look for a single absorption in these regions, unless stated otherwise.]	**Type of Vibration** **causing IR absorption**
Alkanes:  Methane	**3000-2800** (Note: The absorptions can be seen as several distinct peaks in this region.)	H-C-H Asymmetric & Symmetric Stretch
	1500-1440	H-C-H Bend
Alkenes:  1-Propene	**3100-3000**	C=C-H Asymmetric Stretch
	1675-1600	C-C=C Symmetric Stretch
Alkynes: $HC \equiv C-CH_3$ Propyne	**3300-3200**	$\equiv C-H$ Stretch
	2200-2100	$C \equiv C$ Stretch
Aromatic Rings:  Benzene	**3100-3000**	C=C-H Asymmetric Stretch
	1600-1580	C-C=C Symmetric Stretch
	1500-1450	C-C=C Asymmetric Stretch
Phenols & Alcohols:  Phenol  Methanol (Alcohol)	**3600-3100** (Note: Phenols MUST have Aromatic Ring Absorptions too.)	Hydrogen-bonded O-H Stretch (This peak usually appears much broader than the other IR absorptions.
Carboxylic Acids:  Formic Acid	**3400-2400** *(This peak always covers the entire region with a* ***VERY BROAD*** *peak.)*	Hydrogen-bonded O-H Stretch *[Note: This peak can obscure other peaks in this region.]*
	1730-1650	C=O Stretch

Ketones:  Acetone	**1750-1625**	C=O Stretch
Aldehydes:  Ethanal	**1750-1625**	C=O Stretch
	2850-2800	C-H Stretch off C=O
	2750-2700	C-H Stretch off C=O
Esters:  Methyl Formate	**1755-1650**	C=O Stretch
	(1300-1000)	(C-O Stretch)
Ethers:  Diethyl Ether (aka-Ethyl Ether)	**(1300-1000)**	(C-O Stretch)
Amines—Primary:  Ethylamine	**3500-3100** **(TWO PEAKS!)**	N-H Stretch
	1640-1560	N-H Bend
Amines—Secondary:  N-Methylethylamine	**3500-3100** **(ONE PEAK!)**	N-H Stretch
	1550-1450	N-H Bend
Nitriles:  Methanenitrile	**2300-2200**	C≡N Stretch
Nitro Groups:  Nitromethane	**1600-1500**	N=O Stretch
(Note: Both peaks are <200 cm-1 apart.)	**1400-1300**	N=O Bend
Amides:  Methanamide	**3500-3100**	N-H Stretch (similar to amines)
	1670-1600	C=O Stretch
	1640-1550	N-H Bend

(Olivares-pérez, et.al, 2012)

Appendix D : laboratory expermiental result

i. Absorbance measurement of textile wastewater before and after treatment for color measurement

$$\text{Color removal (\%)} = \frac{(A)o-(A)t}{(A)o} * 100\%$$

Where, $(A)_0$ = absorbance value before treatment, $(A)_t$ = absorbance after treatment

Absorbance measurement of textile wastewater before and after treatment at wavelength 660nm and average absorbance of untreated textile wastewater was 1.309

Std	Run	Factor 1	Factor 2	Factor 3	Absorbance before treatment	Absorbance after treatment	Response 1
		A:Dose g	B:pH	C:Mixing time Min	Control	Abs. value	Color removal %
14	1	1.75	7	40	1.309	0.36	72.5
3	2	0.5	11	40	1.309	0.482	63.2
13	3	1.75	7	40	1.309	0.3744	71.4
17	4	1.75	7	40	1.309	0.336	74.3
16	5	1.75	7	40	1.309	0.35	73.4
11	6	1.75	3	60	1.309	0.39	70.4
15	7	1.75	7	40	1.309	0.36	72.7
9	8	1.75	3	20	1.309	0.365	72.1
2	9	3	3	40	1.309	0.419	68
1	10	0.5	3	40	1.309	0.343	73.8
5	11	0.5	7	20	1.309	0.490	62.5
4	12	3	11	40	1.309	0.490	62.5
7	13	0.5	7	60	1.309	0.47124	64
10	14	1.75	11	20	1.309	0.5053	61.4
8	15	3	7	60	1.309	0.521	60.2
6	16	3	7	20	1.309	0.4922	62.4
12	17	1.75	11	60	1.309	0.4752	63.7

ii. Expermental result of treated waste water interms of COD, TSS and TDS

Std	Run	Factor 1	Factor 2	Factor 3	COD	TSS	TDS
		A:Dose g	B:pH	C:Mixing time Min	mg/l	mg/l	mg/l
14	1	1.75	7	40	407.16	144.42	441.83
3	2	0.5	11	40	489.06	230.54	555.96
13	3	1.75	7	40	395.46	151.51	453.13
17	4	1.75	7	40	383.76	142.24	435.05
16	5	1.75	7	40	389.61	146.06	438.44
11	6	1.75	3	60	394.26	196.75	528.84
15	7	1.75	7	40	375.57	147.7	426.01
9	8	1.75	3	20	409.5	177.67	446.35
2	9	3	3	40	298.35	176.58	442.96
1	10	0.5	3	40	424.71	209.28	513.02
5	11	0.5	7	20	464.49	229.44	568.39
4	12	3	11	40	476.19	222.36	532.23
7	13	0.5	7	60	505.44	233.26	585.34
10	14	1.75	11	20	503.1	218.55	522.06
8	15	3	7	60	398.97	225.63	562.74
6	16	3	7	20	416.52	209.28	470.12
12	17	1.75	11	60	545.22	230.53	571.78