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ADDIS ABABA INSTITUTE OF TECHNOLOGY

SCHOOL OF CHEMICAL AND BIO ENGINEERING

EVALUATION OF SUGARCANE BAGASSE AS BIO ADSORBENT IN TEXTILE WASTEWATER TREATMENT

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DECLARATION

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ABSTRACT

This thesis was conducted for evaluation of sugarcane bagasse as bio adsorbent for removing of textile wastewater colour. The sugarcane bagasse was soaked in hydrochloric acid solution for twenty four hours and finally the sugarcane bagasse was dried in oven to remove moisture. The sugarcane bagasse was characterized according ASTM method for moisture content and ash content as well as according ISO method for volatile matter. The experimental result obtained were 29% for moisture content, 4.2% for ash content and 83% for volatile matter. FTIR analysis of sugarcane bagasse was carried out and the experimental result indicates that the presence of different functional groups on the surface of sugarcane bagasse before and after adsorption. In addition to this XRD analysis of sugarcane bagasse was conducted. The higher peak of XRD analysis indicates crystalline cellulose region and the lower peak indicates amorphous structure. The raw textile wastewater was characterized according APHA method for BOD, COD and TDS before and after adsorption as well as pH before adsorption and the results were for BOD it ranges from 48 mg/L – 163 mg/L before adsorption and from 20 mg/L – 83 mg/L after adsorption and for COD 925 mg/L before adsorption and 50 mg/L after adsorption as well as TDS 10,000 mg/L before adsorption and 8400 mg/L after adsorption. Eventually the pH value before adsorption was 9.65. Adsorption was conducted using synthetic azo dye and textile wastewater at the factors of concentration, Adsorbent, pH and contact time and the higher colour removal was achieved at optimum value of concentration 52.5 mg/L, dose of adsorbent 2.5-gram, pH 6.5 and time 75 minute for both adsorption. Based on ANOVA analysis on adsorption of azo dye and textile wastewater on to bagasse, factors of concentration, pH, and time were significant. Kinetic study was carried out for adsorption of azo dye and textile wastewater and pseudo first order model was well fitted for both adsorptions. In addition to this adsorption isotherm was conducted for azo dye and textile wastewater adsorption and the Langmuir isotherm model was well fitted for both adsorptions. To conclude the comparative analysis of azo dye and textile wastewater adsorption using sugarcane bagasse as bio-adsorbent and the result provide that, there was a higher percentage of colour removal with azo dye adsorption when compared with adsorption of textile wastewater and the results were 90% for azo dye colour removal and 81% for textile wastewater colour removal.

Key words; Sugarcane bagasse, azo dye, adsorption process, textile wastewater removal

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TABLE OF CONTENTS

List of Tables

LIST OF ACRONYMS AND ABBREVATIONS

ANOVA = Analysis of variance
APHA = American public health association
ASTM = American society for testing material
BOD = Biological oxygen demand
CCD = Central composite design
COD = Chemical oxygen demand
C.V = Coefficient of variation
DF = Degree of freedom
DP = Degree of polymerization
FC = Fixed carbon content
FTIR = Fourier transform infrared spectrometry
ISO = International organization for standardization
NM = Nanometer
PFO = Pseudo first order
PSO = Pseudo second order

RPM = Revolution per minute

SCB = Sugarcane bagasse

SEM = Scanning electron microscopy

TDS = Total dissolved solids

VC = Volatile matter content

W = Weight

XRD = X-ray diffraction

List of Figures

1. INTRODUCTION

1.1 Background

This study was conducted in order to address the problem found in the treatment systems of textile industries. Textile industries used a huge amount of water and this water produce a huge amount of wastewater specially the dying department produce a large amount of wastewater and this dye based textile wastewater has a potential of hazardous for environment and health. Textile industries are suggested to utilize bio-mass based adsorbent to treat their effluent and in this case sugarcane bagasse was taken to evaluate its capability as bio adsorbent for textile wastewater treatment.

Colored compounds are the most easily recognizable pollutants in the environment because of their appearance *(Liew Abdullah et al., 2005*). Most of the industries such as textile, paper, carpet, and printing use dyes and pigments to color their products. Due to their good solubility, synthetic dyes are common water pollutants and they may frequently be found in trace quantities in industrial wastewater. However, the discharge of dye-bearing wastewater into natural streams and rivers possess a severe problem, as dyes impart toxicity to aquatic life and are damaging the aesthetic nature of the environment. However, wastewater containing dyes is very difficult to treat, since the dyes are recalcitrant organic molecules, resistant to aerobic digestion, and are stable to light, heat and oxidizing agents due to their structure and molecular size. Amongst the numerous techniques of dye removal, adsorption is the procedure of choice and gives the best results as it can be used to remove different types of colouring materials.

The adsorption through activated carbon is a very useful technique; however, the major constraints are high production cost and energy consumption. Therefore, the forest and agricultural waste materials can be an alternative choice for dye removal from wastewater because of their good performance and cost effectiveness. In this regard, sugarcane bagasse was investigated for azo dye and dye based textile wastewater removal in a batch experiment. The maximum dye removal was tested as a function of contact time, adsorbent dosage, pH and initial concentration of azo dye and dye based textile wastewater.

1.2 Statement of the Problem

Evaluation of sugarcane bagasse as bio-adsorbent in textile wastewater treatment aims to provide a sustainable, cost effective and easy way of treatment for textile wastewater with bio degradable and environmentally friend bio mass of sugarcane bagasse. Once if sugarcane bagasse is implemented as bio adsorbent textile wastewater treatment, there is a beneficiation for the industry stake holder and nearby communities. Since sugarcane bagasse is cheap and easily available when compared with other chemicals and other advanced technologies textile industries are not exposed for unnecessary expense. In addition to this nearby communities to the textile industries are well safe from environmental and health risk. However, discharging of properly untreated textile wastewater has been a serious problem facing our country for the last few years since new industrialization started. Most of the methods used for textile wastewater treatment found in Ethiopia are inefficient and less competitive in cost. Now a day textile industries found in Ethiopia invest a certain amount of capital to treat the effluent through utilization of chemicals and other advanced technologies. Properly untreated textile wastewater has environmental and health effect. There are a number of environmental effect of properly untreated textile wastewater specially dye based waste that damage flora and fauna of ecosystem and ground water pollution. As the waste water discharged in to the water bodies, it produces major issues to the society and responsible for the water born disease.

The current treatment of textile wastewater in Ethiopia is not effective and there is sludge formation and the various chemicals used in the treatment plant are expensive and are not environmentally friend. The solution to the problem of inefficient textile wastewater treatment is utilization of sugarcane bagasse I have selected for bio-adsorbent for textile wastewater treatment and this method abstain formation of sludge and it is simple for technical operation and cost feasible as well as the bio adsorbent is biologically bio degradable and environmentally friend. To sum up the current treatment method of textile wastewater is not environmentally friend and not cost feasible and using sugarcane bagasse as bio adsorbent is better and feasible.

1.3 Organization of Thesis

The thesis content consists of the following chapters:
Chapter 1 consist the introduction of thesis research and the objective as well as scope of the study.

Chapter 2 explains the literature about sugarcane bagasse and its importance as well as adsorption mechanism with adsorption principles. In addition to this composition of sugarcane bagasse and the overall production process of textile industry and its effluent consequence is stated in this chapter.

Chapter 3 and it represent about the methodology used for the experments.It explains about the methodology employed to conduct the experiment. It also describes the equipment and apparatus used for the experiment.

Chapter 4 represent result and discussion that were obtained during the expermentations.This chapter focus on obtaining optimal conditions that lead to the best conditions for the best color removal by adsorption. It also explains the best model for adsorption like Langmuir, Freundlich.

Chapter 5 represents the conclusions to the study. It gives conclusions and recommendations.

1.4 Objective

1.4.1 General Objective

- To evaluate Sugarcane bagasse as bio adsorbent for textile wastewater treatment.

1.4.2 Specific Objectives

The specific objectives of the study include:

- Characterization of sugarcane bagasse.
- Characterization of wastewater before and after adsorption and these physico-chemical properties have a great contribution for coloring of textile wastewater.
- To identify and investigate functional groups of adsorbent before and after adsorbtion.
- Analysing the crystalline structure of cellulosic sugarcane bagasse so that to find the arranged and ordered microscopic structure of sugarcane bagasse.

- To investigate the optimum operating conditions of sugarcane bagasse as bio adsorbent with initial concentration, pH, dose of adsorbent and contact time.
- To investigate the effect of contact time, pH,amount of adsorbent as well as initial concentration on the efficiency of sugar cane bagasse as bio adsorbent.

1.5 Significance of the Research

The significance of this study can be seen from two different perspectives. The first one is since sugar cane bagasse is cheap when compared with activated carbon and another adsorbent in Ethiopia; it supports the textile sector of the economy. The second significance of this study is beneficiation of sugarcane bagasse as bioadsorbent can be compatible with the environmental perspectives. Utilization of sugar cane bagasse abstain unnecessary expense as well as it is environmentally friendly. Hence, it can be benefit` textile industry and nearby communities.

1. 6 Scope of Study

Properly untreated textile wastewater has a great problem on the environment and health of organisms. The general objective of the study was to evaluate sugarcane bagasse as bio adsorbent in textile wastewater treatment. The specific objectives were proximate analysis of sugarcane bagasse, analysing of functional group before and after adsorption and to investigate the effect of selected factor. This study was required to address the consequence of textile wastewater. The raw wastewater sample was collected from Yrgalem Addis textile industry. Pre-treatment with HCl was conducted so that hemicelluloses part of bagasse solubilise and exposing of cellulose surface for adsorption. In addition to this a chemical modification using HCl solution was used to eliminate lignin and hemicelluloses on fiber surface so that a significant change in morphological structure of bagasse to occur that lead creation of fibrillated surface that is increasing of surface areas of the fiber, and then adsorption of collected wastewater sample on to bagasse was carried out. Batch studies was conducted using initial concentration, dose of adsorbent, contact time and pH as a major factor to evaluate sugarcane bagasse as bio-adsorbent. The experimental data was analysed statically using ANOVA analysis.

2. LITERATURE REVIEW

2.1 Introduction

Most of the time color pollutants are recognizable pollutants in the environment owing their appearance. Several industries such as textile, paper, and printing use dyes and pigments to color their products. Due to their good solubility, synthetic dyes are common water pollutants and they may frequently be found in trace quantities in industrial wastewater. However, the discharge of dye-bearing wastewater into natural streams and rivers possess a severe problem, as dyes impart toxicity to aquatic life and are damaging the aesthetic nature of the environment. However, wastewater containing dyes is very difficult to treat, since the dyes are recalcitrant organic molecules, resistant to aerobic digestion, and are stable to light, heat and oxidizing agents due to their structure and molecular size. Amongst the numerous techniques of dye removal, adsorption is the procedure of choice and gives the best results as it can be used to remove different types of colouring materials.

Sugarcane bagasse is one type of adsorbent and it has excellent adsorption efficiency for color and other organic compounds. In order to decrease the cost of treatment, attempts have been made to find inexpensive alternative adsorbents. Consequently, a number of low cost and easily available materials, such as waste biomass, are being studied for the removal of different dyes from aqueous solutions at different operating conditions.

Forest and agricultural production by-products have been long considered as potential dye adsorbents. Unfortunately, without prior chemical modification these materials uniformly have very low adsorption capacities for acidic dyes.

The aim of the present study was to determine the optimum operating conditions for treating of textile wastewater specially color component of textile wastewater and synthetic azo dye solution by adsorption technique using hydrochloric acid treated sugarcane bagasse and sodium hydro-oxide treated sugarcane bagasse. Azo dye formula is $C_{15}H_{15}N_3O_2$. It is an azo dye, and is a dark red crystalline powder with melting point between 179-182 °C. It has 95% dye content, and its transition range is pH 4.2 – pH 6.2 (red-yellow).

2.2 Adsorption Mechanism

Activated carbon can be considered as a material of phenomenal surface area made up of millions of pores - rather like a "molecular sponge"(*Ismaeel, et al 2010*). The process by which such a surface concentrates fluid molecules by chemical and/or physical forces is known as Adsorption. A process whereby fluid molecules are taken up by a liquid or solid and distributed throughout that liquid or solid(*"Kinetics and Mechanisms of Adsorption of," 1979)*. In the physical adsorption process, molecules are held by the carbon's surface by weak forces known as Van Der Waals Forces resulting from intermolecular attraction. The carbon and the adsorbate are thus unchanged chemically. However, in the process known as Chemisorptions, molecules chemically react with the carbon's surface (or an impregnate on the carbon's surface) and are held by much stronger forces - chemical bonds. In general terms, to affect adsorption it is necessary to present the molecule to be adsorbed to a pore of comparable size (*Bérend et al., 1995*). In this way the attractive forces coupled with opposite wall effect will be at a maximum and should be greater than the energy of the molecule. For example, a fine pored coconut shell carbon has poor decolorizing properties because color molecules tend to be larger molecular species and are thus denied access to a fine pore structure.

In contrast, coconut shell carbons are particularly efficient in adsorbing small molecular species. Krypton and Xenon, for instance, are readily absorbed by coconut shell carbon but readily adsorbed from large pored carbons such as wood. Maximum adsorption capacity is determined by the degree of liquid packing that can occur in the pores. In very high vapour pressures, multilayer adsorption can lead to capillary condensation even in Mesopores. If adsorption capacity is plotted against pressure (for gases) or concentration (for liquids) at constant temperature, the curve so produced is known as an Isotherm. Adsorption increases with increased pressure and also with increasing molecular weight, within a series of a chemical family. Thus, methane (CH_4) is less easily adsorbed than propane (C_3H_8). This is a useful fact to remember when a particular system has a number of components. After equilibrium, it is generally found that, all else being equal; the higher molecular weight species of a multi-component system are preferentially adsorbed. Such a phenomenon is known as competitive or preferential adsorption - the initially adsorbed low molecular weight species desorbing from the surface and being replaced by higher molecular weight species.

Physical adsorption in the vapour phase is affected by certain external parameters such as temperature and pressure. The adsorption process is more efficient at lower temperatures and higher pressures since molecular species are less mobile under such conditions. Such an effect is also noticed in a system where moisture and an organic species are present. The moisture is readily accepted by the carbon surface but in time desorbs as the preferred organic molecules are selected by the surface. This usually occurs due to differences in molecular size but can also be attributable to the difference in molecular charge.

Generally speaking, carbon surfaces dislike any form of charge - since water is highly charged (ionic) relative to the majority of organic molecules the carbon would prefer the organic to be adsorbed. Primary amines possess less charge on the nitrogen atom than secondary amines that in turn have less than tertiary amines. Thus, it is found that primary amines are more readily adsorbed than tertiary amines. High levels of adsorption can be expected if the adsorbate is a reasonably large bulky molecule with no charge, whereas a small molecule with high charge would not be expected to be easily adsorbed.

Molecular shape also influences adsorption but this is usually of minor consideration. In certain situations, regardless of how the operating conditions can be varied, some species will only be physically adsorbed to a low level. (Examples are ammonia, sulphur dioxide, hydrogen sulphide, and mercury vapour and methyl iodide). In such instances, the method frequently employed to enhance a carbon's capability is to impregnate it with a particular compound that is chemically reactive towards the species required to be adsorbed. Since carbon possesses such a large surface (a carbon granule the size of a "quarter" has a surface area in the order of 0.5 square miles!) coating of this essentially spreads out the impregnate over a vast area. This, therefore, greatly increases the chance of reaction since the adsorbate has a tremendous choice of reaction sites. When the adsorbate is removed in this way the effect is known as Chemisorptions.

2.2.1 Adsorption Principles

Adsorption is the phenomenon by which the molecules of a gas, vapor or liquid spontaneously concentrate at contacting surface without undergoing any reaction. The investigation was conducted to evaluate or assess cheap alternate materials of biological origin as a method of adsorbents in the textile wastewater. The utilization of an adsorbent such as sugar cane bagasse for removing species from a liquid stream depends on the equilibrium between the adsorbed and the free species. Adsorption is a process that occurs when a gas or liquid solute accumulates on the surface of a solid or a liquid, forming a molecular or atomic film(*Saad, 2016*).

In other words, adsorption is the adhesion of atoms, ions, bimolecular or molecules of gas, liquid, or dissolved solids to a surface and this process creates a film of the adsorbate (the molecules or atoms being accumulated) on the surface of the adsorbent. It is a surface phenomenon and a consequence of surface energy. The atoms on the surface of the adsorbent are not wholly surrounded by other atoms and thus, can attract adsorbates (*Rajendran, 2017*). The exact nature of the bonding depends on the details of the species involved, but the adsorption process is generally classified as follows:

(1) Physisorption: It is a type of adsorption in which the adsorbate adheres to the surface through Van der Walls (weak intermolecular) interactions.
(2) Chemisorptions: It is a type of adsorption whereby a molecule adheres to a surface through the formation of a chemical bond.

Adsorption takes place primarily on the walls of the pores or at specific sites inside the particle *(Dąbrowski, 2001*). As the pores are generally small, the internal surface area is greater than the external area. Separation occurs because differences in molecular weight, shape or polarity cause some molecules to be held more strongly on the surface than others *(McCash, 2001)*. In many cases, the adsorbate is held strongly enough to allow complete removal of that component from the fluid.

2.3 Bagasse

Bagasse is the fibrous matter that remains after sugarcane or sorghum stalks are crushed to extract their juice *(Walford, 2008*). It is dry pulpy residue left after the extraction of juice from sugarcane (*Asagekar & Joshi, 2014*). Bagasse, also called megass, fiber remaining after the extraction of the sugar-bearing juice from sugarcane. It is mainly used as a burning raw material in the sugar mill furnaces. The low caloric power of bagasse makes this a low efficiency process (*Amores et al., 2013*). Also, the sugarcane mill management encounters problems regarding regulations of clean air from the Environmental Protection Agency, due to the quality of the smoke released in the atmosphere. Ethanol is not just a good replacement for the fossil fuels, but it is also an environmentally friendly fuel. Apart from this, ethanol is a very versatile chemical raw material from which a variety of chemicals can be produced. SCB wastes are chosen as an ideal raw material in manufacturing new products because of its low fabricating costs and high quality green end material.

It is ideal due to the fact that it is easily obtainable given the extensive sugar cane cultivation making its supply constant and stable. The associated costs of extraction, chemical modifications and/or other pre-treatments of SCB in the transformation process to ready-to-be used materials are potentially reduced as the complex processes are simplified by the mere usage of Bagasse *(Rezende, et al 2011*). When appropriate modifications and manufacturing procedures are applied, bagasse displays improved mechanical properties such as tensile strength, flexural strength, flexural modulus, hardness, and impact strength. Bagasse is also found to be easily treated and modified with chemicals besides blending well with other materials to form new types of composite materials. It also satisfies the greening requirements by being biodegradable, recyclable and reusable. The compression and injection molding processes were performed in order to evaluate which is the better mixing method for fibers (sugarcane bagasse, bagasse cellulose and benzylated bagasse) and Polymer matrixes.

2.3.1 Characterization of Sugarcane Bagasse

From literature *(Omoniyi & Olorunnisola, 2014)* moisture content, volatile matter and fixed carbon content are provided as follow:

Moisture content of sugarcane bagasse is ranged within the interval of 28 -31%.The value of volatile matter is in the range of 69.4 – 81.7%, and the quantity of fixed carbon content of sugarcane bagasse resides in the interval of 12-16%.In addition to this from literature*(Ayeni, et al 2015)* the proximate analysis of sugarcane bagasse resides in the following interval and based on this extractive resides in the interval of 2.14 ±0.6,cellulose 35.28 ± 1.2,hemicelluloses 33.28 ± 0.8,lignin 25.2 ± 1.1 and ash content 4.1 ± 0.3.

2.3.2 Sugarcane Bagasse and its Importance

It is the by-product of sugarcane industries during the extraction of juice from cane. It is dry pulpy residue and fibrous in nature. It is used as a bio-fuel or in industrial level; it is used as a binding material. The application of bagasse in agricultural crop production system can be reduced the application rate of fertilizers(*Neves & Chaddad, 2012).* The use of bagasse (after combustion) is in the production of steam for power generation. Bagasse is also recognized and used for the production of bio ethanol. The important application of bagasse is found in paper making(*Chandel, et al, 2012*). In paper making industries it is utilized as the raw material. The calorific value estimation of bagasse as a fuel also describes its value, which is influenced by its composition and also on the calorific value of the sugarcane crop mainly because of the content of sucrose present. Generally it has been observed that most of the mills produce bagasse of 48 % moisture content, and most boilers burn bagasse at around 50 % moisture. Bagasse generally composed of fiber (cellulose), which contains carbon, hydrogen, oxygen, sucrose (1–2 %), and ash originating from extraneous matter. Sugar factory produces 30 tons of wet bagasse after crushing 100 tons of sugarcane. Bagasse is generally found as a primary fuel source for sugar mills which when burned then generates sufficient electrical energy, used to fulfill all the basic needs of a sugar mill. The most energy projects have been demonstrated and presented in many sugar- cane producing countries*(Har, et al, 2013).* The power generation from sugarcane is a good option as renewable energy that increases sustainable development, increases profitability and competitiveness in the industry.

2.3.3 Applications of Bagasse Fibers

2.3.3.1 Agricultural End-Use: The other application of bagasse fiber nonwovens can be found to make flowerpots. This type of flowerpot made has excellent biodegradability and can be buried in a clay pots. The bagasse nonwoven pot buried in flowerbed is dissolved within only 23 days. When the nonwoven pot is put in a larger plastic pot, it is biodegraded within 50 days.

2.3.3.2 Sugarcane Bagasse Paper: For high quality paper making, sugarcane bagasse is one of the most eco-friendly, sustainable, and renewable resources. The bagasse fiber generally used state-of-the-art technology and creates a bagasse pulp which is suitable for high quality paper making(*da Cruz Sessa et al., 2016*). The important application of bagasse fiber can be found in newsprint papers which are produced from 100 % bagasse fiber. High quality office and printing papers generally have a 20 % internal fiber added to ensure that the paper is suitable for all office and print applications.

2.3.3.3 Production of Ethanol from Sugarcane Bagasse: The second generation bio fuel technology for the production of ethanol is cellulosic ethanol technology. Cellulose plants are the main source for the production of cellulosic bio fuels*(Singh Nee Nigam & Pandey, 2009*). They have categorized it as “energy crops” rather than the crops for food production. Some of the examples are perennial grasses and trees, like switch grass and Miscanthus. Another source of cellulosic biomass is residues (crop) such as stems and leaves. Generally it has been observed that the lignocellulosic biomass is the main feed stock for ethanol and includes various materials like agricultural residues such as corn Stover, husks, bagasse, woody crops, waste paper and municipal and industrial wastes *(Nogueira, 2008*). Environmental issues can be resolved by using or disposal of agricultural waste residues and other wastes for the production of bio ethanol. There is no interference of the lingo cellulosic feed stocks with food security and are important in terms of energy security in all areas, environmentally and also agricultural development and employability.

2.3.3.4 Bio adsorbent for textile wastewater treatment: Effluent from the textile industry is a major source of environmental pollution especially water Pollution. Among the various stages of textile industry, dying plant is the most pollutant producing stage. The textile dyeing waste contains unused or partially used organic compounds, strong color and high Chemical Oxygen

Demand (COD) and Biological Oxygen Demand (BOD). The intensity of pollution depends on fabric. Beside that the dyeing sequence, dyeing equipment and the liquor ratio used in the textile industries can also affect the pollution intensity.Bioadsorbent made using sugar cane bagasse is basically built by macromolecules with humic and fulvic substances, lignin, cellulose, hemicelluloses and proteins that have adsorptive sites such as carbonyl, carboxylic, amine and hydroxyl groups, able to adsorb the dyes and other component of textile wastewater. Therefore sugarcane bagasse can applied as a way of treatment for textile wastewater.

2.4 Chemical Composition of Bagasse

2.4.1 Cellulose and hemicelluloses: They are present in the form of hollow cellulose in bagasse, which contributes to about 70 % of the total chemical constituents present in bagasse.

2.4.2 Lignin: It acts as a binder for the cellulose fibers and also behaves as an energy storage System*(Centre & Industrial, 2016*). Bagasse consists of water, fiber and small quantities of solids in solution in the following proportions. Water 46 - 57 % (mean50%), Fiber 43% - 53 % (mean 47%), Solids in solution (sugar) 2% - 6 % (mean 3%).It is a composition within certain limits as variable and depends in the varieties, their maturity, the harvest technique and efficiency of milling. By definition the fiber of the bagasse is the component which is insoluble in water. It consists of mainly cellulose pentose and lignin. Cellulose is a polysaccharide having the general formula $(C_6H_{10}O_6)$ n and the main constituent of vegetable tissue.

2.5 Physical Composition of Bagasse

Physically bagasse fiber is considered to be made up of 60% true fiber and 40% pith. Physical composition varies within narrow limits. As adsorption process is the main concern sugarcane bagasse has a water absorption capacity of 70-75%, and a fiber dimension 18-26 millimetre. The structure of true fiber includes cellulose, hemicelluloses and lignin *(Cruz, et al, 2018).*The cellulose component of fiber plays a great role in adsorption processes.

2.6 Surface Properties of Sugarcane Bagasse

2.6.1Mechanical Properties

2.6.1.1 Degradation from Water Ingress

An important property of any resin, particularly in a Marin environment is its ability to withstand depredation from water ingress (*Rodrigues, et al, 2011*). All resins will absorb some moisture, adding to laminates weight, but what is more significant is how the absorbed water affects the resin and resin fiber bond in laminate, leading to a gradual and long term loss in mechanical properties.

An epoxy laminate immersed in water for a period of one year will retain around 90% of its inter-laminar shear strength. Whereas both polyester and vinyl resins are prone to water degradation due to the presence of hydrolysable ester groups in their molecular structures, as a result, a thin polyester laminate can be expected to retain only 65% of its inter-laminar shear strength for the same period *(Cerqueira, et al, 2011).*

2.6.1.2Adhesive Properties

Adhesive properties of the resin system are important in realizing the full mechanical properties of a composite (*Benedito, et al, 2009*). The adhesion of the resin matrix to the fiber reinforcement or to a core material in a sandwich construction is important. Epoxy system offer the best performance of all the three resins considered here. Polyester resins generally have the lowest adhesive properties of the three. On the other hand, vinyl-ester resin shows improved adhesive properties over polyester *(El-Fattah, et al, 2015).* Due to this performance property, epoxy resins are frequently found in many high strength adhesives *(Jorge, et al, 2012).* This is due to their chemical composition and the presence of polar hydroxyl and ester group. The strength of bond between resin and fiber is not solely depended on the adhesive property of the resin system but is also affected by surface coating on the reinforcement fibers.

2.6.1.3 Micro-Cracking

The strength of a laminate is usually thought of in terms of how much load it can withstand before it suffer composition failure *(Salim, et al, 2014*). This ultimate or breaking strength is the point at which the resin exhibits catastrophic breakdown and the fiber reinforcements break.

However, before this ultimate strength is achieved, the laminate will spread through the resin matrix. This is known as 'transverse microcracking'and, although the laminate has not completely failed at this point, the breakdown process has commenced. As the ultimate strength of a laminate in tension is governed by the strength of the fibers, these resin micro-cracks do not immediately reduce the ultimate properties of the laminate *(Sai, et al, 2012).* Increased resin/fiber adhesion is generally derived from both the resin chemistry and its compatibility with the chemical surface treatments applied to fibers. Here the well known adhesive properties of epoxies help laminates achieve higher micro-cracking strains.

2.6.2 Physical Properties

The physical properties of sugarcane bagasse are thickness, solubility, water vapour transfer rate, surface structure and color of the hemicelluloses films*(Journal et al., 2015).*The surface structure are fiber structures and pith. The fiber surface is formed by parallel stripes and is partially covered with residual material. In contrast, pith is a more fragile and fragmented structure containing pits, which are small pores connecting neighbouring cells on the surface of the walls. Bagasse is similar in many respects both physically and chemically to other cellulosic waste biomass.

2.6.3 Chemical Properties

Sugarcane bagasse hemicelluloses is predominantly constituted of xylan, which is composed of xylose units in the backbone (*Bansal & Figueiredo, 2016).* Xylose accounts more than 70% in the xylan, in average, with a reported range of 43 to 93%.This polysaccharide shows branch groups of arabinose, acetyl groups and uronic acids. Xylan backbone is made up of xylose linked by β-1, 4 linkages with branch groups at the positions C_2 and C_3 with arabinosyl, uronic acids and acetyl groups. Arabinose is the branch group that appears in a higher amount. It is linked to xylan backbone by α-1, 2 or α-1, 3 linkage; galactose is linked by β-1, 5 linkage to the xylan backbone. Xylan branch groups can be associated with aromatic. Sugarcane bagasse is a heterogeneous material; the fractions of the Culm contain different amounts of xylan *(Mutua,et al, 2016).* The more branched chain correlates to solubility, which is important considering molecule reactivity. The branches also collaborate to chain characteristics, such as charge, which depends on the substituent groups.

Hemicelluloses are naturally associated to cellulose and covalently linked to lignin. The association of lignin and hemicelluloses is done by several types of linkages that are resistant to alkaline or peroxide action. Sugarcane bagasse has hydroxycinnamic acid (ferulic, coumaric and sinapic acids) involved in cross-linking xylan and lignin molecules. Sugarcane bagasse xylan can be esterified to lignin. The hydroxyl groups of lignin units can be linked to carboxylic acid groups of uronic acids. Native or extracted xylan contains molecules with a different number of xylose units, which means different degrees of polymerization (DP). The DP is defined as the number of xylose units in a xylan molecule. Finally hemicelluloses and Cellulose have hydrophilic properties whereas lignin and ash have hydrophobic properties. Hemi cellulose is strongly bound to cellulose fibrils presumably by hydrogen bonds. Hemicelluloses polymers are branched, fully amorphous and have a significantly lower molecular weight than cellulose. Because of its open structure containing many hydroxyl and acetyl groups, hemicelluloses is partly soluble in water and hygroscopic.

Cellulose is a semi crystalline polysaccharide made up of D-glucopyranose units linked together by β-(1-4)-glucosidic bonds and the large amount of hydroxyl group in cellulose gives natural fiber hydrophilic properties. Therefore when the fibers are used to reinforce hydrophobic matrix, it results in a very poor interface and poor resistance to moisture absorption.

Lignins are amorphous, highly complex, mainly aromatic, polymers of phenyl propane units but have the least water sorption of the natural fiber components. Lignin is an aromatic biopolymer constituted mainly of phenyl propane substituted units bonded together to form a polymer of low regularity, crystallinity and optical activity. Although about 20 types of bonds exist within lignin, the largest number is related to links between ether bonds. The cellulose molecule is formed by a crystalline part and an amorphous part. The lingo cellulosic biomass crystalline represents the relative amount of overall crystalline cellulose in the solid fraction of the biomass (*Journal et al., 2015*). To optimize the interfacial properties between fiber and polymer matrix due to hydrophilic nature of natural fibers, a chemical modifications using HCl solution will be used to eliminate lignin and hemicelluloses on fibers surface. A significant change in morphological structure of the bagasse fiber will be occurred after treatment with HCl. As a result a highly fibrillated surface will be created with a decrease of fibers diameter or an

increase of the surface area due to the cleaning of the bagasse surface from impurities, wax cuticle layer, and hemicelluloses structures.

2.7 Textile Wastewater Characteristics and Conventional Treatment

The pollutant features of textile wastes differ widely among various Organic substances such as dyes, starches and detergents in effluent undergo chemical and biological changes which consume dissolved oxygen from the receiving stream and destroy aquatic life *(Marrot & Roche, 2002).* Such organics should be removed to prevent septic conditions and avoid rendering the stream water unsuitable for municipal, industrial, agricultural and residential uses.

2.7.1 Component of Untreated Textile Wastewater

The following are the component of untreated textile wastewater according to literature *(R Ananthashankar, 2013):* pH 6-10, temperature (°C) 35-45 ,total dissolved solids (mg/L) 8,000-12,000, BOD (mg/L) 80-6,000 ,COD (mg/L) 150-12,000 ,total suspended solids (mg/L) 15-8,000 ,total Dissolved Solids (mg/L) 2,900-3,100 ,chlorine (mg/L) 1,000-6,000 ,free chlorine (mg/L) <10 and Sodium (mg/L) 70%, trace elements (mg/L) Fe <10, Zn <10, Cu <10, As <10, Ni <10, B <10, F <10, Mn <10 ,V <10 ,Hg <10 ,PO4 <10 ,oil & grease (mg/L) 10-30 ,free ammonia (mg/L) <10, SO4 (mg/L) 600-1000,silica (mg/L) <15,and color (Pt-Co) 50-2,500.

2.7.2 Conventional Treatments of Textile Effluents

Effluents discharged from the textile industries undergo various physico-chemical treatments such as flocculation, coagulation and ozonation and biological treatment for the removal of nitrogen, organics, phosphorus and metal removal *(Kharat, 2015).* The disadvantages of the physico-chemical treatment process are: (a) the formation and disposal of sludge and (b) the required space. 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. Treatment of textile effluents involves three treatment processes: primary, secondary and tertiary treatments.

2.7.2.1 Primary Treatment

Primary treatment is a physical process. In this process wastewater flow is slowed down and suspended solids settle to the bottom by gravity the material that settles is called sludge or bio solids *(Seif & Malak, 2001*). Sludge from the primary sedimentation tanks is pumped to the sludge thickener more settling occurs to concentrate the sludge prior to disposal. Primary treatment reduces the suspended solids and the B.O.D. of the wastewater.

2.7.2.2 Secondary Treatment

From the primary treatment tanks water is pumped to the trickling filter for secondary treatment. Secondary treatment further reduces the suspended solids and B.O.D of the waste water. Secondary treatment is a biological process utilizes bacteria and algae to metabolize organic matter in the wastewater (*Lin & Chen, 1997*). In this process water runs over a plastic media and organisms clinging to the media remove organic matter from the water. From secondary treatment on the trickling filter water flows to the final clarifiers for further removal of sludge (*Lin & Chen, 1997*). The final clarifiers are another set of primary sedimentation tanks. The final clarifiers remove additional sludge and further reduce suspended solids and B.O.D.

2.7.2.3 Tertiary Treatment

There are several technologies used in tertiary treatments including electro dialysis, reverse osmosis and ion exchange. Electrolytic precipitation of textile effluents is the process of passing electric current through the textile effluent using electrodes (*Wang, et al, 2011*). As a result of electro chemical reactions, the dissolved metal ions combine with finely dispersed particles in the solution, forming heavier metal ions that precipitate and can be removed later. One of the disadvantages is that a high contact time is required between the cathode and the effluent. Reverse osmosis is a well-known technique which makes use of membranes that have the ability to remove total dissolved solid contents along with ions and larger species from the effluents *(Habib, 2017*). Cotton dyeing processes use electrolytes such as NaCl in high concentrations. These high concentrations of salts can be treated using reverse osmosis membrane. Electro dialysis is another process which uses membranes, that has the ability to separate dissolved salts. The electricity used in electro dialysis influences the ions to get transported through a semi permeable membrane by passing an electrical potential across water. The membranes used are

charge specific and anion-selective which allows negatively charged particles to pass through and traps positively charged particles and vice versa. Placing numerous membranes throughout the system hinders the flow of effluent and the effluent would reach a point at which the ions are trapped or settled down and the remaining ions are neutral in charge. Membrane fouling (the process where solutes or other particles get attached to the membrane or into the membrane pore) has to be prevented by removing suspended solids, colloids and turbidity prior to electro dialysis.

Ion exchange method is a commonly used tertiary method which involves the passage of effluents through the beds of ion exchange resins. These ion exchange resins are either cationic or anionic charged. Effluent passing thorough a cationic resin would have its cat ions removed by the resin and replaced with hydrogen ions making it acidic *(Central Pollution Control Board Ministry of Environment and Forests, 2015).* When the acid solution is passed through anion resin, the anions would be substituted with hydroxyl ions. Photo catalytic degradation is another method by which a wide range of dyes can be decolorized depending on their molecular structures (*Petrou & Mantzavinos, 2013*). Adsorption is also found to be effective in the removal of colours. Thermal evaporation using sodium per sulphate is also found to have a good oxidizing potential (*Attia, et al, 2008*). This process is found to be eco- friendly because they do not have the property to form sludge and also do not emit toxic chlorine fumes during evaporation.

2.7.3 Standard for Inland Surface Water: The following standards are provided based on *(Tafesse TB, et al, (2015) BThe Physico-Chemical Studies of Wastewater in Hawassa Textile Industry. J Environ Anal Chem 2: 153. doi:10.4172/2380-2391.1000153)*

Table 1: Guide Line limit of textile wastewater discharging

Name of the parameter	Guide line limit
PH	6 – 9
Biological oxygen demand	≤ 30 mg/L
Chemical oxygen demand	≤ 160 mg/L
Total dissolved substance	≤ 250 mg/L
Total suspended solids	≤ 30 mg/L

2.8 Environmental and Health Effects of Textile Industry wastewater

There is mounting pressure for companies to be environmentally responsible in the way they produce and source materials.

Azo dyes, a type of textile colorant, are integral to the textile industry and make up 70% of commercial dyes. Research has shown that some azo dyes pose very serious health risks to humans if they are used in particular textiles and if they get into certain water supplies. Azo dyes have been shown to damage ecosystems when discharged into water systems by dyeing factories, predominantly in developing countries.

'Azo colorants' are used to color textile fibers, leather, plastics, papers, hair, mineral oils, waxes, foodstuffs and cosmetics. 'Azo dye' is the collective term used to describe a group of synthetic that rose to prominence in the 1880 and are now comprise 70% of all organic commercial dyes. The word 'Azo' signifies the presence of a chemical azo group (-N=N-) in the dye. Azo dyes are popularly used, because they dye cloth at 60°C, while azo-free dyes require 100°C. Also, azo dyes offer an extensive range of colours, better color fastness and four times the intensity of the closest alternatives, making them invaluable to the textile industry (F.M.D. Chequer et al. (2013). Textile Dyes: Dyeing Process and Environmental Impact).

However, under certain (reductive) conditions the azo group can cleave, producing potentially dangerous substances known as aromatic amines. These conditions are met in the digestive tracts and some organs of animals, including humans.

2.8.1 Ecological Impact of Azo Dyes

The textile industry is a heavy polluter of waste gas, solids, water and noise (Gudelj et al., 2011). Wastewater is the most environmentally damaging, and the effluent from textile plants is classified as the most polluting of all the industrial sectors, considering the volume generated as well as its composition *(Mathur,et al, 2012*). The dyestuff lost through the processes of the textile industry poses a major problem for wastewater management. An estimated 200,000 tons of dyestuff is expelled into the global environment every year. The concentration of azo dye in textile effluent can reach 500 parts per million (ppm).

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2.8.2 Health Concerns

Taking poisoning in its worst case scenario, the total exposure of an adult over a lifetime use of a garment covering the whole body is calculated a 723 µg/kg, posing a very low cancer risk, though this risk has not been satisfactorily quantified. Consumption through water can reach much higher levels of exposure with higher absorption rates, and is theoretically more dangerous. No epidemiological studies regarding possible carcinogenic effects of azo dyes in humans are available.

2.8.3 Water Remediation

Because of their chemical stability and synthetic nature, reactive azo dyes are not totally degraded and exhibit slow degradation by conventional wastewater treatment methods (Arfmann, et al, 1997). They are additionally difficult to remove, because they are designed to be stable in aerobic conditions but bio treatment in anaerobic conditions can result in the generation of dangerous aromatic amines. Remediation does not currently capitalize on this fact.

Wastewater is usually treated with activated sludge, and the liquid effluent is released to nearby surface waters. Azo dye stability in both activated sludge and water indicate that relatively stable in the aquatic environment and cannot be effectively degraded in standard wastewater plants. Physicochemical methods can be used to minimize toxicity levels, but neutralization is not complete and a more concentrated sludge is created, effectively transferring the pollution problem between phases. The cost and processing time are also unsatisfactory.

2.8.4 The Most Important Constituents-Dyes: Structure and Properties

Dyes are natural and synthetic compounds that make the world more beautiful through colored products. Textile dyes represent a category of organic compounds, generally considered pollutants, discharged into wastewaters resulting mainly from processes of chemical textile finishing. It is estimated that over 10,000 different dyes and pigments are used industrially and

over 7×10^5 tons of synthetic dyes are annually produced worldwide. A dye is used to impart colour to a material, of which it becomes an integral part. An aromatic ring associated with a side chain is usually required for resonance and thus to impart colour. The characterization of dyes is based on their chemical structure and application. Dyes are composed of the atoms responsible for the dye colour called chromophores as well as an electron-withdrawing or electron-donating substituent that causes or intensifies the colour of chromophores, called auxochrome *(Gürses, et al, 2016).* To be a dye, a compound must contain both the chromospheres and auxochrome.The most important chromophores are azo (–N=N–), carbonyl (–C=O), methine (–CH=), nitro (–NO_2), and quinoid groups. The most important auxochromes are amine (–NH_3), carboxyl (–COOH), sulfonate (–SO_3H), and hydroxyl (–OH). In general, azo dyes can occur in two tautomeric forms, azo (–N=N–) or hydra zone (=N–NH–). The latter is said to be more prone to oxidative fading, which is the most common photo degradation mechanism in the presence of light, moisture, and oxygen.

In general, textile fibers can catch dyes in their structures as a result of van der Waals forces, hydrogen bonds, and hydrophobic interactions (physical adsorption). The uptake of the dye in fibers depends on the dye's nature and its chemical constituents. But, the strongest dye–fiber attachment is a result of a covalent bond with an additional electrostatic interaction where the dye ion and fiber have opposite charges (chemisorption).

Reactive dyes, including many structurally different dyes, are extensively used in the textile industry because of their wide variety of colour shades, high wet fastness profiles, ease of application, brilliant colours, and minimal energy consumption. The three most common groups are azo, anthraquinone, and phthalocyanine dyes, most of which are toxic and carcinogenic. All dyes used in the textile industry are designed to resist fading upon exposure to sweat, light, water, many chemicals including oxidizing agents, and microbial attack.

The textile azo dyes are characterized by relatively high polarity and high recalcitrance. Recalcitrance is difficult to evaluate because of the dependence of degradation on highly variable boundary conditions. Furthermore, azo dyes are relevant in terms of eco- and human toxicity, industrially produced in high quantities, and known to occur in hydrosphere. Azo dyes also have great structural diversity, high molar extinction coefficients, and medium-to-high fastness properties in relation to light as well as to wetness. Depending on pH value, azo dyes can be

anionic (deprotonated at the acidic group), cationic (protonated at the amino group) or non-ionic. Accordingly, knowledge of the acidity constants is indispensable for the characterization of azo dye behaviour. Environmental partitioning is influenced by substituent's as well as the number of carbon atoms and the aromatic structure of the carbon skeleton.

2.8.5 Textile Dye Classification

Textile dyes can be classified in several ways.

(a) Natural dyes, the dyes extracted from vegetable and animal resources and mainly used in textile processing (*Premo & Polymer, 2005)* . (b) Synthetic textile dyes, Recently, textile dyes are mainly classified in two different ways: (a) Based on their application characteristics (i.e., Color Index (CI) Generic Name such as acid, basic, direct, disperse, mordant, reactive, sulphur dye, pigment, vat, azo insoluble) (b) Based on their chemical structure (i.e., CI Constitution Number such as nitro, azo, carotenoid, diphenylmethane, xanthene, acridine, quinoline, indamines, sulphur, amino- and hydroxy ketone, anthraquinone, indigoid, phthalocyanine, inorganic pigment, etc.).

Considering only the general structure, textile dyes are also classified as anionic (direct, acid, and reactive dyes), non-ionic (disperse dyes), and cationic dyes (azo basic, anthraquinone disperse, and reactive dyes).The major textile dyes can be included in the two high classes: azo or anthraquinone (65–75 % of total textile dyes).

Azo dyes the annual world production of azo dyes is estimated to be around 1 million ton and more than 2,000 structurally different azo dyes are currently in use. Azo dyes which are aromatic compounds with one or more –N = N– groups, constitute the largest class of synthetic dyes used in commercial applications constituting 60–70 % of all dyestuffs produced. This linkage (–N = N–) may be present more than once and thus mono azo dyes have one azo linkage while there are two linkages in diazo dyes and three in triazo dyes. Azo dyes have one or more azo groups (R1-N = N-R2) having aromatic rings mostly substituted by sulfonate groups. These complex aromatic substituted structures make a conjugated system and are responsible for the intense colour, high water solubility, and resistance to degradation of azo dyes under natural conditions.

2.8.6 Toxicity of Dyestuffs

Dyes are the most important chemical constituents used in the textile industry, which impart colour to yarn or cloth. Wastewater effluent, generated from the textile industry, is a complex mixture of many polluting substances ranging from organ chlorine-based pesticides to heavy metals associated with dyes and the dyeing process. During dyeing processing, a large amount of the dyestuffs is released due to inefficiencies in the dyeing process and is directly lost to the wastewater, which ultimately finds its way into the environment. In addition, antimicrobial agents resistant to biological degradation are frequently used in the manufacture of textiles, particularly for natural fibers such as cotton. For instance, azo dyes, which amount to around 60 % of textile dyes, display strongly adverse effects on the growth of methanogenic bacterial cultures. This toxicity may be due mainly to the azo functional group itself rather than to the products of reductive cleavage. Therefore, the effluent is also resistant to biodegradation. The toxic effects of the azo dyes may result from the direct action of the agent itself or of the aryl amine derivatives generated during reductive biotransformation of the azo bond. The azo dyes entering the body by ingestion can be metabolized to aromatic amines by the azo reductases of intestinal microorganisms*(Ventura-camargo & Marin-morales, 2013*). If the dyes are nitro dyes, they can be metabolized by the nitro reductases produced by the same microorganisms. Mammalian liver enzymes and other enzymes may also catalyze the reductive cleavage of the azo bond and the nitro reduction of the nitro group. In both cases, if N-hydroxyl amines are formed, these compounds are capable of causing DNA damage.

Unfortunately, heavy metals have often been used in dye fixatives and also in dyes. Typically, transition metals such as chrome, copper, nickel, and cobalt are used. These metals can form multiple bonds with organic dyestuffs and/or fibers. Metals can be present in dyes for two reasons: first, metals are used as catalysts during the manufacture of some dyes and can be present as impurities; second, in some dyes the metal is chelated with the dye molecule, forming an integral structural element.

Toxic chemicals sometimes found in the dyeing process according to the literature *(Swedish Chemicals Agency, 2014).*Dioxin a carcinogen and possible hormone disrupter, toxic heavy metals such as chrome, copper, and zinc known carcinogens, formaldehyde a suspected carcinogen and azo dyes group which give off carcinogenic amines.

2.8.7 Characteristics and Composition of Textile Wastewater

In addition to the problem caused by the loss of dye during the dyeing process, the textile industry is generating large volumes of effluent (*Bisschops & Spanjers, 2003*). These effluents are complex mixtures of many pollutants, ranging from original colours lost during the dyeing process to associated pesticides and heavy metals, and if these pollutants will not be properly treated, they can cause serious contamination of the water sources.

Textile industries utilize a huge amount of water. The amount of water used varies widely, depending on the specific processes operated at the mill, the equipment used, and the prevailing philosophy of water use. The daily water consumption of an average-sized textile mill having a production of about 8,000 kg of fabric per day is about 1.6 million litters. Sixteen percent of this is consumed in dyeing and 8 % in printing. Textile industries typically generate 200–350 m^3 of wastewater per ton of finished product resulting in an average pollution of 100 kg chemical oxygen demand (COD) per ton of fabric. The dye house releases two types of wastewater, namely, dye bath water and wash water/rinse water. The dye bath water mainly consists of complex dyestuff and various intermediate complexes. It was noticed that in a typical factory the effluent from the dye bath had COD 5000–6000, total dissolved solids (TDS) 52,000, Suspended Solids 2,000 mg L−1, and pH 9. After dyeing, the fabrics are washed by rinsing in water to remove the excess dye present. The wastewater generated due to this operation is commonly called “wash water” having COD 400–860, TDS 3,200 mg L−1 and pH 8. Effluents contain a high organic load and biochemical oxygen demand, low dissolved oxygen concentrations, strong color, and low biodegradability.

During dyeing processes, the entire dye is not fixed to the fiber and a certain amount of the dye remains in dye bath, which is released with effluents. During textile processing, inefficiencies in dyeing result in large amounts of the dyestuff being directly lost to the wastewater, which ultimately finds its way into the environment. The amount of dye lost is dependent upon the class of dye application used, varying from only 2 % loss when using basic dyes to a 50 % loss when certain reactive dyes are used . Very low concentrations of dyes in effluent are highly visible and their presence is undesirable. A huge amount of effluent from textile mills is being discharged on land or into watercourses. This effluent is characterized by

high biological oxygen demand (BOD), COD, sodium and other dissolved solids as well as micronutrients and heavy metals.

Water is also needed for cleaning the printing machines to remove loose color paste from printing blankets, printing screens, and dyeing vessels. The other feature of this industry, which is a backbone of the fashion garment industry, is the large variation in the demand for type, pattern, and color combination of fabric resulting in significant fluctuation in waste generation volume and load *(Hussain, et al, 2004)*.

2.8.8 Environmental and Health-Related Issues of Textile Wastewater

The environmental issues associated with residual dye content or residual color in treated textile effluents are always a concern for each textile operator that directly discharges, both sewage treatment works and commercial textile operations, in terms of respecting the color and residual dye requirements placed on treated effluent discharge *(Malik,et al, 2014)*.

Water pollution caused by industrial effluent discharges has become a worrisome phenomenon due to its impact on environmental health and safety (*Forgacs, et al, 2004*). Textile industries contribute immensely to surface water deterioration and are categorized among the most polluting of all industrial sectors. Effluents from textile industries are complex mixtures of chemicals varying in quantity and quality (*Chequer et al., 2013)*. These industries can generate both inorganic and organic waste mixed with wastewaters from the production processes, which leads to change in both biological and chemical parameters.

2.8.8.1 Environmental Issues

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.

The key environmental issues associated with textile industry are water use, treatment, and disposal of aqueous effluent (*Zhezhova, et al, 2014)*. 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.

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 *(Shaikh, 2009)*. The extent of environmental pollution due to dye bath water is very high. Environmental pollution caused by the release of a wide range of azo dyes through industrial wastewater is a serious problem in the present day(*Lade, et al, 2015)*. 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 sulphur, naphthol, 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. Also, the accumulation of color hinders sunlight penetration, disturbing the ecosystem of the receiving water.

In addition when this effluent is allowed to flow in the fields, it clogs the pores of the soil resulting in loss of soil productivity. The texture of soil gets hardened and penetration of roots is prevented. The wastewater that flows in the drains corrodes and incrustates the sewerage pipes. If wastewater is allowed to flow in drains and rivers, it affects the quality of drinking water in hand pumps making it unfit for human consumption. The color in watercourses is accepted as an aesthetic problem rather than an eco-toxic hazard. Therefore, the public seems to accept the blue, green, or brown color of rivers but a “non natural” color such as red and purple usually causes the most concern. Wastewater also leads to leakage in drains increasing their maintenance cost. Other environmental issues of equal importance are air emission, notably volatile organic compounds (VOC) s, and excessive noise or odour as well as workspace safety.

Most processes performed in textile mills produce atmospheric emissions. Textile mills usually generate nitrogen and sulphur oxides from boilers. Other significant sources of air emissions in textile operations include resin finishing and drying operations, printing, dyeing, fabric preparation, and wastewater treatment plants. Hydrocarbons are emitted from drying ovens and from mineral oils in high-temperature drying/curing. These processes can emit formaldehyde, acids, softeners, and other volatile compounds.

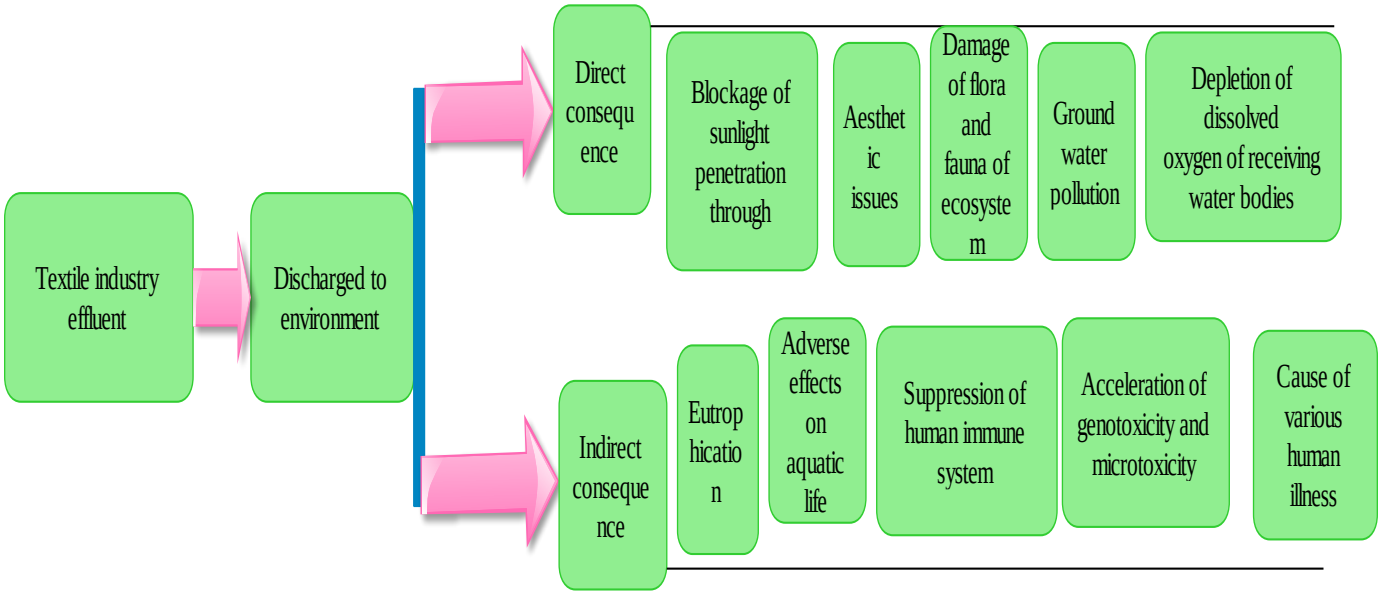

Figure 1: Schematic representation of the effects of textile wastewater discharged into the environment

2.8.8.2 Health Problems

The direct discharge of textile wastewater into water bodies like rivers, etc., pollutes the water and affects the flora and fauna (*Akarslan & Demiralay, 2015*). Depending on exposure time and dye concentration, dyes can have acute and/or chronic effects on exposed organisms. Depletion of dissolved oxygen in water is the most serious effect of textile waste as dissolved oxygen is very essential for marine life and this also hinders the self-purification process of water.

The dyes used in textile industries are potential health hazards as they may be converted to toxic and/or carcinogenic products under anaerobic conditions. Many dyes are toxic to fish and mammalian life; they inhibit growth of microorganisms and affect flora and fauna. Apart from this, several dyes and their decomposition derivatives have proved toxic to aquatic life (aquatic plants, microorganisms, fish, and mammals). They are also carcinogenic in nature and can cause intestinal cancer and cerebral abnormalities in the fetus. Textile dyes can cause allergies such as contact dermatitis and respiratory diseases, allergic reaction in the eyes, skin irritation, and irritation to mucous membrane and the upper respiratory tract. Reactive dyes form covalent bonds with cellulose, woollen, and polyamide (PA) fibers. It is assumed that, in the same way,

reactive dyes can bind with $–NH_2$ and –SH groups of proteins in living organisms. Additionally, fairly intensive studies have inferred that such colored allergens may undergo chemical and biological assimilations, cause eutrophication, consume dissolved oxygen, prevent re-oxygenation in receiving streams, and have a tendency to sequester metal ions accelerating genotoxicity and micro toxicity. A high potential health risk is caused by adsorption of azo dyes and their breakdown products (toxic amines) through the gastrointestinal tract, skin, lungs and also formation of haemoglobin adducts and disturbance of blood formation *(Nergis,et al, 2009)*. Several azo dyes cause damage to DNA that can lead to the genesis of malignant tumors. Electron-donating substituent in ortho and para position can increase the carcinogenic potential of these dyes. Some of the best-known azo dyes (e.g., Direct Black 38 azo dye, a precursor of benzidine; azodisalicylate, a precursor of 4-phenylenediamine) and their breakdown derivatives that induce cancer in humans and animals are benzidine and its derivatives and also a large number of anilines (e.g., 2-nitroaniline, 4-chloroaniline, 4, 4'-dimethylendianiline, 4-phenylenediamine, etc.), nitrosamines, dimethylamines, etc. In addition to the environmental problem, the textile industry consumes large amounts of potable water. In many countries where potable water is scarce, this large water consumption has become intolerable and wastewater recycling has been recommended in order to decrease the water requirements.

Inhaling dust produced during cotton, flax, or hemp handling causes byssinosis, which is a respiratory syndrome. Today, byssinosis is among one of the most significant health problems in the entire textile industry. The noise level resulting from the machines used in the textile industry, especially from the dry processes, may violate the limit allowed by the law and cause hearing problems. The use of dyestuffs and pigments may cause a number of adverse effects to health. Health effects may be exerted directly at the site of application (affecting the workers) and later in the life cycle (affecting the consumers).

3. MATERIALS AND METHODS

3.1 Chemicals and Materials Used

All chemicals and reagents used in this study were of analytical grade and commercially available. Raw biomass material was sugarcane bagasse. Reagent used to characterize the COD of textile raw wastewater, and acetone, sulphuric acid, hydrochloric acid and sodium hydroxide as well as potassium hydro oxide were the chemicals used in this study. Azo dye that is methyl red was obtained from Yrgalem Addis textile factory. Distilled water was used in the entire accomplishment of the experiment.

3.2 Experimental Method

3.2.1 Collection and preparation of adsorbent: The adsorbent used for this study was sugarcane bagasse. The sugar cane bagasse was collected from wonji sugar factory in December 23. The sugarcane bagasse was soaked in 0.5M hydrochloric acid solution for twenty four hours by exposing the soaked bagasse for stirrer, and then adjusted to a pH of 4.32 with 0.3 Molar of NaOH (Sodium hydro oxide).Finally the pH adjusted and soaked sugarcane bagasse was washed with distilled water for three round so that to remove all the dirty particle, lignin and acid. Finally the sugarcane bagasse was dried in oven at 130 ℃ for twelve hours in order to remove the moisture in secured and well manner.

3.2.2 Preparation of standard solution: A stock solution of azo dye that is methyl red was prepared by dissolving one gram of dye in 1000 ml volumetric flask followed by dilution of distilled water to the concentration of 45 mg/L, 48.75 mg/L, 52.5 mg/L,56.25mg/L and 60mg/L, and for textile raw wastewater the concentrations were set at the same concentration with azo dye that is methyl red concentration. Distilled water was used to prepare all solution of azo dye that is methyl red.

3.2.3 Preparation of textile wastewater: The raw textile wastewater was taken from Yrgalem Addis textile factory from dyeing department. The raw waste water was characterized for the parameters of total dissolved solids (TDS), biological oxygen demand (BOD), chemical oxygen demand (COD) and pH values as per APHA standards.

3.3 Characterization of Textile Wastewater

3.3.1 Determination of Biological Oxygen Demand (BOD)

157 mL of raw waste water was taken to characterize biological oxygen demand (BOD) and then the raw waste water was characterized for biological oxygen demand (BOD) with in twenty four hours by adjusting the raw waste water in to a pH of seven so that to create comfortable environment for the microorganisms and using potassium hydro oxide and nitrifying bacteria inhibitor ($C_4H_8N_2S$) inserted into BOD incubator for five days. The biological oxygen demand (BOD) of textile raw wastewater was characterized before and after adsorption.

3.3.2 Determination of Chemical Oxygen Demand (COD)

First the chemical oxygen demand (COD) reactor was hotted for thirty minutes, and then the COD reagent was mixed with 2 mL of textile raw wastewater, and it reacts for two hours in the COD reactor for both before and after adsorption of textile raw wastewater.

3.3.3 Determination of Total Dissolved Solids (TDS) and pH

The wastewater was also characterized the composition of total dissolved solids (TDS) by taking the textile raw wastewater 20 mL and filtered by using a 90 mm of qualitative filter paper on a vacuum filtration and the sample was inserted in to oven at a temperature of 105 ℃ for eighteen hours using crucible in order to obtain the dissolved solids without any loss, then when the sample was weighted using analytical balance to obtain the yield of total dissolved solids (TDS) of textile raw wastewater from 20 mL of textile raw wastewater.

Textile raw wastewater was characterized its pH value, and the pH value before adsorption was basic property that is 9.65.

3.4 Characterization of sugarcane bagasse

3.4.1 Determination of Volatile Matter

The volatile matter was determined according to ISO 562/1974.Four gram of two samples of the sugarcane bagasse was burned in a crucible at a temperature of 800 ℃ in the muffle furnace

for five minutes and allowed to cool down in a desiccator. The volatile matter content was calculated by using the following equation:

$$\%\text{Volatile matter content} = \frac{(Initial\ weight - Final\ weight)}{Initial\ weight} \times 100 \quad (1)$$

3.4.2 Determination of Moisture Content

The moisture content of sugarcane bagasse was determined using oven at a temperature of 105 ℃ based on the ASTM D 1037 (1991). The moisture content was characterized by weighing the sample and inserted in to the oven and the sample to stay in the oven for 20 to 25 minute specifically the samples retention time were 25 minute in the oven. Once the samples were dried in the oven, the dried samples were withdrawn from the oven and weighed the samples until constant weight was maintained from the samples by put in and put out the samples in and out of the oven consecutively. Here three samples were taken and the average value was calculated. The moisture content was calculated by using the equation:

$$\%\ \text{Moisture of bagasse} = \frac{(w_i - w_f)}{w_i} \times 100\% \quad (2)$$

Where:

w_i = Initial mass of bagasse

w_f = Final mass of bagasse

3.4.3 Determination of Ash Content

Ash content was characterized based on the ASTM D 2017 (1998).The ash content of sugarcane bagasse was characterized by inserting three samples of 4 gram each in to furnace for 520^{o}C for four hours using a pre-weighted crucible. The crucible was transferred in to a desiccator for cooling and then finally the output ash content was weighed. The ash content was calculated by the following equation:

$$\%\ \text{Ash content} = \frac{(w_2 - w_o)}{w_1 - w_o} \times 100\% \quad (3)$$

Where:

w_o = weight of the crucible

w_1= Weight of the crucible + Sample before incineration

w_2 = Weight of the crucible + Sample after incineration

3.4.4 Determination of Fixed Carbon

The fixed carbon of sugarcane bagasse was characterized in accordance of Debdoudi et al. (2005) using the relation below:

$$Fc = 100 - m\ ash - mvm \quad (4)$$

Where:

Fc = Fixed carbon content;

m ash = Mass of ash content

mvm = Mass of volatile matter content

3.5 Proximate Analysis of Sugarcane Bagasse

3.5.1 Extractives

6 gram of dried sugarcane bagasse was loaded into Soxhelet extractor set up using qualitative filter paper and 50 mL of acetone was used as solvent for extraction. Residence times for the boiling and rising stages was carefully adjusted to 72.3℃ operating temperature and for a 4 hour run period. After extraction, the sample was air dried at room temperature. Finally the main purpose of extraction of sugarcane bagasse in the Soxhelet was to remove none chemically bound components of sugarcane bagasse such as protein, sucrose, nitrate/nitrite, chlorophyll and waxes.

3.5.2 Characterization of Hemicelluloses

0.8 gram of extracted dried sugarcane bagasse was mixed with NaOH 120 mL of 0.1mol/L.The mixture was boiled for 3.5 hour with distilled water in water bath with operating

temperature of 73 ℃.It was filtered using a vacuum filtration and adjusted to a pH of 7 after cooling. Finally the residue was dried at a constant weight in the oven at a temperature of 105℃. The difference between the sample weight before and after this treatment was the hemicellulose content (%w/w) of dried sugarcane bagasse.

3.5.3 Characterization of Lignin

2 gram of dried extracted sugarcane bagasse was weighed and it was mixed with H_2SO_4 and 20 mL of 98% H_2SO_4 in 560 mL of distilled water. The sample was kept at 2 hours shaking time to allow for complete hydrolysis at 200 rpm.The second step of hydrolysis was made to occur in an autoclave for 1 hour at 121℃. The slurry was then cooled at room temperature. The hydrolyzed samples were filtered through vacuum filtration using a filtering qualitative filter paper. The lignin component was determined by dissolving the lignin component using H_2SO_4. The lignin content was calculated as the difference initial mass before degrading and final mass after degrading using H_2SO_4.

3.5.4 Characterization of Cellulose

The cellulose content (%w/w) was calculated by difference, assuming that extractives, hemicelluloses, lignin, ash, and cellulose are the only components of the entire biomass.

3.6 Equipment Used

The following equipment was used during the accomplishment of this work:

- ✓ Spectrophotometer: It was used to analyze and measure dye concentration and intensity of color.
- ✓ Analytical balance: It was used for weighing of the sample.
- ✓ pH meter: It was used to measure acidity and basic property of the sample.
- ✓ Stirrer: It was used for mixing of the sample preparation.
- ✓ Mesh sieves: It was used to prepare uniformly distributed size particles through mesh analysis of 2mm.
- ✓ Mortar: It was used to size and crush the bagasse sample.
- ✓ FTIR: It was used to identify the functional groups.
- ✓ X-Ray Diffraction: It was used to detect the crystalline structure of sugarcane bagasse.

- ✓ Autoclave: For hydrolysis of sugarcane bagasse to characterize lignin component.
- ✓ Muffle furnace: It was used to burn sugarcane bagasse to characterize volatile matter.
- ✓ Furnace: It was used for incineration of sugarcane bagasse to characterize ash content.
- ✓ Oven: It was used to dry the sugarcane bagasse to characterize moisture content.
- ✓ Shaker: It was used to shake the samples with the desired shaking time.
- ✓ BOD incubator: It was used to incubate or hold the BOD sample till five days.
- ✓ COD reactor: It was used to react the textile raw waste water with the CSB1500 reagent.
- ✓ Flasks: The flasks were used to hold the volume of the samples with desired concentration.
- ✓ Nanocalory spectrophotometer of macherey-nagel: It was used to measure the COD of textile raw wastewater before and after adsorption.

3.7 Adsorption Experiment

Adsorption was performed in batches. The adsorption of dye from aqueous solution of azo dye that is methyl red and waste water were taken for adsorption experiment using sugarcane bagasse as bio adsorbent. Batch one experiment was conducted with a concentration of 45 mg/L,48.75 mg/L,52.5 mg/L,56.25 mg/L and 60mg/L. Batch two experiments was accomplished with adsorbent of 1gram,1.75gram,2.5gram,3.25gram and 4gram.Batch three experiment was performed with a pH value of 4,6.5,9,11.5,and 14. Batch four experiments were carried out with time in the shaker with a shaking time of 30 minutes, 52.5 minute, 75 minute, 97.5 minute and 120 minute. The Langmuir isotherm used to describe adsorption process for different compounds. The model assumes uniform energies of adsorption onto the surface and no transmigration of adsorbate in the plane of the surface. During adsorption process when equilibrium was achieved, dye up taking capacity for each sample was calculated as follow:

$$q_e = \frac{c_o - c_e \times v}{m} \quad (5)$$

Where:

m = Mass of adsorbent (gram)

v = volume of the solution (L)

c_o = initial concentration of azo dye that is methyl red or wastewater (mg/L)

q_e= The quantity of azo dye that is methyl red or wastewater adsorbed at equilibrium (mg/g)

The initial pH value was adjusted by using either NaOH or HCl.The percentage removal of azo dye that is methyl red or wastewater was calculated by the following equation:

%Removal = $\frac{C_o - C_e}{C_o} \times 100$ (6)

Where:

C_o = Initial concentration of azo dye or waste water (mg/L)

C_e = Final concentration of azo dye or wastewater (mg/L)

3.8 Adsorption Kinetics

The kinetic study of adsorption in wastewater plays an important role because it affords important insight into the reaction pathways and into the mechanism of the reaction. Kinetic models have been proposed to explain the mechanism of a solute sorption from aqueous solution onto an adsorbent:

- ✓ Pseudo first order kinetic model.
- ✓ Pseudo second order kinetic model.

3.8.1 Pseudo-First Order Model

The pseudo first-order kinetic model has been widely used to predict the dye adsorption kinetics. The dye adsorption kinetics following the pseudo first-order model is given by (Ho and McKay, 1999a):

$$\frac{d_q}{d_t} = k_1(q_e - q_t) \quad (7)$$

Where:

k_1 (min−1) is the rate constant of the pseudo-first-order adsorption,

q_t(mg/g) denotes the amount of adsorption at time t (min) and q_e (mg/g) is the amount of adsorption at equilibrium. After definite integration by application of the conditions q_t =0 at t = 0 and $q_t = q_t$ at t = t, Equation (7) becomes:

$$\log(q_{e-}\, q_t) = \log q_e \left(\frac{k_1}{2.303}\right) \times t \quad (8)$$

By plotting $\ln(q_e - q_t)$ versus t, the adsorption rate can be calculated.

3.8.2 Pseudo Second Order Model

The adsorption kinetic data can be further analyzed using Ho'spseudo second order kinetics (McKay and Ho, 1999b, c).This is represented by:

$$\frac{d_q}{d_t} = k_2(q_{e\,-}q_t)^2 \quad (9)$$

Integrating of equation 9 and application of the conditions

q_t= 0 at t = 0 and $q_{t=}\, q_t$ at t = t gives

$$\frac{t}{q_t} = \left(\frac{1}{k_2 q_e{}^2}\right) + \frac{t}{q_e} \quad (10)$$

Where:

k_2= g/ (mgmin) is the rate constant, $k_2$and q_e can be obtained from the intercept and slope of t/q_t versus time.

3.9 Adsorption Isotherm

Adsorption equilibrium is established when the amount of solute being adsorbed onto the adsorbent is equal to the amount being desorbed. Adsorption isotherm explains the interaction between adsorbate and adsorbent and is critical for design of adsorption process. The Langmuir and Freundlich are the most frequently used models to describe the experimental data of adsorption. In the present work these two isotherms were applied to investigate the adsorption process of azo dye and textile raw waste water on sugarcane bagasse at different conditions of process parameters. The adsorption study was carried out for azo dye and textile raw wastewater concentrations varying from 45 to 60 mg/L.

3.9.1 Langmuir Isotherm Model

The Langmuir adsorption is the best model among the entire isotherm model and it is successfully applied in many adsorption processes. The Langmuir equation is given by:

$$q_e = \frac{q_m k_L c_e}{1+k_L c_e} \quad (11)$$

The linearization of it gives the following form:

$$\frac{c_e}{q_e} = \frac{1}{q_m k_L} + \frac{c_e}{q_m} \quad (12)$$

Where c_e, equilibrium azo dye or wastewater concentration, q_m and k_L are the Langmuir constants related to maximum adsorption capacity (mg/g).

3.9.2 Freundlich Isotherm Model

Freundlich isotherm model is one of the most widely used mathematical models which fit the experimental data over a wide range of concentration. This isotherm model is based on heterogeneous surface, distribution of active sites. The Freundlich equation is given by (Singh et al., 2011):

$$q_e = K_F\, c_e^{1/n} \quad (13)$$

The logarithmic form of equation:

$$\ln q_e = \ln k_F + \frac{1}{n} \ln c_e \quad (14)$$

Where:

q_e = the amount of azo dye or wastewater adsorbed after adsorption per specific amount of adsorbent (mg/g),

c_e = equilibrium concentration (mg/L),

k_F and n are Freundlich equilibrium constants

4. RESULTS AND DISCUSSIONS

Result and discussion provided the following main points of experimental result. First it deals about characterization of sugarcane bagasse and textile wastewater. Second it presents about adsorption experiment and FTIR analysis of sugarcane bagasse and XRD analysis and third it provides about experimental ANOVA analysis for azo dye and textile wastewater adsorption. Finally it deals about kinetic study.

4.1 Proximate Characterization of Sugarcane Bagasse

Proximate analysis of sugarcane bagasse was done based on ASTM D 1037 (1991) for moisture content and ASTM D 2017 (1998) for ash content, ISO 562/1974 for content of volatile matter as well as Debdoudi et al.(2005) for fixed carbon. The result of characterization was presented in table below.

Table 2: Reported proximate analysis of sugarcane bagasse

Characteristics	Value (%)
Moisture content	29
Ash content	4.2
Volatile matter	83
Fixed carbon content	12.8
Extractives	2.4
Hemicellulose	33.5
Lignin	25.5
Cellulose	34.4

The comparative proximate analysis of sugarcane bagasse based on(*Ayeni et al., 2015*) *and(Omoniyi & Olorunnisola, 2014)* provided as follow:

Table 3: Comparative proximate analysis of sugarcane bagasse

Characteristics	Value (%)
Extractives	2.14±0.6
Cellulose	35.28±1.2
Hemicellulose	33.28±0.8
Lignin	25.20±1.1
Ash	4.1±0.3
Moisture content	28 - 31
Volatile matter	69.4 - 81.7
Fixed carbon	12 - 16

4.2 Proximate Analysis of Sugarcane Bagasse

4.2.1 Extractives

Extraction was carried out to characterize the component of sugarcane bagasse of lignin, hemicelluloses and cellulose components. After extraction amount of sugarcane bagasse was 2.4 % and extraction removes none chemically bound components of sugarcane bagasse such as protein, sucrose, nitrate/nitrite, chlorophyll and waxes.

4.2.2 Hemicellulose

Hemicellulose was conducted next to extractives when the experiment was accomplished. The amount of hemicelluloses obtained was 33.5%.

4.2.3 Lignin

Lignin was the third component of sugarcane bagasse characterized. The amount of lignin obtained was 25.5 %.

4.2.4 Cellulose: Cellulose was the fourth component of sugarcane bagasse characterized and its result was 34.4%.

4.2.5 Ash content: Ash content was another proximate analysis of sugarcane bagasse and its result was 4.2%.

4.3 Characterization of Dye Based Textile Wastewater

The dye based textile wastewater was characterized before and after adsorption. The raw wastewater was characterized for the parameters of TDS (total dissolved solids), biological oxygen demand (BOD), chemical oxygen demand (COD) and pH values. The biological oxygen demand (BOD) Values of raw wastewater before adsorption was recorded from Day one up to day five and the results are provided in figure 2 below as follow:

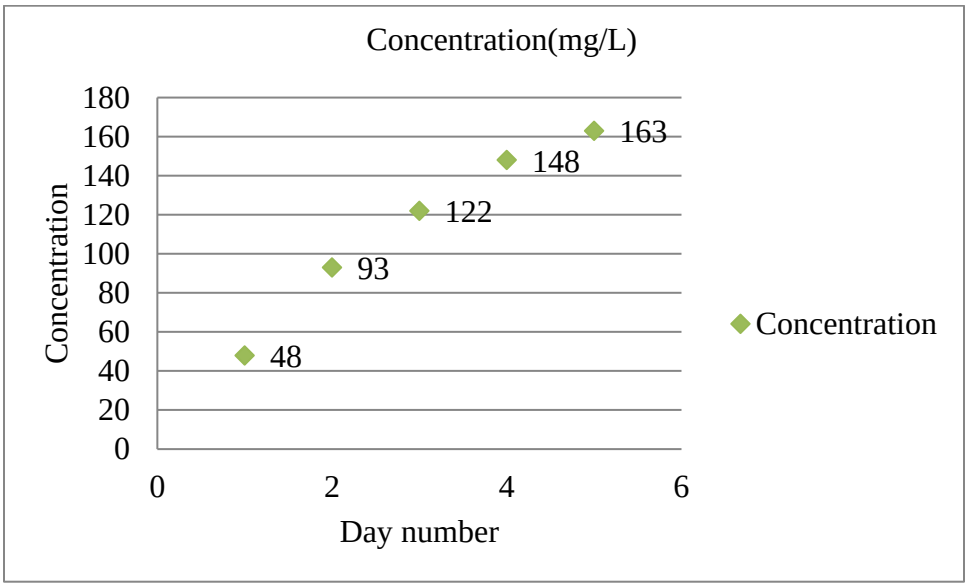

Figure 2: The biological oxygen demand (BOD) Values of raw wastewater before adsorption

The adsorption condition was accompanied with a controlling factor of concentration, adsorbent, pH and time. The raw wastewater color was cloudy red. At the adsorption condition factor of concentration, pH and time were the most significant that affects the adsorption condition. The adsorption condition was accompanied with first order kinetic Model and this shows the adsorption condition was physical adsorption. In addition to this the adsorption

condition was in the form of homogenous phase as it was compatible with Langmuir isotherm model. The quantity of wastewater treated was expressed in terms of percentage of color removal. The treated textile wastewater was a percentage of 81.The textile wastewater has the following characteristics before adsorption: The textile wastewater has high BOD value before adsorption and its maximum value was 163 mg/L. In addition to this textile wastewater has high COD value before adsorption and its value was 925 mg/L and it has high value of TDS that was 10,000 mg/L and pH of textile waste water was basic with a value of 9.65.

In addition to this BOD (biological oxygen demand) after adsorption was recorded and the results are provided in figure 3 below.

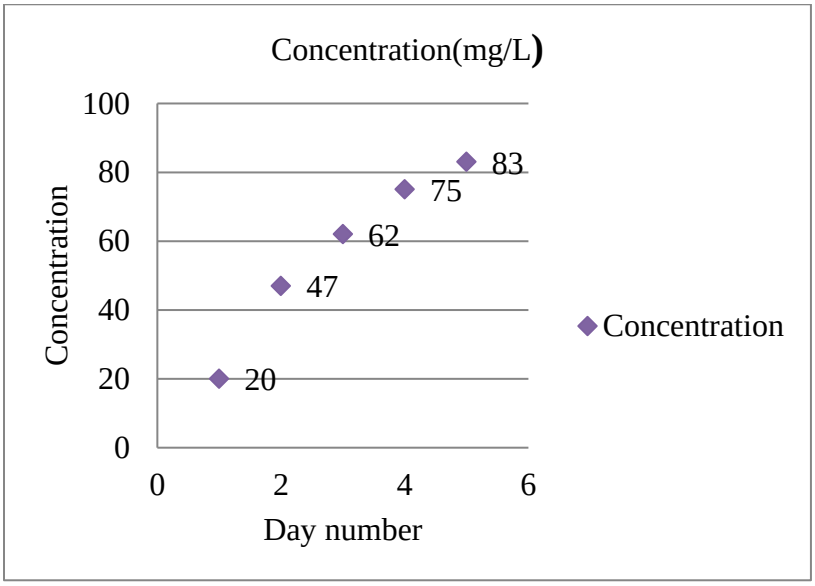

Figure 3: The biological oxygen demand (BOD) Values of raw wastewater after adsorption

In addition to this the chemical oxygen demand values wastewater was characterized before and after adsorption and the values were 925 mg/L and 50 mg/L respectively. The third parameter of textile wastewater characterized was TDS (total dissolved solids) and it accounts a value of 10,000 mg/L before adsorption and after adsorption was 8400 mg/L.Eventually the parameters of textile wastewater characterized before adsorption was pH value and it accounts 9.65 that is basic property.

4.4 Adsorption Experiment

The adsorption experiment was carried out at a parameter of pH, contact time, initial concentration as well as dose of adsorbent. The result of absorbance of standard concentration for synthetic azo dye solution and textile raw wastewater was reported and the results are indicated in fig 4 below.

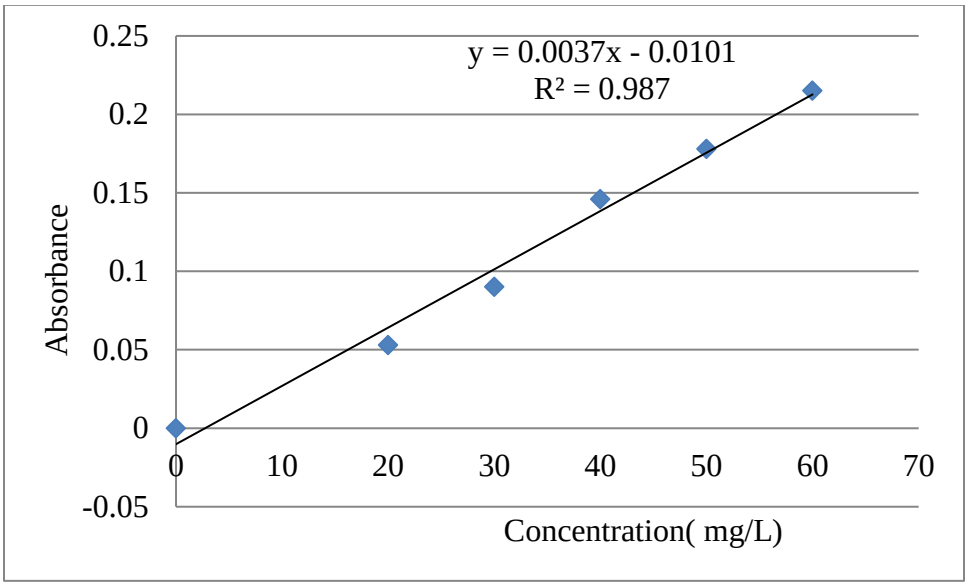

Figure 4: Calibration curve for textile wastewater and azo dye adsorption

Azo dye and textile raw wastewater was evaluated at the same operating condition that means at the same pH, contact time, initial concentration as well as dose of adsorbent. All the above parameters were applied for both azo dye solution and textile raw wastewater at the same operating conditions respectively. First adsorptions of azo dye solution were carried out then, adsorption of textile raw wastewater using sugarcane bagasse as bio adsorbent. Adsorption of azo dye solution and textile raw waste water were carried out so that to draw comparative analysis of azo dye solution and textile raw wastewater.

4.4.1 Effect of Initial Concentration for Synthetic Dye Removal

The initial concentration of azo dye (methyl red) was investigated in the range of from 45 -60 mg/L. From the experimental result it was found that concentrations of 52.5 mg/L of azo dye have higher removal efficiency.

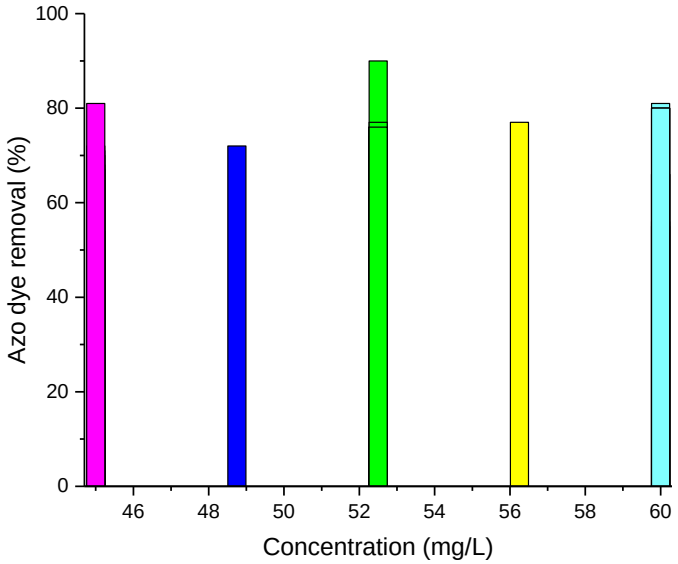

Figure 5: Effect of concentration of azo dye adsorption

From the graph we can observe that the removal percentage is somewhat nearest in range and numbers that means the removal percentage is relatively similar, and the range difference among the experiment was small. The results of percentage removal fluctuate among the run of experiment. That means there was no too much larger difference in most of the experiments, and the high percentage of azo dye removal resides with a concentration of 52.5 mg/L with a percentage removal of 90%.

4.4.2 Effect of Adsorbent for Synthetic Dye Removal

Generally the percentage of removal azo dye (methyl red) was fluctuated as the dose of adsorbent increase, and the maximum percentage removal of azo dye (methyl red) was obtained at 2.5 gram of adsorbent sugarcane bagasse as bio adsorbent. The other conditions were a factor of concentration, pH and time and these operating conditions were significant for the adsorption process. For example value of pH was highly affected for the adsorption and a pH nearest to neutral was highly effective.

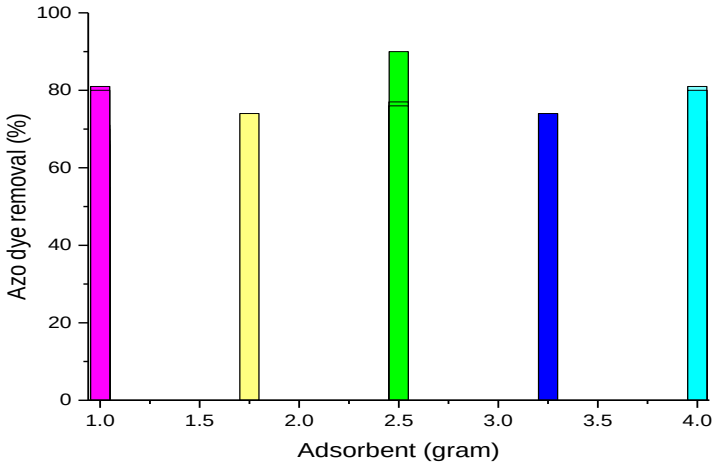

Figure 6: Effect of adsorbent on azo dye adsorption

4.4.3 Effect of pH for Synthetic Dye Removal

The adsorption was highly dependent on pH.That means pH was a controlling parameter during the adsorption process. To investigate the effect of pH on adsorption process, the adsorption experiments were conducted at a pH of 4, 6.5, 9, 11.5 and 14 using 0.1 M of HCl and 0.1 M of NaOH for adjustment of the desired pH.The higher removal of azo dye was resides at the pH value of 6.5 with a percentage removal of 90.

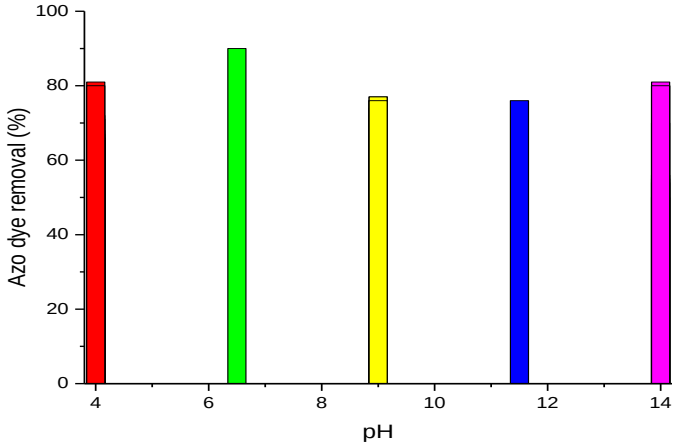

Figure 7: Effect of pH on azo dye adsorption

Effect of pH was analyzed with a factor of concentration, time and adsorbent. The factors of concentration and time were significantly affect the adsorption condition and in this case a time

of 75 minute and a concentration of 52.5 mg/L yields a maximum percentage of azo dye removal.

4.4.4 Effect of Time for Synthetic Dye Removal

The time dependent behaviour of azo dye adsorption between the adsorbent and adsorbate was studied using shaker for shaking time of different adsorption time and the times were 30 minute ,52.5 minute,75 minute,97.5 minute and 120 minute. The times required for adsorption have a great impact on adsorption that is on percentage removal of azo dye removal.

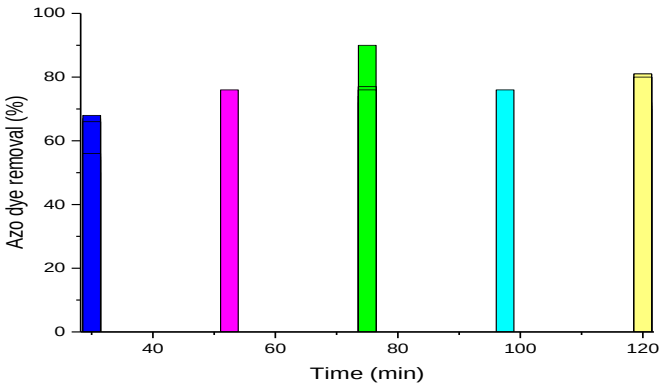

Figure 8: Effect of time on azo dye adsorption

From the graph it can be observed that the percentage of removal azo dye via adsorption using sugarcane bagasse as bio adsorbent was highly effective and the results of adsorptions were very effective. Finally the higher percentage of azo dye removal was achieved at a time of 75 minute with a percentage removal of 90.Effect of time was evaluated at a factor of concentration, adsorbent and pH.The factors of concentration and pH significantly affect the adsorption condition with concentration value of 52.5 mg/L and pH value of 6.5.Finally the factors of dose of adsorbent was not that much affect for adsorption condition. The effect of selected factors for adsorption of textile wastewater was reported as follow:

A. Effect of Initial Concentration for Removal of Color from Dye Based Textile Waste water

The removal of textile dye based wastewater was affected by the initial concentration of textile dye based waste water. The initial concentration was evaluated at a range of 45-60 mg/L. The

higher removal percentage of textile dye based wastewater was observed at a concentration of 52.5 mg/L which is 81 %.

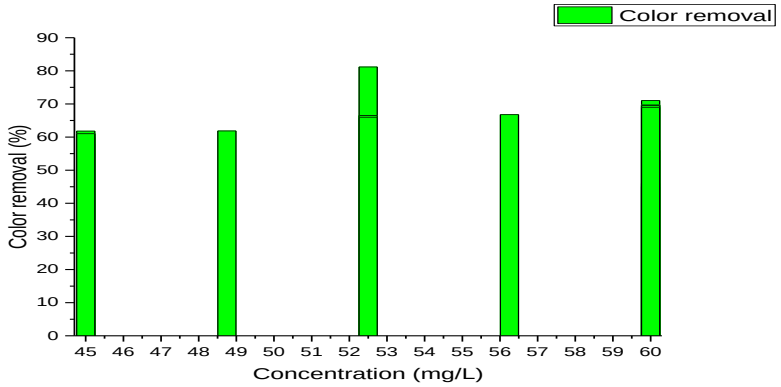

Figure 9: Effect of concentration on adsorption of textile wastewater

As it can be observed from the graph, there is no constant increment and decreasing as concentration increase, that is the percentage of removal was not linearly increasing or decreasing as initial concentration increase. Finally, the higher removal percentage was obtained at a concentration of 52.5 mg/L and the result was 81 %.

B.Effect of Adsorbent for Removal of Color from Dye Based Textile Wastewater

As it can be observed from the fig 10 below, the higher percentage removal was achieved at an adsorbent of 2.5 gram and the result was 81 %.

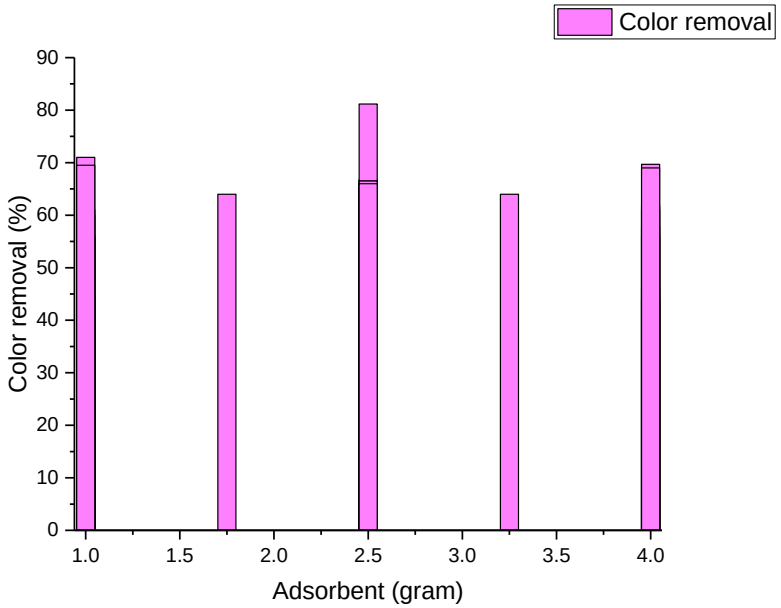

Figure 10: Effect of Adsorbent on adsorption of textile wastewater

C. **Effect of pH for Removal of Color from Dye Based Textile Wastewater**

The textile dye based wastewater was dependent on pH. pH has a significant effect on adsorption of textile dye based wastewater. Different pH values were applied on the experiment to investigate the effect of pH.The values of pH were ranged from 4-14.The maximum up take (removal) of color from textile dye based wastewater was achieved at a pH of 6.5 with a result of 81%.

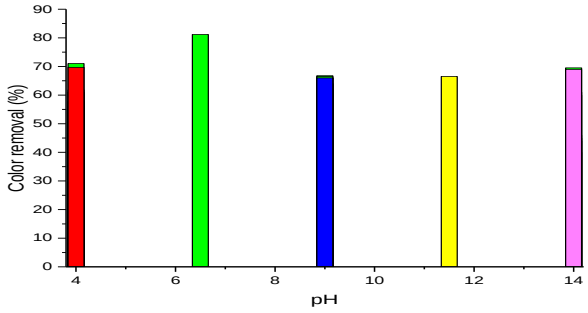

Figure 11: Effect of pH on adsorption of textile wastewater

From the graph 11 it can be observed, variation of pH values have a great effect on percentage of color removal of dye based textile wastewater. As it was observed from the graph the higher percentage removal of color from dye based textile wastewater resides at a pH of 6.5.

D.**Effect of Contact Time for Removal of Color from Dye Based Textile Wastewater**

To remove the color of textile dye based wastewater, it was investigated by applying different shaking times using sugarcane bagasse as bio adsorbent. As it was indicated by the experiment contacting time have a significant effect on adsorption of textile dye based wastewater using sugarcane bagasse as bio-adsorbent. The time allocated during the experiment was ranged from 30 upto120 minute.

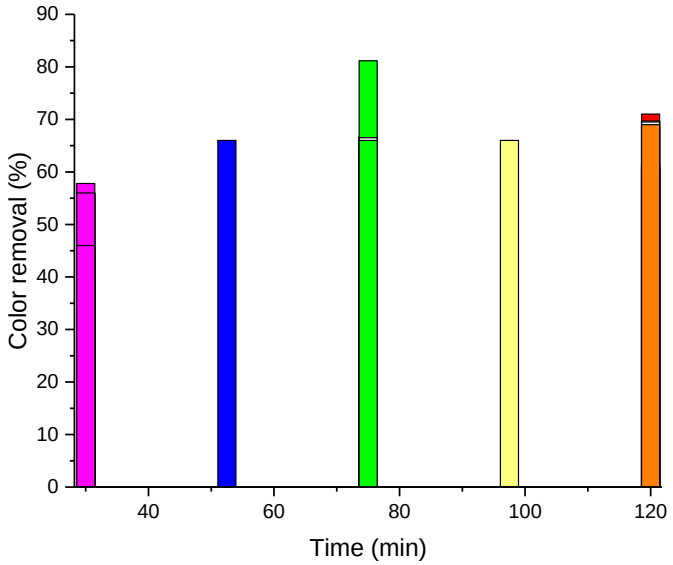

Figure 12: Effect of time on adsorption of textile wastewater

From the graph of figure 12 it was observed that, there was no constant increment and decreasing as contact time increase. That means the percentage removal of color from dye based textile wastewater was fluctuated from one point of contacting time to another. Generally the high percentage of color from dye based textile wastewater was found at a contacting time of 75 minute, with a percentage result of 81.

4.5 FTIR Analysis of Sugarcane Bagasse

FTIR analysis of sugarcane bagasse was conducted for raw sugarcane bagasse, chemically treated sugarcane bagasse, bagasse after adsorption using synthetic dye of azo dye, bagasse after adsorption of dye based textile wastewater.

Adsorption of textile wastewater depends on chemical nature of the bio-adsorbent. Hence the adsorption of textile wastewater using sugarcane bagasse as bio-adsorbent depends on the chemical nature of surface of sugarcane bagasse. That means the chemical reactivity placed on the surface of sugarcane bagasse accompanied with several functional groups have a great contribution for better adsorption of textile wastewater.

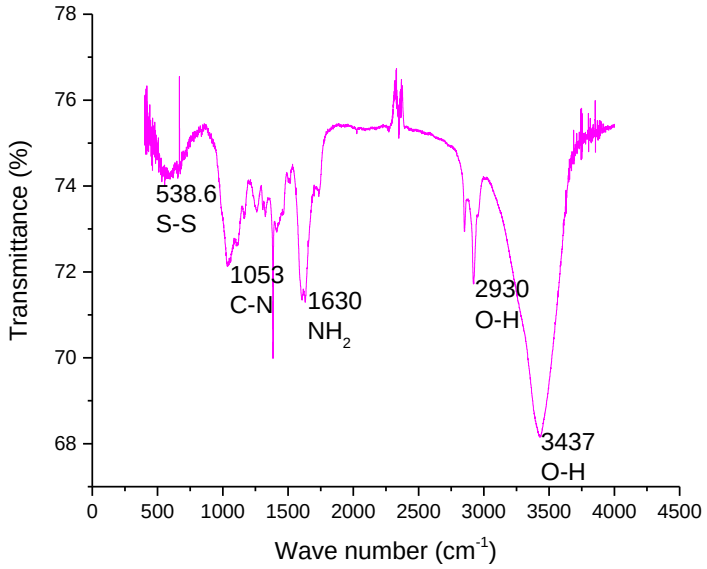

Figure 13: FTIR analysis of non treated sugarcane bagasse

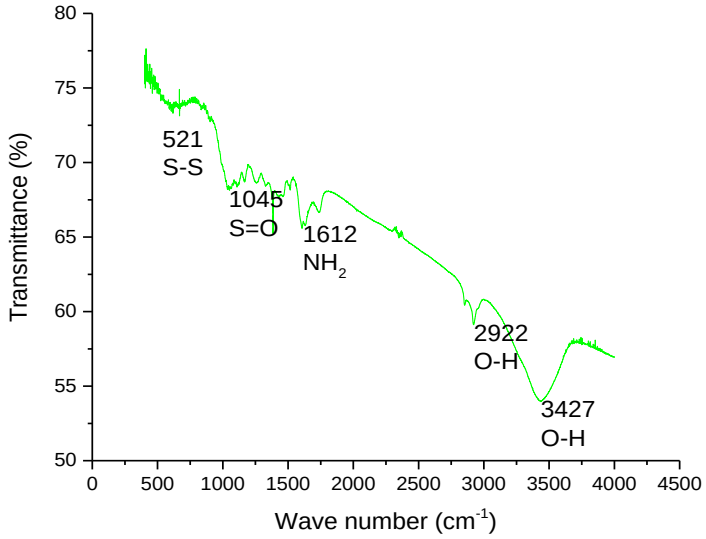

Figure 14: FTIR analysis of treated sugarcane bagasse

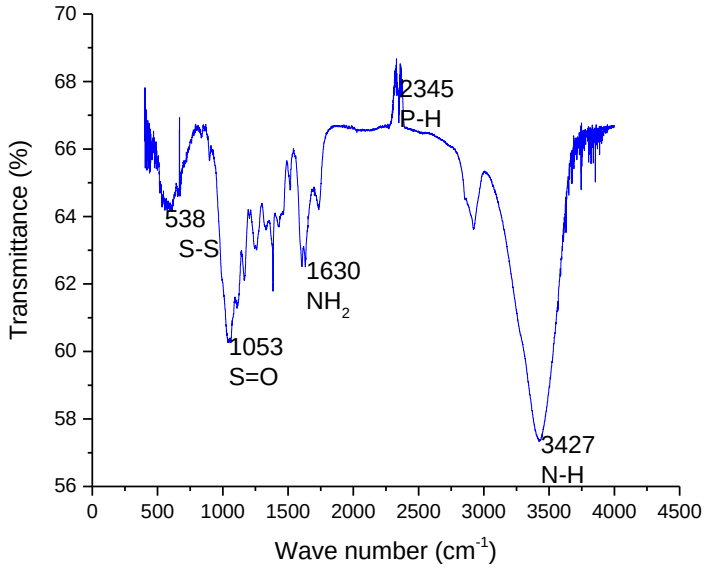

Figure 15: FTIR analysis of sugarcane bagasse after adsorption of azo dye

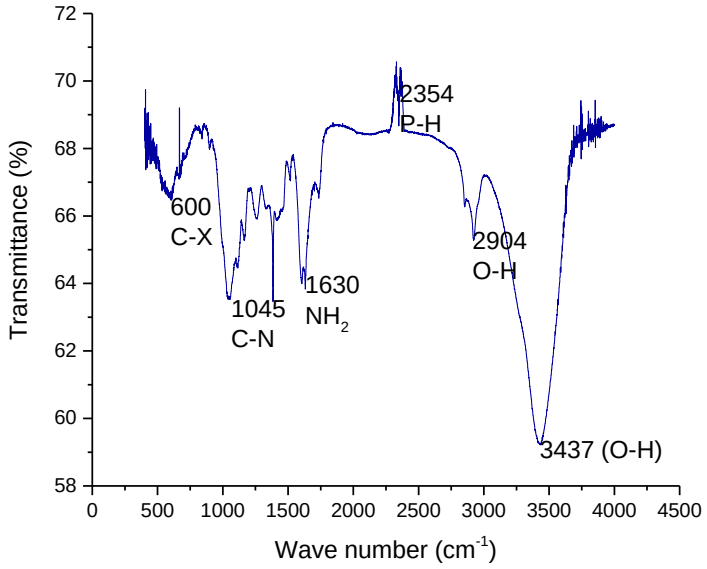

Figure 16: FTIR analysis of sugarcane bagasse after adsorption of textile wastewater

For non treated sugarcane bagasse,treated sugarcane bagasse,bagasse after adsorption of azo dye and bagasse after adsorption of textile dye based wastewater the FTIR analysis sugarcane bagasse and the functional group representations are provided as follow:

Table 4: FTIR analysis of sugarcane bagasse

Class	**Group**	**Wave number (cm^{-1})**
Hydrocarbon		
Alkane	C-H	2850 – 3000
	C-C	800 – 1000
Aromatic	C-H	3000 – 3100
	C=C	1450 – 1600
Alkenes	C – H	3080 – 3140
	C=C	1630 – 1670
Alkynes	C-H	3300 – 3320
	C-C	2100 – 2140

Oxygen compounds		
Alcohol	O -H	3300 – 3600
	C- O	1050 – 1200
Ether	C-O	1070 -1150
Aldehyde	C=O	1720 -1740
	C-H	2700 – 2900
Carboxylic acid	C=O	1700 -1725
	O-H	2500 – 3300
	C-O	1100 – 1300
Ester	C=O	1735 – 1750
	C-O	1000 – 1300 (2 band)
Kenton	C=O	1700 – 1725
Acyl halides	C=O	1785 – 1815
Anhydrides	C=O	1750; 1820 (2 band)
	O-C	1040 – 1100
Amides	C=O	1630 – 1695
	N-H	1500 - 1560

Class	**Group**	**Wave number (cm^{-1})**
Isocyanates	-N=C=O,	2100 - 2270
Isothiocyanates	-N=C=S,	
Diimides, azides	- N =C=N,	
Ketenes	$-N_3$, C=C=O	
Nitrogen compounds		
Amines	N-H	3300 – 3500
	C-N	1000 – 1250
	NH_2	1550 – 1650
	NH_2 and N-H	660 – 900
Nitrites	C≡ *N*	2240 – 2260

Oxidized nitrogen functions		
Oxime (=NOH)	O-H	3550 – 3600
	C=N	1665 ±15
	N-O	945 ±15
Amine oxide (N-O)	Aliphatic	960 ±20
	Ormatic	1250 ± 50
N=O	Nitroso	1550 ± 50
	Nitro	1530 ± 20
Alkyl bromide	C-H	667
Sulphur compounds		
Thiols	S-H	2550-2600
Esters	S-OR	700 – 900
Disulfide	S-S	500 – 540
Thiocarbonyl	C=S	1050 - 1200
Sulfoxide	S=O	1030 – 1060
Sulfone	S=O	1325 ± 25; 1140

Class	**Group**	**Wave number (cm^{-1})**
Sulfonic acid	S=O	1345
Sulfonyl chloride	S=O	1365 ± 5; 1180 ± 10
Sulphate	S=O	1350-1450
Phosphorus compounds		
Phosphine	P-H	2280-2440
		950 -1250
Phosphonic acid	(O=) PO-H	2550 – 2700
Esters	P-OR	900-1050
Phosphine oxide	P=O	1100 – 12200
Phosphonate	P=O	1230 – 1260
Phosphate	P=O	1100 – 1200

Class	Group	Wave number (cm^{-1})
Phosphor amide	P=O	1200 – 1275
Silicon compounds		
Silane		
	Si-OR	1000 -1110
	Si-CH_3	1250 ± 10

Generally the FTIR analysis of sugarcane bagasse before and after adsorption can be explained as follow: The FTIR analysis of sugarcane bagasse before adsorption provides alkyl bromides and other alkyl halides that have a C-X bond, where x is a halogen:bromine,chlorine,fluorine,or iodine as well as disulfide and at higher peak the FTIR analysis provides oxidized nitrogen functions such as oxime (=NOH).The FTIR analysis of after adsorption indicates at the lower peak the presence of alkane (C-C) and the presence of nitrogen compounds such as NH_2 and N-H and esters as well as at the higher peak the experimental result indicate the presence of nitrogen compounds such as (N-H) as well as hydrocarbons of alkyne,alkene and aromatic as well as alkane of C-H.

4.6 XRD Analysis of Sugarcane Bagasse

XRD measure the average spacings between layers or rows of atoms. XRD determine the orientation of a single crystal or grain as well as find the crystal structure of an unknown material. In addition to this it measure the size, shape and internal stress of small crystalline regions. X-ray diffraction analysis was carried out to evaluate the crystallinity degree of the raw sugarcane bagasse as indicated in the figure 17 and the higher peak indicates the crystalline cellulose region. At the lower peak of the graph there is amorphous structure.

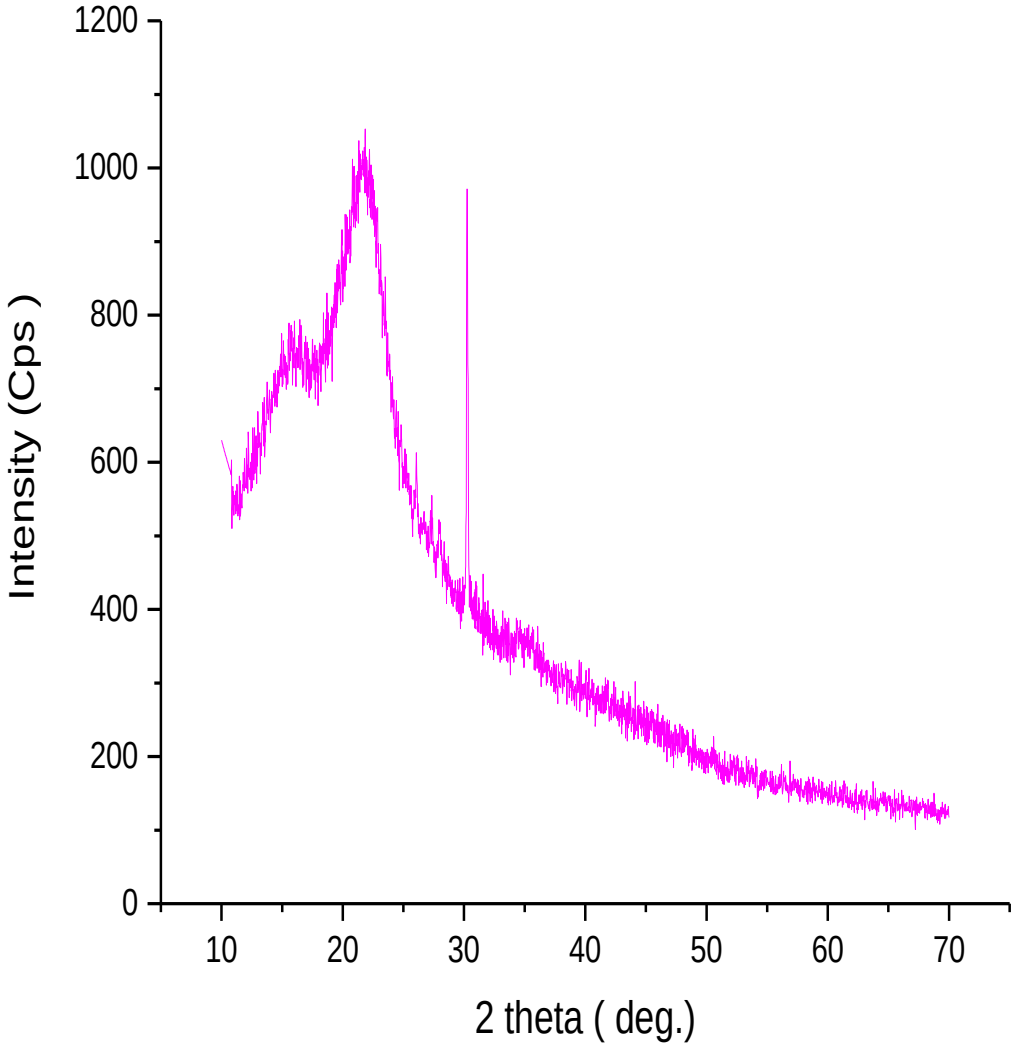

Figure 17: XRD analysis of sugarcane bagasse

4.7 Experimental ANOVA Analysis of Azo Dye Adsorption

Table 5: ANOVA analysis of azo dye adsorption

Response 1 Azo dye removal
ANOVA for Response Surface Quadratic Model
Analysis of variance table [Partial sum of squares - Type III]

Source	Sum of Squares	df	Mean Squar	F Value	p-value Prob > F	
Model	2126.68	14	151.91	11.73	0.0001	significant
A-Concentration	*68.02*	*1*	*68.02*	*5.25*	*0.0426*	
B-Adsorbent	*17.52*	*1*	*17.52*	*1.35*	*0.2694*	
C-pH	*163.88*	*1*	*163.88*	*12.66*	*0.0045*	
D-Time	*1072.06*	*1*	*1072.06*	*82.79*	*< 0.0001*	
AB	*18.06*	*1*	*18.06*	*1.39*	*0.2625*	
AC	*0.063*	*1*	*0.063*	*4.827E-003*	*0.9459*	
AD	*33.06*	*1*	*33.06*	*2.55*	*0.1384*	
BC	*14.06*	*1*	*14.06*	*1.09*	*0.3197*	
BD	*1.56*	*1*	*1.56*	*0.12*	*0.7349*	
CD	*217.56*	*1*	*217.56*	*16.80*	*0.0018*	
A^2	*22.63*	*1*	*22.63*	*1.75*	*0.2130*	
B^2	*31.06*	*1*	*31.06*	*2.40*	*0.1497*	
C^2	*83.00*	*1*	*83.00*	*6.41*	*0.0279*	
D^2	*5.34*	*1*	*5.34*	*0.41*	*0.5341*	
Residual	142.44	11	12.95			
Lack of Fit	*141.94*	*10*	*14.19*	*28.39*	*0.1451*	*not significant*
Pure Error	*0.50*	*1*	*0.50*			
Cor Total	2269.12	25				

The Model F-value of 11.73 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise.

Values of "Prob > F" less than 0.0500 indicate model terms are significant.
In this case A, C, D, CD, C^2 are significant model terms.
Values greater than 0.1000 indicate the model terms are not significant.
If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The "Lack of Fit F-value" of 28.39 implies the Lack of Fit is not significant relative to the pure error. There is a 14.51% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

Std. Dev.	3.60
Mean	71.73
C.V. %	5.02
PRESS	802.14
R-Squared	0.9372
Adj R-Squared	0.8573
Pred R-Squared	0.6465
Adeq Precision	12.471

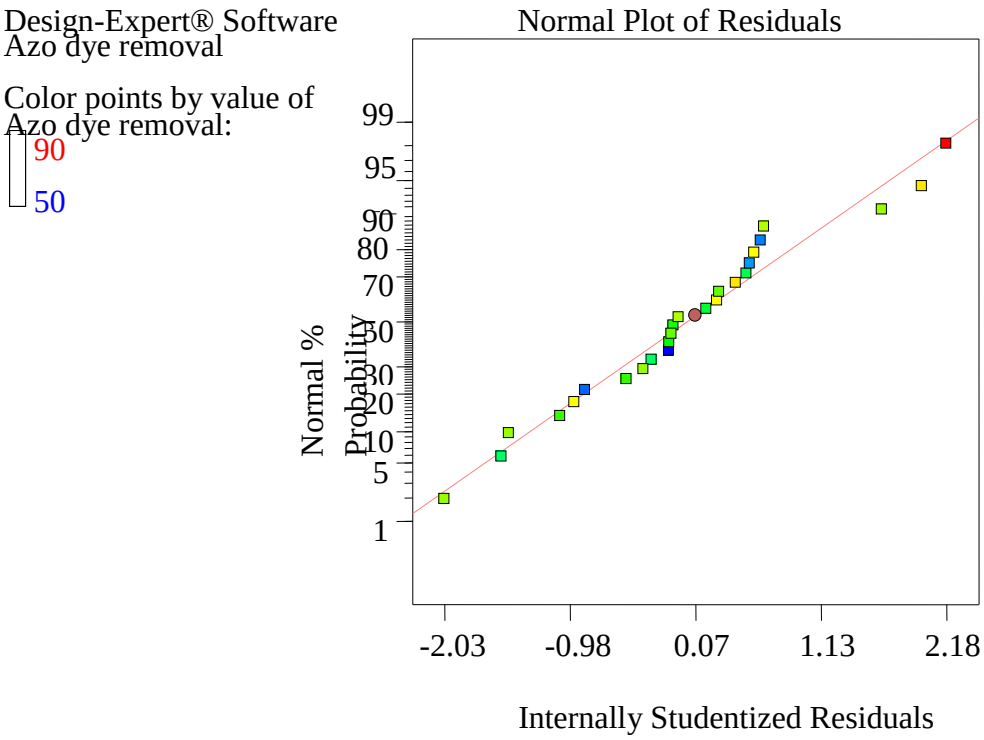

Figure 18:Normal probability plot of the studentized residuals for azo dye adsorption

Normality shows whether or not a set of experimental data is normally distributed by plotting the data against theoretical normal distribution to form straight line. Accordingly the normal probability of azo dye adsorption shown in the figure 18 displays an approximate straight line and this shows the data is good and normally distributed.AS it can be observed from the figure 18 the normal probability plot of the studentized residuals indicate that the residuals are

well normalized in the adsorption system.The residuals in the plot are well densed or compressed and this indicates the well normality of residuals.In addition to this most of the points were allocated in the line in a straight manner and this indicates that good normal probability of the experimental data.

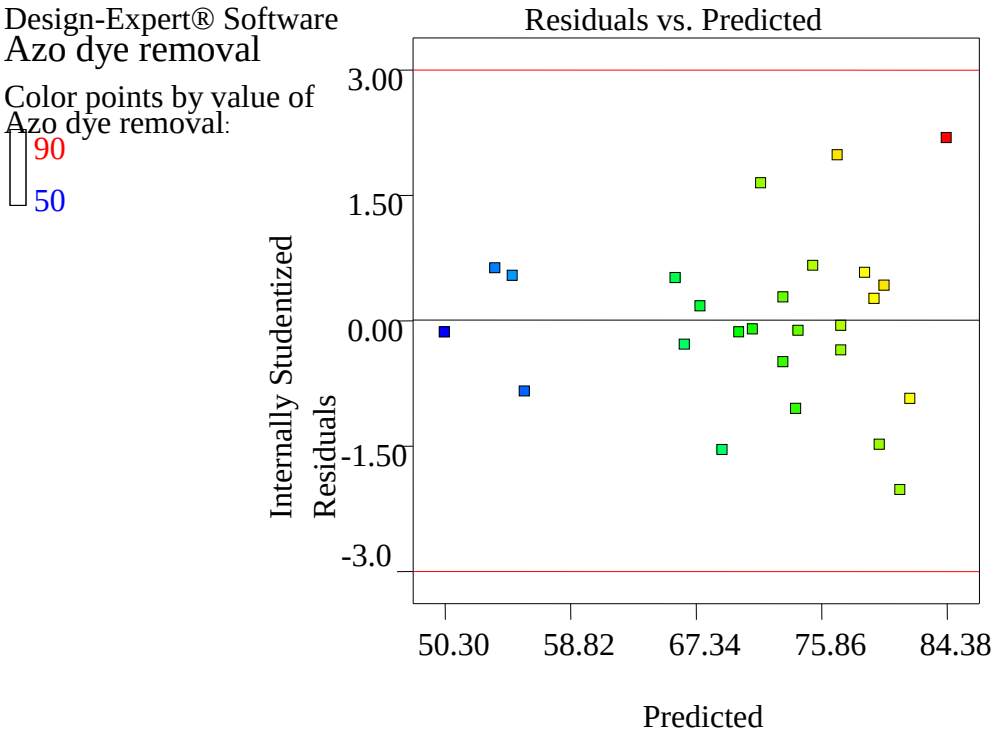

Figure 19: Studentized residuals versus predicted values for azo dye adsorption

Residual plot are used to check for all assumptions whether or not satisfied and the selected model is correct. For a well fitted model, residual plots data are scattered randomly which produces a structurles shape. Accordingly when the residual versus predicted azo dye adsorption observed from figure 19 the plotted data are well scattered randomly and it displays a structurles shape and this indicates the selected model was well fitted with the experimental data.

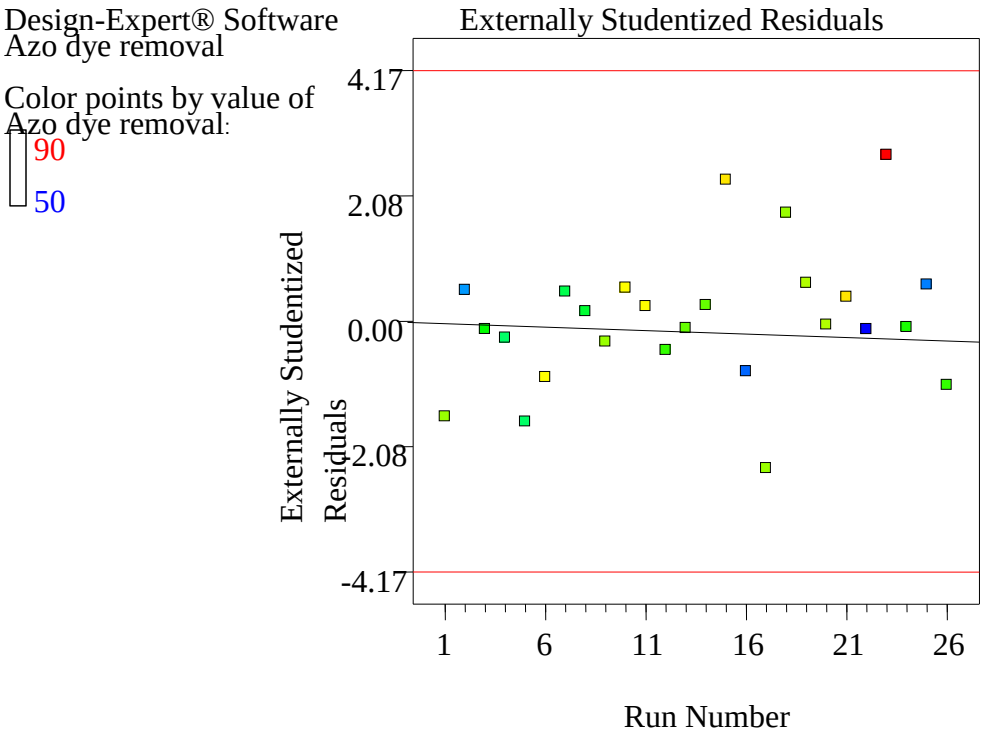

Figure 20: Externally Studentized Residuals for azo dye adsorption

The externally studentized residuals indicates the influential values and in this case the influential values resides in the run 21 with a value of concentration 52.5 mg/L,pH 6.5 and contact time 75 minute with a maximum yield of 90% azo dye removal.

From the interaction factors as it can observed from the figure 21 below the interaction of pH and time have a significant effect on adsorption of azo dye using sugarcane bagasse as bio adsorbent and in this case at a time of 75 minute and pH of 6.5 gives a maximum yield of 90% azo dye removal.In addition to this a single interaction of factors observed as indicated in the figure 21 below and the factor was pH and its value at different levels have a great effect on the adsorption system and in this case a pH value of 6.5 gives a maximum yield of azo dye removal.

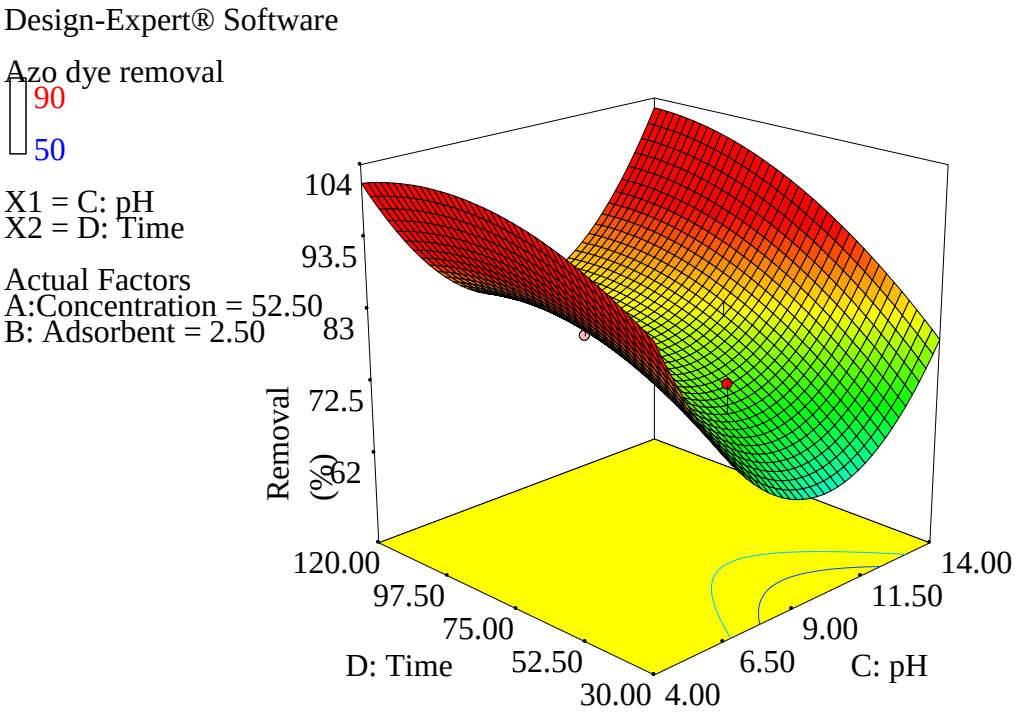

Figure 21:Interaction of CD for azo dye adsorption

As it can be observed from figure 21 for interaction of CD (pH and time) as pH decrease from 14 to 6.5 azo dye removal increase and as contact time increase from 30 minute to 75 minute azo dye removal increase and finally the maximum azo dye removal was achieved at pH 6.5 and time of 75 minute to a percentage removal of 90.

The effect of initial concentration, dose of adsorbent, pH and time was analyzed. To optimize different parameters for adsorption of azo dye using sugarcane bagasse as bio adsorbent quadratic model was used. Here effect of parameters on the response was examined.26 experimental conditions at two centre point and 1 replicate with $\alpha = 0.5$ were carried out. Experimental result of percentage removal of azo dye was fed to design expert 7 using quadratic model. As it was observed from ANOVA the quadratic model was the most compatible model to describe the effect of selected factors for adsorption of azo dye using sugarcane as bio adsorbent. To explain the adequacy of model, R-squared and lack of fit were used. As R^2 is nearest to 1, then the model is well compatible to describe the response as a function of the selected factors. The value of R^2 is 0.9372 that is 93.72% the data used was fitted.

Adeq precision measured the signal to noise ratio and in this case a ratio greater than 4 is desirable and in the case of this the signal to noise ratio is 12.471 indicates an adequate signal. From the ANOVA result the model F-value of 11.73 implies the model is significant. There is only a 0.01% chance that a model F-value this large could to occur due to noise. The p value is used to quantify whether or not F is a large enough to show statistical significance. Values of probability >F less than 0.05 indicate model terms are significant where as values of greater than 0.1 shows the model terms are not significant. The ANOVA result provided p-values of each model terms. Based on the result the single factors A, C, D (concentration, pH and time) and the interaction factors C^2, and CD (interaction between time and pH as well as interaction of pH itself) are significant in the model. The ANOVA also indicates lack of fit and in this case lack of fit was 0.1451 and it was not significant. Diagnostic plots were carried out with normal probability versus studentized residuals to check for normality of residuals. Plot of studentized residuals versus predicted values to check for constant error. Plot of externally studentized residuals were also carried out to look for outline of experimental run that is influential values.

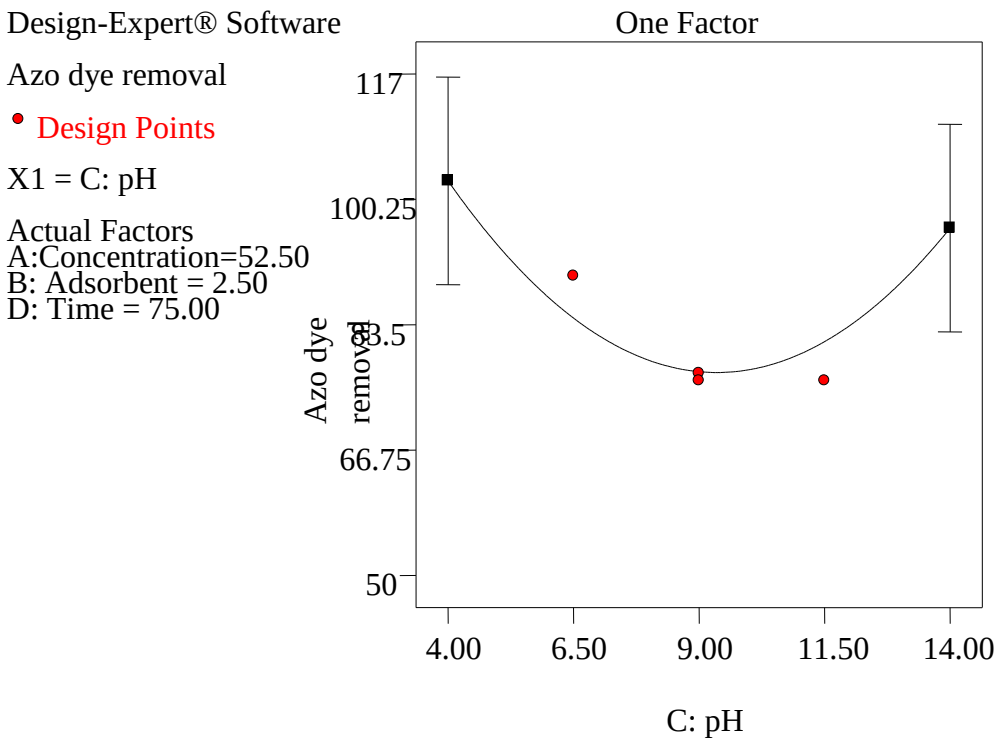

Figure 22: Interaction effect of pH for azo dye adsorption

The graph indicated in the figure 22 shows a single interaction of pH as indicated from ANOVA analysis. Based on ANOVA from the selected factor except pH the rest factors were not have a single interactive effect on adsorption. Therefore only a pH of single interaction was indicated by graph.

4.7.1 Effect of Selected Factors for Azo Dye Removal

The response of the experiment was affected by single factor and interactive variables. In this case concentration, pH and contact time affects significantly and from the interactive factor, effect of between Concentration and pH as well as between pH values significantly affects the response. As indicated in the figure 23 below perturbation plot is an important plot to show the effect of all factors at a point in design space. One factor is changing while other factors kept constant to represent the response in the plot. When a response variable is sensitive to the respective factors the shape displays steepy slope or curvature for a selected factor. In contrast to this non sensitive response to the selected factor is represented by a flat line. According to the plot of perturbation 3 of the selected factors have a positive effect on azo dye removal. As it can be seen from the plot of perturbation the steepy slope or curvature of D and C as well as D and A was observed which shows the response is well sensitive for C, A and D factors namely concentration, pH and time.

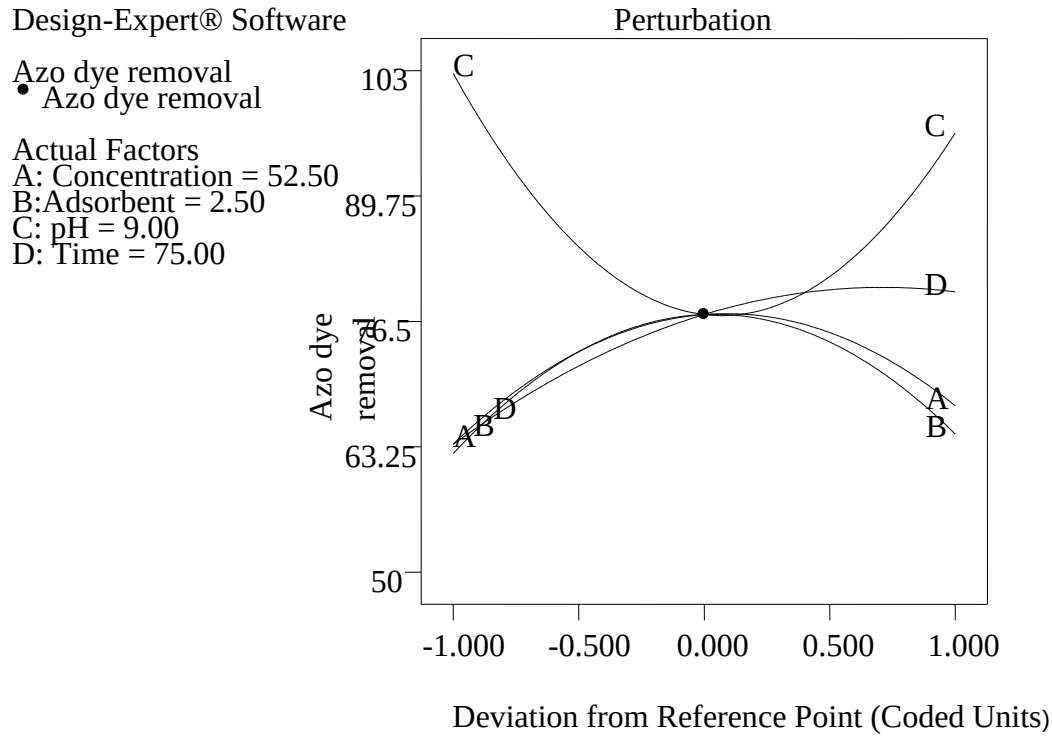

Figure 23: Perturbation plot depicting the effect of operational factor for Adsorption of azo dye using sugarcane bagasse as bio adsorbent.

4.7.2 Conclusion for Selected Model and ANOVA Analysis of Azo Dye Removal

To draw conclusion the model was applied at CCD (central composite design) and the selected model was quadratic model. The selected factors were applied at a desired operating condition to examine their significance. Based on this the quadratic model best compatible to explain the adsorption of azo dye using sugarcane bagasse as bio-adsorbent and it magnifies that concentration, pH and contact time were significant for the operation. The significant interactive effects were between time and pH as well as pH at different values. Finally the selected model was quadratic model and the model was valid to explain the adsorption of azo dye on to sugarcane bagasse.

4.8 Experimental ANOVA Analysis of Textile Wastewater Adsorption

Table 6 :ANOVA analysis of textile wastewater adsorption

Response 1 Color removal
ANOVA for Response Surface Quadratic Model
Analysis of variance table [Partial sum of squares - Type III]

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	2111.64	14	150.83	15.86	< 0.0001	significant
A-concentration	*110.32*	*1*	*110.32*	*11.60*	*0.0059*	
B-adsorbent	*2.72*	*1*	*2.72*	*0.29*	*0.6037*	
C-pH2	*30.39*	*1*	*230.39*	*24.22*	*0.0005*	
D-time	*926.28*	*1*	*926.28*	*97.39*	*< 0.0001*	
AB	*4.40*	*1*	*4.40*	*0.46*	*0.5105*	
AC	*3.99*	*1*	*3.99*	*0.42*	*0.5305*	
AD	*54.06*	*1*	*54.06*	*5.68*	*0.0362*	
BC	*1.46*	*1*	*1.46*	*0.15*	*0.7027*	
BD	*3.31*	*1*	*3.31*	*0.35*	*0.5671*	
CD	*137.35*	*1*	*137.35*	*14.44*	*0.0029*	
A^2	*31.40*	*1*	*31.40*	*3.30*	*0.0965*	
B^2	*32.56*	*1*	*32.56*	*3.42*	*0.0913*	
C^2	*98.20*	*1*	*98.20*	*10.33*	*0.0083*	
D^2	*6.35*	*1*	*6.35*	*0.67*	*0.4311*	
Residual	104.62	11	9.51			
Lack of Fit	*104.45*	*10*	*10.45*	*63.98*	*0.0970*	*not significant*
Pure Error	*0.16*	*1*	*0.16*			
Cor Total	2216.26	25				

The Model F-value of 15.86 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise.

Values of "Prob > F" less than 0.0500 indicate model terms are significant.
In this case A, C, D, AD, CD, C^2 are significant model terms.
Values greater than 0.1000 indicate the model terms are not significant.
If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The "Lack of Fit F-value" of 63.98 implies there is a 9.70% chance that a "Lack of Fit F-value" this large could occur due to noise. Lack of fit is bad -- we want the model to fit. This relatively low probabiity (<10%) is troubling.

Std. Dev.	3.08
Mean	61.33
C.V. %	5.03
PRESS	441.39
R-Squared	0.9528
Adj R-Squared	0.8927
Pred R-Squared	0.8008
Adeq Precision	14.791

The effect of initial concentration,dose of adsorbent,pH and time was analyzed.To optimize different factors for adsorption of dye based textile wastewater using sugarcane bagasse as bioadsorbent quadratic model was used.Here effect of factors on the response was examined.26 experimental conditions and one replicate with two center point at $\alpha = 0.5$ were carried out.The experimental result of percentage color removal of dye based textile wastewater was fed to design expert 7 using quadratic model.As it was observed from ANOVA analysis the quadratic model was the most compatible model to describe the effect of selected factors for adsorption of dye based textile wastewater using sugarcane bagasse as bio adsorbent.To explain the adequacy of model R-squared and lack of fit were used.As R^2 is nearest to one ,then the model is well fitted to describe the response as function of the selected factors.In this case as it can be seen from ANOVA analysis the value of R^2 is 0.9528 that is 95.28% and it shows the data used was fitted about 95.28% with the operating condition.

Adeq precision measured the signal to noise ratio and in this case a ratio greater than 4 is desirable and in the case of this the signal to noise ratio was 14.791 and it indicates an adequate signal. As it can be seen from the ANOVA result the model F-values of 15.86 implies the model was significant.There was only a 0.01% chance that a model F-value this large could occur due to noise.The p-value was used to quantify whether or not F was a large enough to show statistical significance.Values of probability >F less than 0.05 indicate the model terms were significant.Where as values of greater than 0.1 indicate the model terms were not significant.The ANOVA result provided p-values of each model terms.Based on the result three of the factors namely A,C and D (concentration,pH and time) and from the interaction factors there were interaction effect namely AD,CD and C^2 (interaction between concentration and time,time and pH as well as interaction of pH) were significant model terms in the operating conditions.The

ANOVA result also indicate lack of fit and in this case lack of fit F-values of 63.98 implies the lack of fit is not significant relative to the pure error.There is a 9.7 % chance that a lack of fit value this large could occur due to noise.Non significant lack of fit is good so that the model to fit.Diagonistic plots were carried out with normal probability versus studentized residuals to check for normality of residuals.A plot of studentized residuals versus predicted values to check for constant error.A plot of externally studentized residuals were also carried out to look for out line of experimental run i.e.influential values.

4.8.1 Effect of Selected Factors for Textile Wastewater Removal

The response of the experiment was affected by single factor and interactive variables.In this case concentration, pH and time from the single factor and from the interactive factor,an interaction between concentration and time,an interaction between pH and time as well as an interaction between pH values significantly affects the operating condition with the selected model.

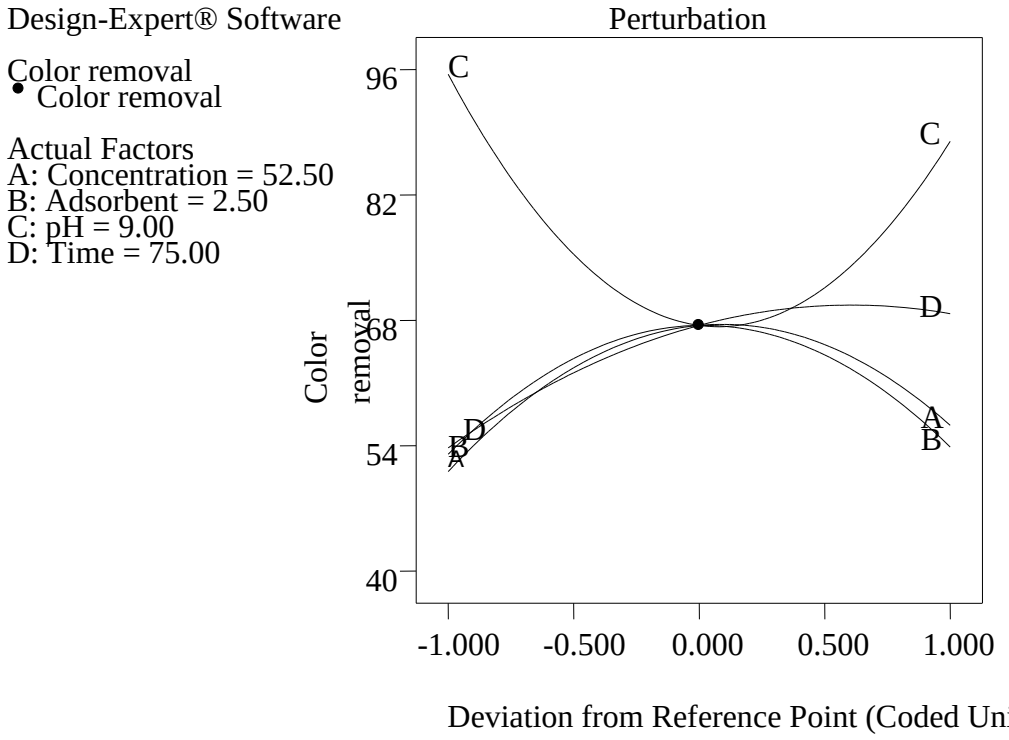

Figure 24: Perturbation plot depicting the effect of operational factor for Adsorption of textile wastewater using sugarcane bagasse as bio adsorbent.

As shown in the above figure 24 Perturbation plot is an important plot to show the effect of all factors at a point in design space. One factor is changing while other factors kept constant to represent the response in the plot. When a response variable is sensitive to the respective factor the shape displays steepy slope or curvature for a selected factor. In contrast to this non-sensitive response to the selected factor is represented by a flat line. Accordingly to the plot of perturbation three of the selected factors have a positive effect on color removal of textile wastewater. As it can be seen from the plot of perturbation the steepy slope or curvature of D and A as well as C and D as it is observed which shows the response is well sensitive for A,C,and D factors namely concentration, pH and time.

In contrast to this a flat line is observed with the lines corresponds with B which shows the response is non sensitive for B factor that is dose of adsorbent. The perturbation plot support the ANOVA result in suggesting of significant factors for operating condition as it was observed.

4.8.2 Conclusion for Selected Model and ANOVA Analysis of Textile Wastewater Removal

To draw conclusion the model was applied at CCD (Central composite design) and the selected model was quadratic model. The selected factors were applied at desired operating condition to examine their significance. Based on this the quadratic model best compatible to explain the adsorption of textile wastewater using sugarcane bagasse as bio adsorbent and it magnifies concentration, pH and time were significant for the operating condition.

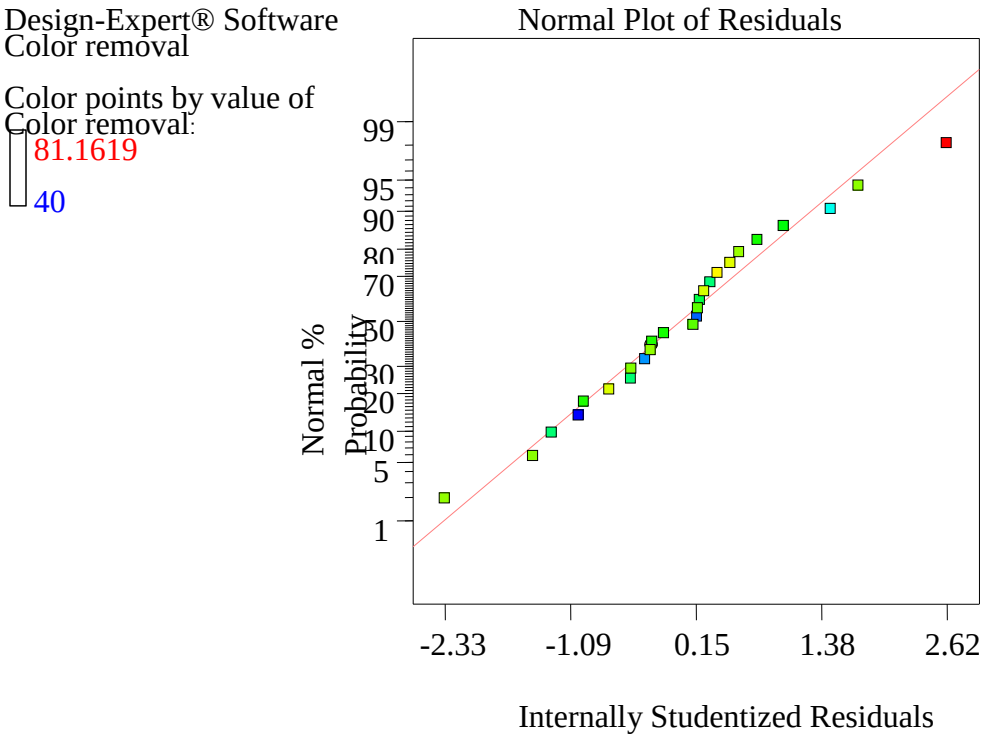

Figure 25: Normal probability plot of the studentized residuals

The normal plot of residuals deals about normality assumption. Normality shows whether or not a set of experimental data is normally and well distributed and this one is represented or demonstrated by plotting the data against the theoretical in order to form a nearest straight line. To say good experimental data, if the residual data points allocated along the straight line. Accordingly the normal probability indicated in the figure 25 above, the residual data points are approximately allocated on the straight line and this shows a good experimental data.

As it can be observed from the figure 25 normal probability plot of the studentized residuals indicate that the residuals are well normalized in the adsorption system.The residuals in the graph are well densed or compressed along the straight line and this indicates the well normality of residuals data.

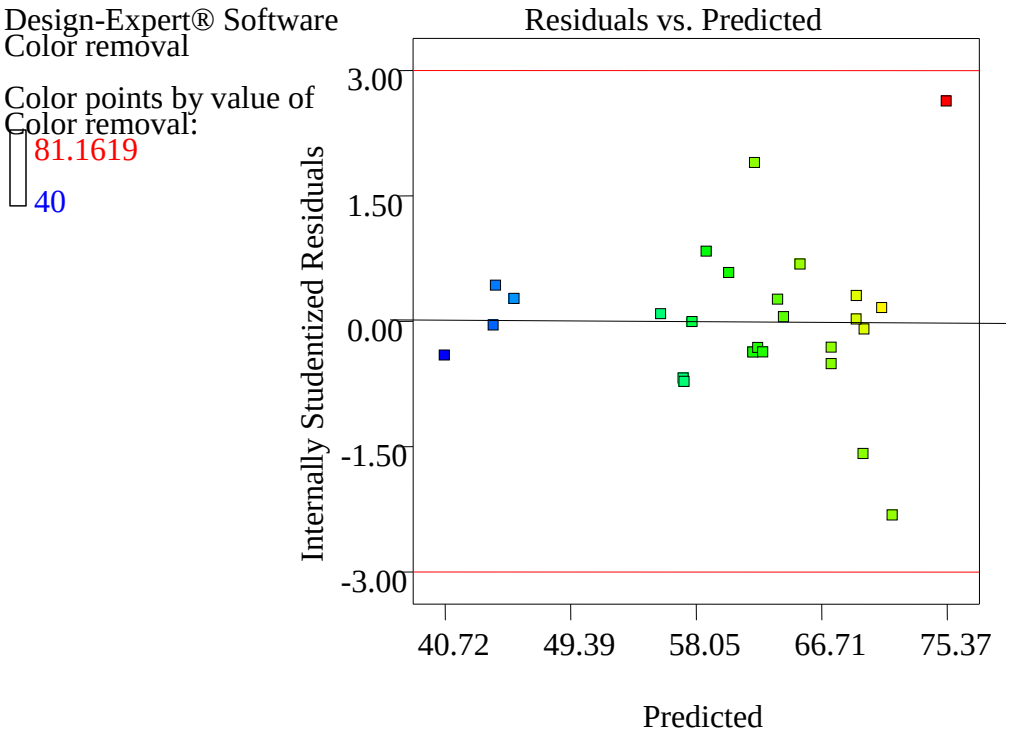

Figure 26: Studentized residuals versus predicted values

The residual plots are used to check that all the assumption of a particular design of experiment is achieved and the model is correct. For a well fitted model, residual plot data are scattered and distributed properly in a random manner and this produce a structure less shape.Accordinglly as it can be seen from figure 26 the residual plot data are well scattered and distributed in a random manner and this produces a structure less pattern and this shows the selected model was well fitted with the experimental data.

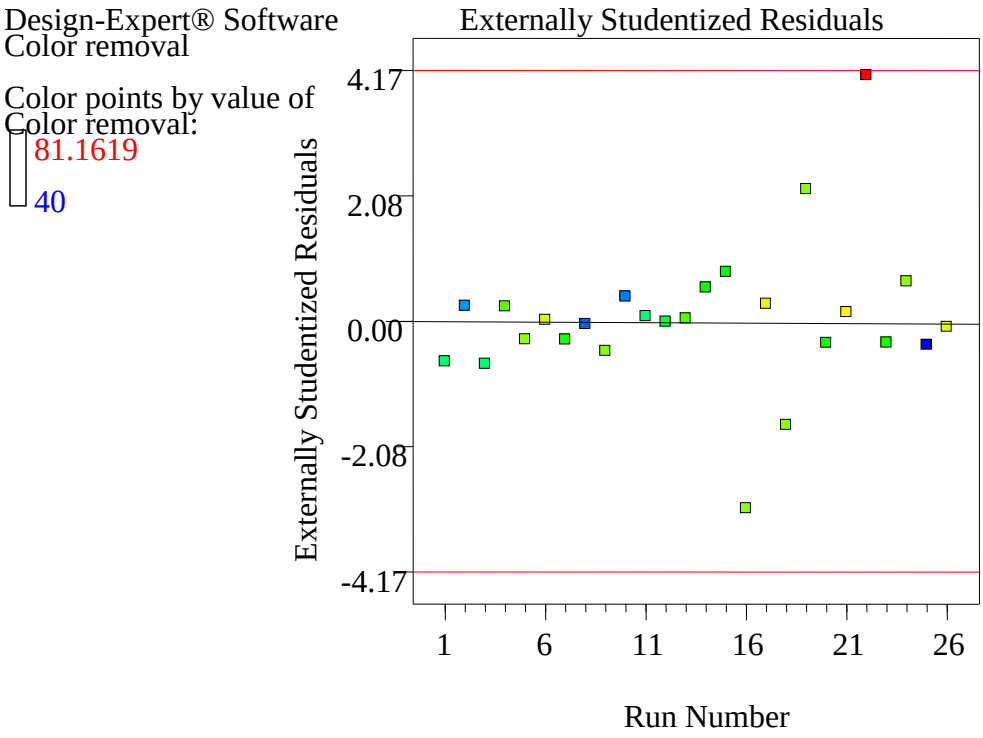

Figure 27:Externally Studentized Residuals

As it can be observed from the figure 27 above the externally studentized residuals indicates the influential values and in this case the influential values resides in the run 21 with a values of concentration 52.5 mg/L,pH 6.5 and contact time 75 minute with a maximum yied of 81.16% color removal of textile wastewater.

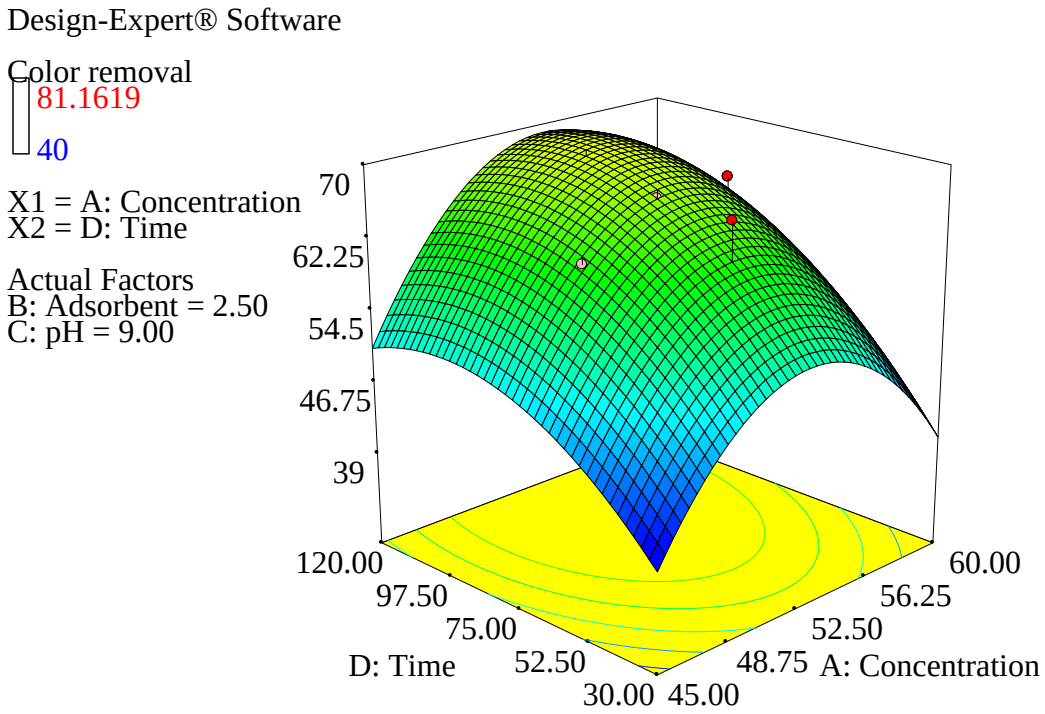

Figure 28: Interaction for AD

As it can be observed from the graph of figure 28 interaction for AD ,the interaction of time and concentration have a significant effect on adsorption of textile wastewater using sugarcane bagasse as bio adsorbent and in this case at time of 75 minute and concentration of 52.5 mg/L gives a maximum yield of 81.16% color removal. As it can be seen from figure 28 as time increase the percentage of removal textile wastewater color increase and as initial concentration increase the percentage of textile wastewater removal increase. In addition to this interaction of CD is shown below in the figure 29 and the interaction magnifies there is a significant effect on adsorption of textile wastewater using sugarcane bagasse as bio adsorbent and in this case pH of 6.5 and time 75 minute gives a maximum yield with a value of 81.16% of color removal.

Generally the shape of the graph of figure 28 shows a maximum value of color removal of textile wastewater and the maximum value resides at a concentration of 52.5 mg/L and contacting time of 75 minute with a percentage removal of textile wastewater color 81.16.

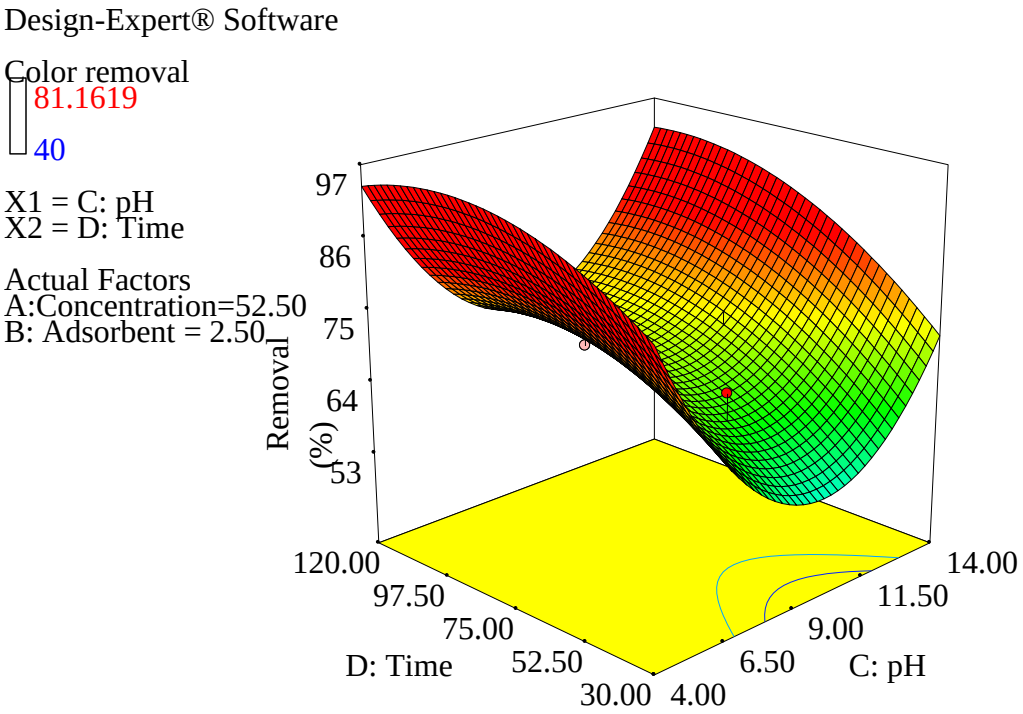

Figure 29: Interaction for CD

As it can be seen from figure 29 for interaction of CD as time increase from 30 minute to 75 minute percentage of removal textile wastewater color increase, and in contrast as pH decrease from 14 to 6.5 the percentage removal of textile wastewater color increase, and finally the maximum yield was obtained at pH 6.5 and time 75 minute with a percentage removal of textile wastewater color 81.16.

As it can be observed from the figure 30 the factor of pH at different values have a great effect on the adsorption system and in this case a pH value of 6.5 gives a maximum yield with a value of 81.16% of color removal.

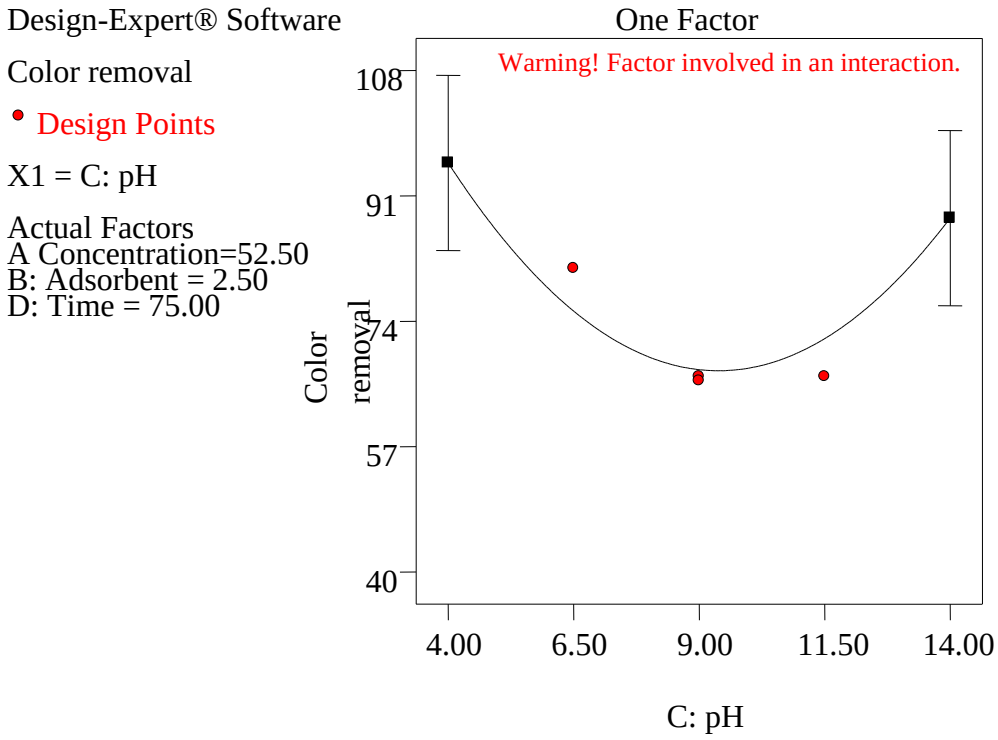

Figure 30: Interaction for C^2

4.9 Kinetic Study

The kinetic study is helpful in prediction of adsorption rate constants, equilibrium adsorption capacity and adsorption mechanism *(Bousba & Hassen, 2014)*. The capability of pseudo-first-order PFO and pseudo-second-order PSO kinetic models were examined in this study at one initial concentration of azo dye and textile wastewater (52.5 mg/L) and at constant temperature of 25°C. The linear form of pseudo first-order model of Lagergren is generally expressed as follows:

$$\ln(q_e - q_t) = \ln(q_e) - k_1 t \quad (1)$$

where q_e and q_t are the adsorption capacity of azo dye and textile wastewater at equilibrium and at time t (min) respectively and k1 is the PFO rate constant (min−1). K_1 and q_e values were determined from the slope and intercept of the plots of ln (q_e−q_t) versus t.

The linear form of PSO model is expressed as follows:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^{\,2}} + \frac{1}{q_e} \qquad (2)$$

Table 7: The PFO and PSO kinetic parameters for the adsorption of azo dye on to sugarcane bagasse

Pseudo first-order model PFO

Co (mg/L)	q_e exp (mg/g)	k_1 (min^{-1})	Intercept (log qe)	R^2
52.5	18	0.1	6.492	0.908

Pseudo second-order model PSO

K_2 (g mg^{-1} min^{-1})	Intercept (log qe)	R^2
0.49	33.65	0.884

As it can be observed from the table 7 above the pseudo first order and pseudo second order model the experimental data was well fitted with the pseudo first order model with R^2 = 0.908 and this experimental result indicate that pseudo first order model was highly fitted to explain the adsorption of azo dye on to sugarcane bagasse. The second adsorption was adsorption of textile wastewater on to sugarcane bagasse and the kinetic experimental result showed that pseudo first order model was highly compatible with the experimental result with R^2 = 0.982 and finally the

experimental data was well fitted for pseudo first order model, as a result this model was well enough to explain the adsorption of textile wastewater on to sugarcane bagasse.

Table 8: The PFO and PSO kinetic parameters for the adsorption of textile wastewater on to sugarcane bagasse

Pseudo first-order model PFO				
Co (mg/L)	q_e exp (mg/g)	k_1 (min^{-1})	Intercept (log qe)	R^2
52.5	20	0.083	5.752	0.982
Pseudo second-order model PSO				
K_2 (g mg^{-1} min^{-1})	Intercept (log qe)	R^2		
0.336	24.89	0.786		

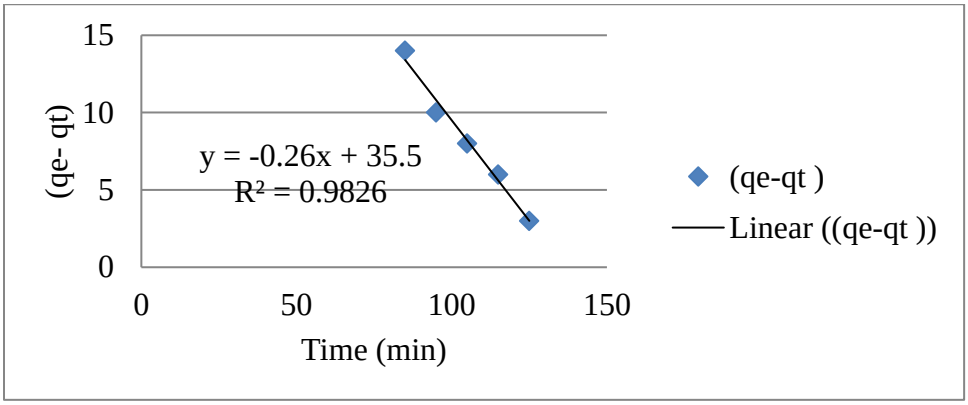

Figure 31: Pseudo first order plot for adsorption of textile waste water using sugarcane bagasse as bio adsorbent

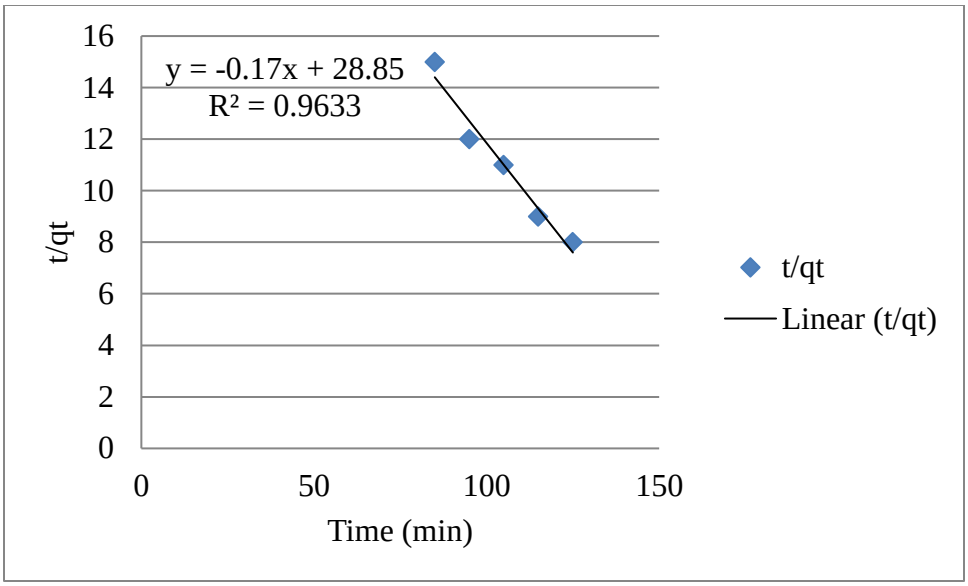

Figure 32: Pseudo second order plot for adsorption of textile waste water using sugarcane bagasse as bio adsorbent

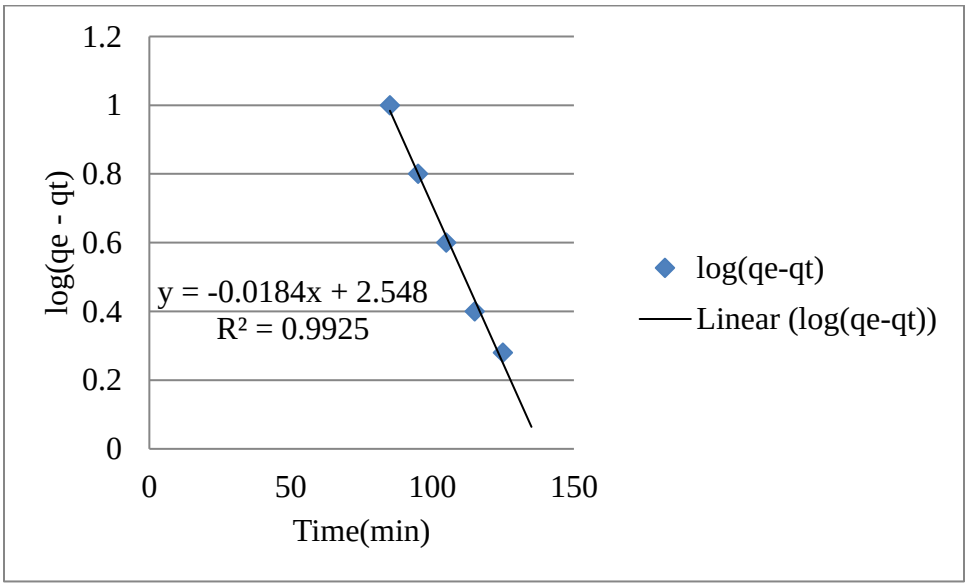

Figure 33: Pseudo first order plot for adsorption of azo dye using sugarcane bagasse as bio adsorbent

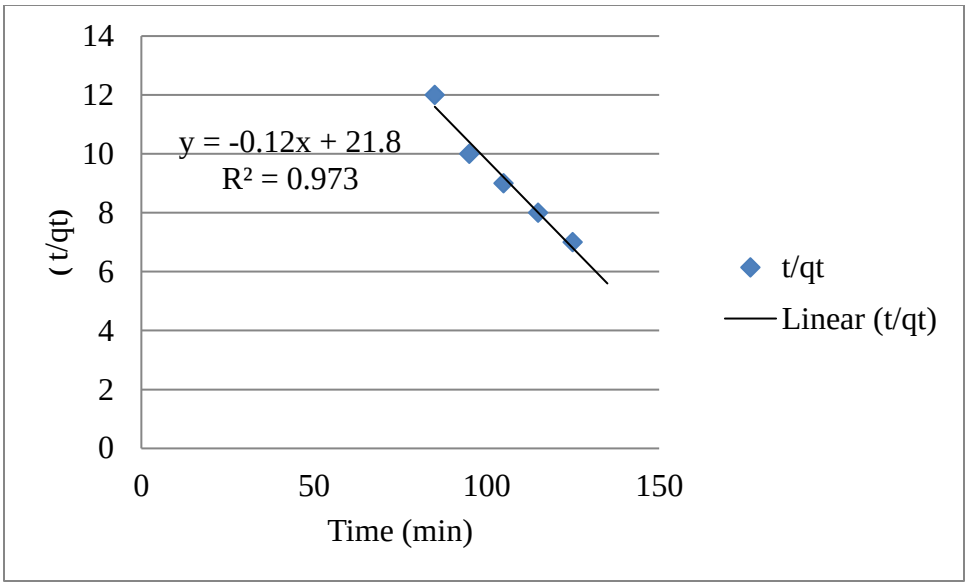

Figure 34: Pseudo second order plot for adsorption of azo dye using sugarcane bagasse as bio adsorbent

4.9.1Adsorption Isotherm

The equilibrium data for the adsorption are commonly known as adsorption isotherms. It is essential to know them so as to compare the effectiveness of different adsorbent materials under different operational conditions and also to design and optimize an adsorption system. Adsorption of azo dye and dye based textile wastewater were applied for adsorption isotherms. In this study, two isotherms models were used, Langmuir and Freundlich. As it indicated below the figure 35 and figure 36 the gab of correlation coefficient among the two models were not that much large and the Langmuir isotherm model was well fitted for adsorption of azo dye using sugarcane bagasse as bio adsorbent. The Langmuir correlation coefficient (R^2) shows the Langmuir model was well fitted about 95.9% with the adsorption system of azo dye using sugarcane bagasse as bio adsorbent.

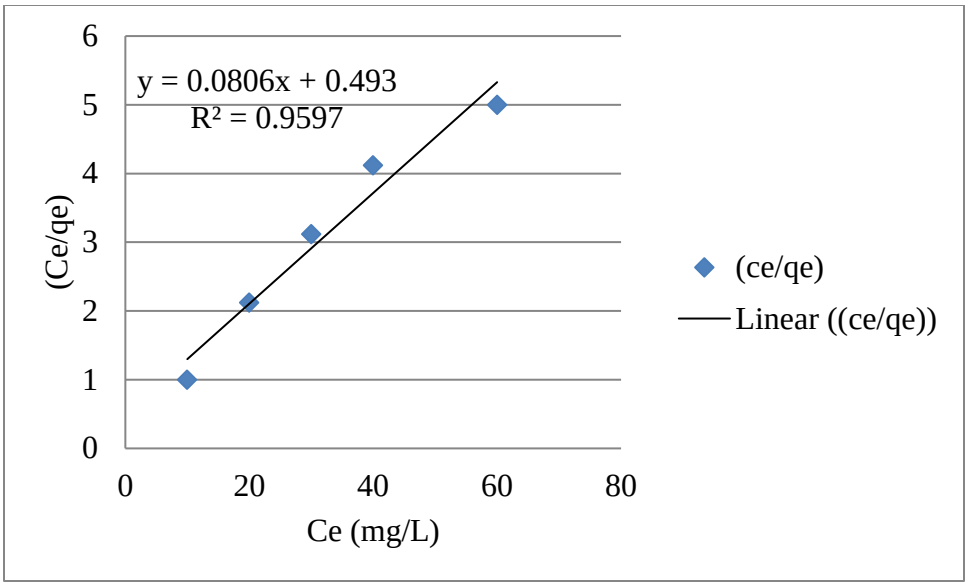

Figure 35: Langmuir adsorption isotherm for azo dye adsorption using sugarcane bagasse as bio adsorbent

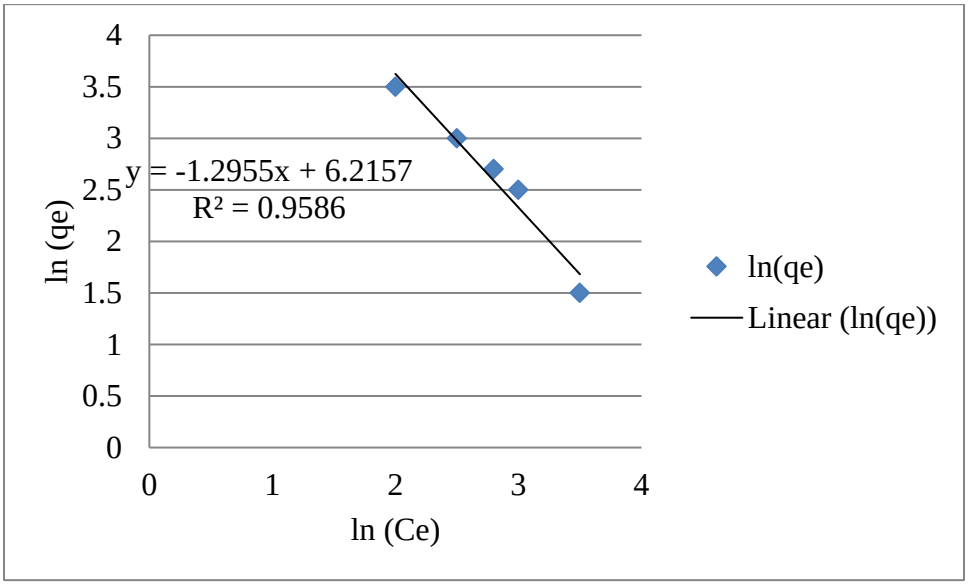

Figure 36: Freundlich adsorption isotherm for azo dye adsorption using sugarcane bagasse as bio adsorbent

Table 9: Isotherm constant for azo dye adsorption

Isotherms	Values
Langmuir	
q_m (mg/g)	-0.183
K_L (L/mg)	6.493
R^2	0.867
Freundlich	
K_F (mg/g)	73
N	-1.538
R^2	0.865

In addition to this the isotherm models were applied for adsorption of dye based textile wastewater and as it can be observed below in the figure 37 for the models Langmuir adsorption isotherm for textile wastewater and Freundlich adsorption isotherm in the figure 38 using sugarcane bagasse as bio-adsorbent the comparative analysis of both models indicate that Langmuir isotherm model is well fitted for adsorption with $R^2 = 0.866$ value.

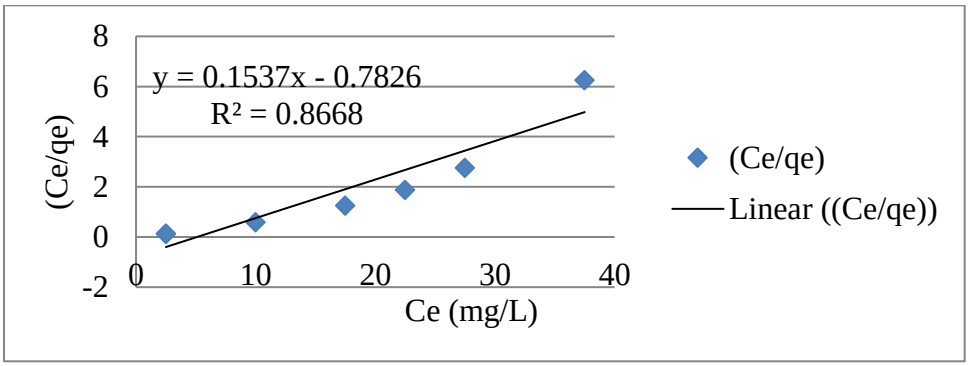

Figure 37: Langmuir adsorption isotherm for textile wastewater adsorption using sugarcane bagasse as bio adsorbent

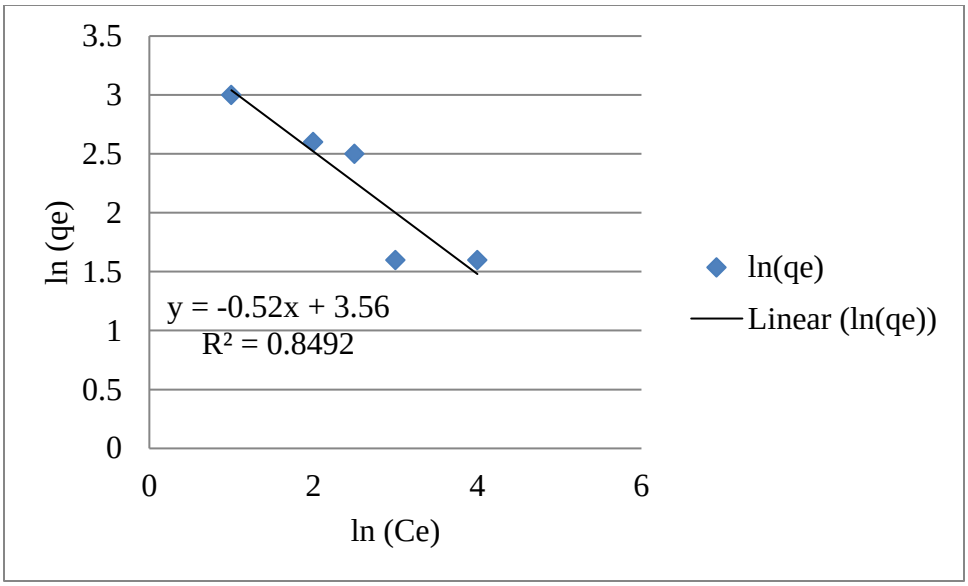

Figure 38: Freundlich adsorption isotherm for textile wastewater adsorption using sugarcane bagasse as bio adsorbent

Table 10: Isotherm constants for textile wastewater adsorption

Isotherms	**Values**
Langmuir	
q_m (mg/g)	-0.7
K_L (L/mg)	9.52
R^2	0.866
Freundlich	
K_F (mg/g)	29.9
N	-2.967
R^2	0.759

5. CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusion

Characterization of textile waste water was carried out and the result before adsorption was 48 mg/L up to 163 mg/L for BOD. The adsorption experiment was carried out using synthetic azo dye and textile waste water, and the comparative analysis of these two adsorption provided that azo dye was removed a percentage of 90 and dye based textile wastewater was removed with a percentage of 81.The optimum operating conditions for azo dye adsorption was accompanied with the controlling factors of concentration 52.5 mg/L,dose of adsorbent 2.5 gram, pH 6.5 and contact time 75 minute as well as for dye based textile wastewater ,the optimum operating conditions were with concentration 52.5 mg/L,dose of adsorbent 2.5 gram, pH 6.5 and contacting time 75 minute. In addition to this FTIR analysis of sugarcane bagasse was carried out, and the result indicate that the functional group before adsorption was oxidized nitrogen functions such as alkyl bromide and sulphur compounds such as disulfide at the lower peak and at the higher peak oxidized nitrogen functions such as oxime (=NOH).The FTIR analysis of after adsorption indicates at the lower peak the presence of alkane (C-C) and the presence of nitrogen compounds such as NH_2 as well as at the higher peak the experimental result indicate the presence of nitrogen compounds as well as hydrocarbons of alkyne,alkene and alkane. The experimental ANOVA analysis of azo dye adsorption and dye based textile wastewater was accomplished using a quadratic model and the model was significant for both. The experimental result of ANOVA analysis for azo dye adsorption a controlling parameter of concentration,pH and time were significantly affect the experimental operating condition, where as adsorption of dye based textile wastewater a controlling parameter of concentration,pH and time were significantly affect the experimental operating condition.

Kinetic study was carried out for adsorption of azo dye and dye based textile wastewater and pseudo first order model was well fitted adsorption of azo dye and dye based textile wastewater using sugarcane bagasse as bio-adsorbent. Finally adsorption isotherm was carried out for adsorption of azo dye and dye based textile wastewater and Langmuir isotherm model was well fitted for both adsorption.

5.2 Recommendations

The following points should be put into practice for further performance of adsorption using sugarcane bagasse as bio adsorbent:

- When textile industries use sugarcane bagasse as bio adsorbent it is very important to adapt their effluent with in a pH of 6 – 6.5, for better comfortable and effective for adsorption of textile wastewater on to sugarcane bagasse.
- To establish well suitability of adsorption using sugarcane bagasse, it is recommended to carry out BET test so that to analyze surface area and volume of the pores of sugarcane bagasse.
- Temperature should be taken as a controlling factor to evaluate the sugarcane bagasse as bio adsorbent.
- SEM (Scanning electron microscopy) test should be applied to know the detail morphology of sugarcane bagasse so that to adapt it for better performance of adsorption.

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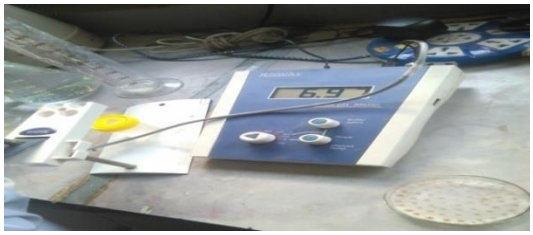
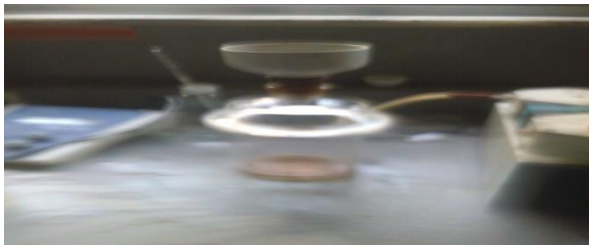
APPENDICES

Appendix A: Laboratory Equipment

Shaker



pH meter



Vacuum filter

Soxhelet

Mesh sieve with different size

Appendix B: Laboratory Preparation and Adsorption

Prepared sugarcane bagasse

Samples prepared for adsorption

Raw textile wastewater

Result for azo dye adsorption

Image of bagasse after adsorption of azo dye

Appendix C: Experimental Data

Table: Experimental data for azo dye adsorption was reported as follow:

Run number	Initial concentration(mg/L)	Final concentration(mg/L)	Absorbance	Azo dye removal(%)
1	45	14.85	0.03455	67
2	60	20.4	0.0512	66
3	45	14.4	0.0332	68
4	60	20.4	0.0512	66
5	45	22.5	0.0575	50
6	60	27	0.071	55
7	45	20.7	0.0521	54
8	60	26.4	0.0692	56
9	45	13.05	0.02915	71
10	60	11.4	0.0242	81
11	45	12.6	0.0278	72
12	60	12	0.026	80
13	45	13.5	0.0305	70
14	60	12	0.026	80
15	45	8.55	0.01565	81
16	60	12	0.026	80
17	48.75	13.65	0.03095	72
18	56.25	12.9375	0.0288	77
19	52.5	13.65	0.03095	74
20	52.5	13.65	0.03095	74
21	52.5	5.25	0.00575	90
22	52.5	12.6	0.0278	76
23	52.5	12.6	0.0278	76
24	52.5	12.6	0.0278	76
25	52.5	12.075	0.026	77
26	52.5	12.6	0.0278	76

Table: Experimental data for textile raw wastewater adsorption was reported as follow:

Run number	Initial concentration(mg/L)	Final concentration(mg/L)	Absorbance	Color Removal (%)
1	45	19.89	0.049	55.8
2	60	26.4	0.069	56
3	45	18.99	0.047	57.8
4	60	26.4	0.069	56
5	45	27	0.071	40
6	60	33	0.089	45
7	45	25.2	0.066	44
8	60	32.4	0.087	46
9	45	17.389	0.042	61
10	60	17.29	0.042	71
11	45	17.19	0.042	61.8
12	60	18.19	0.045	69.68
13	45	17.89	0.044	60
14	60	18.289	0.045	69.5
15	45	17.389	0.042	61
16	60	18.49	0.045	69
17	48.75	18.589	0.046	61.86
18	56.25	18.69	0.046	66.77
19	52.50	18.75	0.046	64
20	52.50	18.779	0.046	64
21	52.50	9.890	0.0196	81.16
22	52.50	17.59	0.043	66.5
23	52.50	17.68	0.043	66
24	52.50	17.789	0.043	66
25	52.50	17.59	0.043	66.5
26	52.50	17.89	0.044	66

Table: Experimental data for kinetics of textile wastewater adsorption

Number	t(min)	C_O(mg/L)	m(gram)	q_t(mg/L)	C_t(mg/L)	(q_e - q_t)	(t/q_t)	V(L)
1	85	52.5	2.5	7	37.5	14	12	1
2	95	52.5	2.5	10	27.5	10	9.5	1
3	105	52.5	2.5	12	22.5	8	8.75	1
4	115	52.5	2.5	14	17.5	6	8.2	1
5	125	52.5	2.5	17	10	3	7.35	1
6	135	52.5	2.5	20	2.5	_	_	1
7	145	52.5	2.5	20	2.5	_	_	1

Table: Experimental data for kinetics of azo dye adsorption

Number	t(min)	Co(mg/L)	m(g)	q_t(mg/g)	C_t (mg/L)	(q_e-q_t)	t/q_t	V(L)
1	85	52.5	2.5	5.6	40	13	15	1
2	95	52.5	2.5	8	32.5	10	11.8	1
3	105	52.5	2.5	10	27.5	8	10.5	1
4	115	52.5	2.5	13	20	5	8.8	1
5	125	52.5	2.5	16	12.5	2	7.8	1
6	135	52.5	2.5	18	7.5	_	_	1
7	145	52.5	2.5	18	7.5	_	_	1