Decolorization of selected dyes in aqueous solution using *Helianthus annuus*

By

Navanithi. D

(15PTF004)

A Thesis submitted to the

Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore-641 043

In Partial Fulfillment of the Requirements for the Degree of

Master of Science
in
Textiles and Fashion Apparel

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Certified as Bonafide Research Work

Signature of the

Head of the department

10/4/17

Signature of the Guide

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1. INTRODUCTION

Textile industries consume a large volume of water, organic and inorganic chemicals for making various textile goods and as a result, large volume of wastewater is discharged onto the land with or partially or without treatments (Sivakumar et al., 2012). The raw materials particularly dyes used in the textile industry determine the volume of water required for production as well as wastewater generation (Irina et al.,2008). The wastewater generated from the various processing units in a textile industry are desizing, scouring, bleaching, mercerizing, dyeing, printing, and packing.

Dyes are basically natural or synthetic, organic compounds that can connect themselves to surfaces or fabrics to provide bright and lasting colour (Chincholi et al.,2014). They are applied in various industries such as leather, textile, paper, rubber, cosmetics, plastic, pharmaceuticals and food industries. Most of them are complex organic molecules and are resistant to many things such as the action of detergents (Yagub et al.,2014). Coloured dye wastewater is regarded as a direct result of the production of the dye and also as a consequence of its use in the textile and related industries. There are more than 100,000 commercial dyes known with an annual production of over 7 X 105 tonnes per year (Sen et al.,2011). It is estimated that 2% of the dyes are discharged in effluent from manufacturing operation, while 10% was discharged from textile and associated industries (Allen et al.,2004). Discharge of these dyes into the water streams will affect the people who may use these effluents for living purposes such as washing, bathing and drinking (Sharma et al.,2000). Some dyes can cause allergy, dermatitis, skin irritation, cancer and mutations in humans (Bhatnagar et al.,2005).

Dyes are also associated with environmental concern with their absorption and reflection of sunlight entering the water, which will inhibit the growth of bacteria, limiting it to levels insufficient to biologically degrade impurities in the water (Mckay et al.,2007). Furthermore, dyes can also affect the aquatic plants as they reduce sunlight transmission through water. Therefore, the removal of such coloured compounds from waste effluents becomes environmentally important because even a small amount of dye in water can be toxic and highly visible (Chiou et al.,2004).

The wastewater discharged from the textile industries are exceeding the standard for discharge into receiving waters and hence, wastewater from the textile industries have to be treated before being discharged to the environment to meet permissible limit of wastewater discharge standards (Sivakumar, 2014).

Many approaches have been taken to reduce water consumption and by recycling the wastewater from the textile industries. Various methods, including aerobic and anaerobic microbial degradation, coagulation (Guendy,2010), activated carbon (Syafalni et al., 2012), electrochemical processes, reverse osmosis (Ramesh et al., 2013), ozonation, adsorption (Shankar et al.,, 2014), catalytic oxidation and membrane processes (Abdulraheem et al., 2012). can be employed to remove various pollutants in a textile industry wastewater including colour removal. However, these technologies are often ineffective in removal of colour, expensive and less adaptable to dye-loaded wastewaters (Srinivasan et al.,2010).

The process of adsorption enables removing not only color-producing compounds but also other substances posing threat to the natural environment from water (Filipkowska et al.,2010). Adsorption is a choice because it allows removal of different types of dyes (Gupta et al., 2009). Even though activated carbon is effective and widely used as adsorbent, the costs of production are quite high, and therefore, many researches have been performed to study different types of low-cost materials as adsorbents (Bhatnagar et al.,2010). Compared to commercial activated carbon and resins, agricultural by-products from renewable resources are less expensive and biodegradable (Gupta et al., 2011). Waste materials or by-products from agriculture have been used as alternative adsorbent to remove dyes from wastewater (Gupta et al,.2009). Therefore there is a need for the development of low cost, easily available materials which can adsorb dyes from textile waste water (Mittal et al.,2010).

Hence the present study “Decolorization of selected dyes in aqueous solution using *Helianthus annuus*” was designed with the following objectives,

- To screen various adsorbents for their efficiency in decolourizing the textile dye solution
- To optimize various parameters such as adsorbent dosage, initial dye concentration, time, temperature and pH for efficient removal of dye from aqueous solution

- To reuse the treated dye solution for dyeing the selected fabric
- To compare the properties of the fabrics dyed with fresh water and decolourized water

2. REVIEW OF LITERATURE

2.1 Overview of pollution

Pollution is considered to be the unwanted material into the environment. These residues may be unwanted or unprocessed raw materials, or they may be by-products (Vivien 2012). Though these pollutants are unavoidable, they can be reduced to some extent and can be discharged which will reduce the effect on environment. The pollution in a textile mill can be air pollution or water pollution or noise pollution. Among the three types, water pollution is considered to be more hazardous. Because of scarcity of land many textile industries are located nearby households. So any minor disaster will ultimately cause greater damage to large population near and far the unit (Chakraborty, et.al, 2012). According to United Nations Environment Programme (UNEP, 2010) every year 400-500 million tons of deadly chemicals like cyanide, sulphur and other radioactive substances are discharged into water.

Many industries like oil refineries, textiles, and chemical industries in India create water pollution above the permissible level. These industries can‘t be separated from each other. One way or the other, they are interdependent to each other so continues the process of environmental pollution. The industries aim at higher profit rate hence they withdrew the act of ethical behaviour. When technology and science was lagging, the textile industries used natural dyes which are no harm to living beings and surroundings, but gone are those days. In order to achieve target the industries stick on to the usage of synthetic fibers which are non biodegradable and ultimately spoil the water and land resources when discharged without proper treatment. Thus the rate of water pollution depends upon the amount of water and chemical substances used for the manufacturing process. The effects caused by other pollutants in textile wastewater, and the presence of very small amounts of dyes (<1 mg/L for some dyes) in the water, which are nevertheless highly visible, seriously affects the aesthetic quality and transparency of water bodies such as lakes, rivers and others, leading to damage to the aquatic environment (Little, 2008).

According to WHO people in the universe irrespective of their status and societal conditions, have right to have sufficient quantity of water with high quality similar to their basic needs. In recent years the fatality of death endorsed because of water pollution everywhere and majority of the diseases are due to this pollution (Chikogu et.al, 2012). The pollution due to untreated water causes major environmental pollution and becoming a threat in many industrialist areas of developing countries. These effluents may be infectious or radioactive some times.

The textile industry is the major contributor in many nations in terms of economy and employment. The small and large scale operations in this textile sector though contribute much to the economy also cause hazardous effect to the environment (Parvathi, et al., 2009). The textile manufacturing processes involve large consumption of water, energy and various chemicals which will generate waste at the end or as a by-product. The major problem with regard to water usage is the untreated effluents which are discharged directly into nearby water bodies (Jaganathan 2014). When a manufacturing process starts there arise large quantity of waste in terms of water, energy and other chemical substances which will readily or indirectly affects the environment to a greater extent. The pollution created by this textile processing may be in the form of air, water or noise which is considered to be hazardous to health and for the surroundings. Each time the manufacturing process varies according to the requirement of the customer so the waste generated varies each time depending up on the type of chemicals and raw materials used (Parvathi, et.al,2009).

2.2 Water pollution

Water pollution is considered to be the biggest environmental threat all over the world. In India government is investing more to control water pollution but the results are below the satisfactory level (Chakraborty et al, 2012). Water quality is usually measured in terms of rate of occurrence of a substance in aqueous solution. The textile industry accounts for 2.07% of water consumption (Carmen et.al,2012). The consumption for the textile industry includes various processes such as sizing, dyeing and other end product processes. The major problem arises when these chemicals are directly discharged into water bodies thus causing water pollution. This water pollution not only affects human beings and house hold animals but also aquatic animals to the same extent (Jaganathan et al.,2014). The textile fabric production consist of various stages, initially the cotton are mixed in various proportions to make fibers. The fibers now undergo the process of spinning and convert into yarn. Now the process of dyeing takes place where many chemical agents for colouring purpose is used. In order to avoid detaching the yarns undergo the process of sizing where they are washed with cellulose and amylase substances. Now the processed yarns are weaved into end product while undergoing these processes various pollutants are discharged as chemical waste. When these water are discharged without any proper treatment then it will lead to hazardous effects to living organisms, so before discharging they should be properly treated either biological or physical or chemical means (Carmen et.al,2012).

2.3 Textile industry as a source of water pollution

One of the main sources of water pollution is the textile industry and its dye-containing wastewaters (i.e. 10,000 different textile dyes with an estimated annual production of 7.105 metric tonnes are commercially available worldwide; 30% of these dyes are used in excess of 1,000 tonnes per annum, and 90% of the textile products are used at the level of 100 tonnes per annum or less). 10-25% of textile dyes are lost during the dyeing process, and 2-20% are directly discharged as aqueous effluents in different environmental components (Zaharia et al., 2012). In particular, the discharge of dye-containing effluents into the water and environment is undesirable, not only because of their colour, but also because of dyes released and their breakdown products are toxic, carcinogenic or mutagenic to life forms mainly because of carcinogens, such as benzidine, naphthalene and other aromatic compounds (Suteu et al., 2010). Without adequate treatment these dyes can remain in the environment for a long period of time (Hao et al., 2000).

Textile industry uses a large quantity of chemicals such as

- *Detergents and caustic*, which are used to remove dirt, grit, oil, and waxes.
- *Sizing agents*, which are added to improve weaving.
- *Oils*, which are added to improve spinning and knitting.
- *Latex and glues*, which are used as binders.
- *Dyes, fixing agents, and many in-organics*, which are used to provide the brilliant array of colours the market demands.
- *A wide variety of special chemicals*, which are used such as softeners, stain release agents, and wetting agents.

Many of these chemicals become part of the final product whereas the rest are removed from the fabric, and are purged in the textile effluent.

The wastewater composition depends on the different organic-based compounds, inorganic chemicals and dyes used in the industrial dry and wet-processing steps (Robinson et al., 2001). Textile effluents from the dyeing and rinsing steps represent the most coloured fraction of textile wastewaters, and are characterized by extreme fluctuations in many quality indicators such as COD, BOD, pH, colour, salinity and temperature. The colour of textile wastewater is mainly due to the presence of textile dyes, pigments and other coloured compounds. A single dyeing operation can use a number of dyes from different chemical classes resulting in a complex wastewater. Moreover, the textile dyes have complex structures, synthetic origin and recalcitrant nature, which makes them obligatory to remove from industrial effluents before being disposed into hydrological systems (Anjaneyulu et al, 2005).

The decolourization treatments applied for different textile effluents include current and also advanced non-biological (i.e. specific mechano-physical, chemical, electrochemical processes, etc.) and also biological processes (Suteu et al., 2009 b;). The colour in watercourses is accepted as an aesthetic problem rather than an eco-toxic hazard. Therefore, the public seems to accept blue, green or brown colour of rivers but the 'non-natural' colour as red and purple usually cause most concern (Zaharia et al, 2011). The polluting effects of dyes against aquatic environment can be also the result of toxic effects due to their long time presence in environment (i.e. half-life time of several years), accumulation in sediments but especially in fishes or other aquatic life forms, decomposition of pollutants in carcinogenic or mutagenic compounds but also low aerobic biodegradability. Due to their synthetic nature and structure mainly aromatic, the most of dyes are non-biodegradable, having carcinogenic action or causing allergies, dermatitis, skin irritation or different tissular changes (Börnick and Schmidt, 2006).

One of the most difficult tasks confronted by the wastewater treatment plants of textile industries is the removal of the color of these compounds, mainly because dyes and pigments are designed to resist biodegradation, such that they remain in the environment for a long period of time (Hao et al., 2000).

2.4 Dyes

Dyes may be defined as substances that, when applied to a substrate provide colour by a process that alters, at least temporarily, any crystal structure of the coloured substances (Othmer, 2004). Such substances with considerable colouring capacity are widely employed in the textile, pharmaceutical, food, cosmetics, plastics, photographic and paper industries (Zollinger, 2007). The dyes can adhere to compatible surfaces by solution, by forming covalent bond or complexes with salts or metals, by physical adsorption or by mechanical retention (Bafana et.al.,2011). It is estimated that over 10,000 different dyes and pigments are used industrially and over 7 x 105 tons of synthetic dyes are annually produced worldwide (Robinson et al.,2001).

Textile materials can be dyed using batch, continuous or semi-continuous processes. The kind of process used depends on many characteristics including type of material as such fibre, yarn, fabric, fabric construction and garment, as also the generic type of fibre, size of dye lots and quality requirements in the dyed fabric. Among these processes, the batch process is the most common method used to dye textile materials (Perkins, 2010).In the textile industry, up to 200,000 tons of these dyes are lost to effluents every year during the dyeing and finishing operations, due to the inefficiency of the dyeing process (Ogugbue et al., 2011). Unfortunately, most of these dyes escape conventional wastewater treatment processes and persist in the environment as a result of their high stability to light, temperature, water, detergents, chemicals, soap and other parameters such as bleach and perspiration (Couto, 2009).

The dye can be fixed to the fiber by several mechanisms, generally in aqueous solution, and may involve primarily four types of interaction: ionic, Van der Waals and hydrogen interactions, and covalent bonds (Christie, 2001).

2.4.1 Classification

Dyes are of dyes chemical compounds that can affix themselves on the surface of fabrics to impart color (Yagub et al.,2014). It refers as a substance, usually organic, which is designed to be absorbed or adsorbed by, made to react with, or deposited within a substrate to impart color to that substrate with some degree of permenance (Burkinshaw, 2016).

Dyes can be classified mainly into two types based on their origin

Natural dyes

Natural dyes can be derived from the three primary sources—plants, animals and minerals. They are mostly negatively charged. Positively charged natural dyes can also exist, but they are not very common. Irrespective of the source of origin, natural dyes can be categorized as:

1. Substantive dyes:

   Those dyes which become chemically fixed to the fiber without the aid of any other chemicals or additives are referred to as substantive dyes or direct dyes.
2. Adjective dyes:

   Those natural dyes which require an added substance or mordant to make the dyes colorfast are known as adjective dyes or mordant dyes. Most of the natural dyes belong to this category (Cardon, 2007).

Synthetic dyes

Dyes derived from organic or inorganic compounds are known as synthetic dyes. Because of their easy use, cost effectiveness and wide range of colors, these dyes became popular. (Mittal et al., 2009).

Acid dyes

Acid dyes are highly water soluble anionic dyes which contain one or more sulfonic groups/substituents and other acidic groups. They have better light fastness compared to basic dyes. The existence of sulfonic acid groups upsurge their solubility in water and give the dye molecules a negative charge.

Acid dyes can be further divided into two sub-groups

1. Acid-levelling dyes:

   These dyes are smaller or medium sized, and show a moderate affinity for wool fibers. Hence, due to average affinity these dyes are not very resistant to washing.
2. Acid-milling dyes:

These dyes are larger than acid levelling dyes, and show a much stronger intermolecular attractions for wool fibers. Due to this strong affinity these dyes are very resistant to washing. (Gupta et al., 2011).

Basic dyes

These water soluble dyes possess cationic functional groups. They are generally amino and substituted amino compounds soluble in acid. They become attached to the fibers by formation of ionic bonds with fiber's anionic groups. (Raval et al., 2016).

Direct dyes

Although these dyes are water soluble anionic dyes, they cannot be classified as acid dyes because the acid groups are not the means of attachment to the fiber. Since, these dyes do not require any kind of fixing, they are called direct dyes (Saravanan et al.,2016).

Mordant dyes

A latin word mordant means ‘to bite’. It acts as ‘fixing agent’ to improve the color fastness of some acid dyes, because they have the ability to form insoluble colored complexes with metal ions. They are usually metal salts. Besides, alum there is a large range of other metallic salt mordants available. Aluminium, chromium and iron salts are frequently used for synthetic mordant dyes (Cardon, 2007).

Reactive dyes

Reactive dyes will form a covalent bond while they react with the cellulosic fiber. As a result they are very difficult to remove, once attached with the fiber. Initially, these dyes were designed for cellulose fibers but now some fiber-reactive dyes for protein and polyamide fibers are also commercially available (Nidheesh et al.,2013).

Disperse dyes

These dyes have substantially low solubility in water. Their structure is small, planner and non-ionic with attached polar functional groups, such as $–NO_2$ and –CN. They are mainly used for dyeing of polyesters because they can interact with the polyester chains by forming dispersed particles (Dawood et al., 2014).

Solvent dyes

These dyes are insoluble in water but soluble in alcohols, chlorinated hydrocarbons and liquid ammonia. They are mainly used for coloring plastics, synthetic fibers, gasoline, oils and waxes (Sharma et al., 2011).

Sulphur dyes

Sulphur dyes are applied to cotton from an alkaline reducing bath with sodium sulphide as the reducing agent. These dyes are water insoluble but they are soluble in their reduced form and exhibit affinity for cellulose. They are low cost and have good fastness to light, washings and acids. The actual structures of sulphur dyes are largely unknown although it is considered that they possess sulphur-containing heterocyclic rings (Bello et al.,2014).

2.5 Decolorization Techniques

Decolourization technologies can be divided into three categories: biological, chemical and physical (Robinson et al.,2001). All of them have advantages and drawbacks. Because of the high cost and disposal problems, many of these conventional methods for treating dye wastewater have not been widely applied at large scale in the textile and paper industries (Pereira et al.,2003). At present, there is no single process capable of adequate treatment, mainly due to the complex nature of the effluents (Marco et al.,2007). In practice, a combination of different processes is often used to achieve the desired water quality in the most economical way. Combined adsorption-biological treatments in order to improve the biodegradation of dyestuffs and minimize the sludge production (Mcmullan et.al.,2001).

2.5.1 Biological Treatment

Biological treatment is often the most economical alternative when compared with other physical and chemical processes (Grimau et al.,2006). A wide variety of microorganisms are capable of decolorizing a wide range of dyes, including bacteria, fungi, yeasts, actinomycetes and algae (Saratale et al.,2009). Biodegradation methods such as fungal decolorization, microbial degradation, adsorption by (living or dead) microbial biomass and bioremediation systems are commonly applied to the treatment of industrial effluents because many microorganisms such as bacteria, yeasts, algae and fungi are able to accumulate and degrade different pollutants. However, their application is often restricted because of technical constraints. Biological treatment requires a large land area and is constrained by sensitivity toward diurnal variation as well as toxicity of some chemicals, and less flexibility in design and operation (Bhattacharyya et al.,2003). Biological treatment is incapable of obtaining satisfactory color elimination with current conventional biodegradation processes (Robinson et al.,2001). Although many organic molecules are degraded, many others are recalcitrant due to their complex chemical structure and synthetic organic origin (Dabrowski, 2001). In particular, due to their xenobiotic nature, azo dyes are not totally degraded. The isolation of potent species and their degradation is one of the interesting biological aspects of effluent treatment (Chen et al.,2008).

2.5.2 Chemical Treatment

Chemical methods include coagulation or flocculation combined with flotation and filtration, precipitation-flocculation with Fe(II)/$Ca(OH)_2$, electro flotation, electro kinetic coagulation, conventional oxidation methods by oxidizing agents (ozone), irradiation or electrochemical processes (Forgacs et al.,2004). These chemical techniques are often expensive and although the dyes are removed, accumulation of concentrated sludge creates a disposal problem. There is also the possibility that a secondary pollution problem will arise because of excessive chemical use. Recently, other emerging techniques, known as advanced oxidation processes, which are based on the generation of very powerful oxidizing agents such as hydroxyl radicals, have been applied with success for pollutant degradation. Although these methods are efficient for the treatment of waters contaminated with pollutants, they are very costly and commercially unattractive. The high electrical energy demand and the consumption of chemical reagents are common problems (Ragavacharya, 2007). The process gives satisfactory removal of disperse, vat and sulphur dyes. However, the limitation of this technique is the production of concentrated sludge in large quantity, and the removal of reactive, azo, and basic dyes by this technique is not at par or up to the mark.

2.5.3 Physical Treatment

Physical treatment methods employ the application of physical forces for the separation of dyes from wastewater. Physical methods include different precipitation methods (such as coagulation, flocculation, and sedimentation), adsorption (on a wide variety of inorganic and organic supports), filtration, reverse osmosis, ultra filtration, and nano filtration (Uday et al.,2016). The major disadvantage of the membrane processes is that they have a limited lifetime before membrane fouling occurs and the cost of periodic replacement must thus be included in any analysis of their economic viability (Nguyen et al.,2013). Liquid-phase adsorption is one of the most popular methods for the removal of pollutants from wastewater since proper design of the adsorption process will produce a high-quality treated effluent. This process provides an attractive alternative for the treatment of contaminated waters, especially if the adsorbent is inexpensive and does not require an additional pre-treatment step before its application. Adsorption is a well known equilibrium separation process and an effective method for water decontamination applications (Dabrowski, 2001). Adsorption has been found to be superior to other techniques for water re-use in terms of initial cost, flexibility and simplicity of design, ease of operation and insensitivity to toxic pollutants. Adsorption also does not result in the formation of harmful substances. In recent years, the search for low-cost adsorbents that have pollutant –binding capacities has intensified. Materials locally available such as natural materials, agricultural wastes and industrial wastes can be utilized as low-cost adsorbents (Crini ,2005).

2.6 Adsorption

Adsorption techniques for wastewater treatment have become more popular in recent years owing to their efficiency in the removal of pollutants, which are difficult to treat with biological methods. Adsorption can produce high quality water while also being a process that is economically feasible. Decolourisation by adsorption is influenced by many factors including dye/adsorbent interaction, adsorbent's surface area, particle size, temperature, pH and contact time.

Physical adsorption occurs when weak interparticle bonds exist between the adsorbate and adsorbent. Examples of such bonds are Van Der Waals, hydrogen and dipole-dipole. In the majority of cases physical adsorption is easily reversible. Chemical adsorption occurs when strong interparticle bonds are present between the adsorbate and adsorbent due to an exchange of electrons. Examples of such bonds are covalent and ionic bonds. Chemisorption is a kind of adsorption which involves a chemical reaction between the surface and the adsorbate. New chemical bonds are generated at the adsorbent surface. Examples include macroscopic phenomena that can be very obvious, like corrosion, and subtler effects associated with heterogeneous catalysis. The strong interaction between the adsorbate and the substrate surface creates new types of electronic bonds. In contrast with chemisorption is physisorption, which leaves the chemical species of the adsorbate and surface intact. It is conventionally accepted that the energetic threshold separating the binding energy of "physisorption" from that of "chemisorptions" is about 0.5 eV per adsorbed species. Chemisorption is deemed to be irreversible in the majority of cases (Crini, 2008). Most adsorbents are highly porous materials. As the pores are generally very small, the internal surface area in orders of magnitude is greater than the external area.

Activated carbon is the most popular for adsorbent the removal of pollutants from wastewater (Babel et al.,2003). The effectiveness of adsorption on commercial activated carbons (CAC) for removal of a wide variety of dyes from wastewaters has made it an ideal alternative to other expensive treatment options Because of their great capacity to adsorb dyes, CAC are the most effective adsorbents. This capacity is mainly due to their structural characteristics and their porous texture which gives them a large surface area and their chemical nature which can be easily modified by chemical treatment in order to increase their properties. However, activated carbon presents several disadvantages. It is quite expensive, the higher the quality, the greater the cost, non-selective and ineffective against disperse and vat dyes. The regeneration of saturated carbon is also expensive, not straightforward, and results in loss of the adsorbent (Forgacs et al.,2004).

2.7 Adsorbent

The substance which adsorbs the adsorbate is considered as adsorbent (Gupta et al., 2012**).** Adsorption does not result in the formation of harmful substances. However, the use of activated carbon as an adsorbent is disadvantageous because it is relatively expensive (Crini, 2006), A number of nonconventional low-cost materials have recently been tested and proposed as alternative adsorbent to activated carbon for the removal of dyes from aqueous solutions(Sidiras et al., 2011). Adsorbent can be considered low-cost since it is abundant in nature, inexpensive, requires little processing, and is an effective material (Crini, 2006). Recently, a wide variety of materials has been used as low-cost adsorbents, which are mainly vegetable waste such as citrus fruit peel (Dutta et al., 2011). Application of waste materials is gradually becoming of great concern, because these wastes are unused resources and can cause serious disposal problems. For better use of inexpensive and abundant agricultural waste, it has been investigated as a low-cost adsorbent, owing to its relatively high fixed carbon content and porous structure (Mohan et al.,2002).

2.8 Sunflower plant

Sunflower is an annual plant. Its botanical name *Helianthus annus* comes from Greek word *helios* (sun) and *anthos* (flower). The genus *Helianthus* is native to temperate North America and contains 14 annual and 37 prennial species (Schilling, 2006). It is an annual plant of large proportions, which can reach over 3 meters high. It is a plant with few branches and the stem is thick, erect and massive (http://www.botanical-online.com).

Sunflower is used in died (nuts and flour), for oil (skin protection from skin and to decorate the hair), to obtains colour (yellow and red), for medicinal purposes (anti-inflammatory and diuretic effects), and as an ornamental plant (religious ceremonies)(Enrique et al., 2015).

A large amount of agro-industrial waste generated every year in all over the world contained high lingocellulosics and starch content. Lignocellulose is the major component of biomass, comprising around half of the plant matter produced by photosynthesis (also called photomass) and representing the most abundant renewable organic resource in soil.

2.9 Adsorption isotherm

Adsorption properties and equilibrium data, commonly known as adsorption isotherms, describe how pollutants interact with adsorbent materials and so, are critical in optimizing the use of adsorbents (Hema, 2007). Equilibrium isotherm is described by a sorption isotherm, characterized by certain constants whose values express the surface properties and affinity of the sorbent. Sorption equilibrium is established when the concentration of sorbate in the bulk solution is in dynamic balance with that at the sorbent interface (Oladoja et al., 2008). The analysis of equilibrium adsorption data by fitting them to different isotherm models is an important step to find the suitable model that can be used for design purposes (Haghseresht and Lu., 2008). In order to optimize the design of an adsorption system to remove dye from solutions, it is important to establish the most appropriate correlation for the equilibrium curve (Goswami et.al.,2005). An accurate mathematical description of equilibrium adsorption capacity is indispensable for reliable prediction of adsorption parameters and quantitative comparison of adsorption behaviour for different adsorbent systems (or for varied experimental conditions) within any given system (Hameed, et al.,2008). The surface phase may be considered as a monolayer or multilayer (Salleh et al., 2011).

Adsorption equilibrium is established when the amount of dye being adsorbed onto the adsorbent is equal to the amount being desorbed (Tanyildizi, 2011). It is possible to depict the equilibrium adsorption isotherms by plotting the concentration of the dye in the solid phase versus that in the liquid phase (Bharathi et al.,2012) . The distribution of dye molecule between the liquid phase and the adsorbent is a measure of the position of equilibrium in the adsorption process and can generally be expressed by one or more of a series of isotherm models (Kyzas, 2010). The shape of an isotherm may be considered with a view to predicting if a sorption system is "favourable" or "unfavourable". The isotherm shape can also provide qualitative information on the nature of the solute–surface interaction (Chiou,2003). The two well known adsorption isotherm models are Langmuir and Freundlich isotherms. The Langmuir isotherm assumes monolayer adsorption onto a surface containing a finite number of adsorption sites of uniform strategies of adsorption with no transmigration of adsorbate in the plane of surface (Fytianos et al., 2000). While the Freundlich isotherm model assumes heterogeneous surface energies, in which the energy term in the Langmuir equation varies as a function of the surface coverage (Kyzas,2010). The Langmuir isotherm is developed by assuming that a fixed number of adsorption sites are available, and that the adsorption is reversible. The Langmuir isotherm may be used when the adsorbent surface is homogeneous (Bharathi, 2013).

2.10 Cotton

The word cotton is derived from ‘quton’ which means a plant found in a conquered land. The cotton industry contributes in a big way to industrial output, employment and balance of payments, making it one of the most important industries in the country (Seema, 2011). Cotton is the backbone of world’s textile trade. Many of our everyday textile fabrics are made from cotton (Gordan, 2003).

Cotton is also called the white gold. Cotton has high value due to aesthetics, its pleasant texture and matte look. Because of its high capacity to absorb, hold and dry moisture, cotton offers maximum comfort under extreme heat and humidity (Seema, 2011).

The world’s consumption of cotton fibre increases more rapidly than the production (William, 2010). It is a fibre that ‘breathes’. It can be made into loosely woven, light weight, soft, smooth material on one hand and tightly woven, almost air resistant fabric on the other. It is a preferred fabric for children, since cotton is non-allergenic (Seema, 2011). Fabrics that are hard wearing and cotton fabrics are capable of infinite variety of weave and colouring (Gordan, 2003).

3. EXPERIMENTAL PROCEDURE

3.1 Collection of adsorbents

3.2 Preparation of dye solution

3.3 Screening of different adsorbents for decolourizing basic yellow 13 and acid red 51 dyes

3.4 Characteristics of adsorbent

3.4.1 Determination of pH

3.4.2 Determination of moisture content

3.5 Optimization of various parameters for decolourization of selected dyes

3.5.1 Effect of adsorbent dosage

3.5.2 Effect of initial concentration of the dye on adsorption

3.5.3 Effect of temperature

3.5.4 Effect of time

3.5.5 Effect of pH

3.6 Decolourization of dye solution under optimized conditions

3.7 Reuse of decolourized solution for dyeing

3.8 Selection of fabric

3.8.1 Desizing of fabric

3.9 Dyeing

3.10 Scanning Electron Microscopic (SEM) Analysis

3.11 UV-Vis Spectrophotometer analysis

3.12 FTIR analysis

3. Experimental procedure

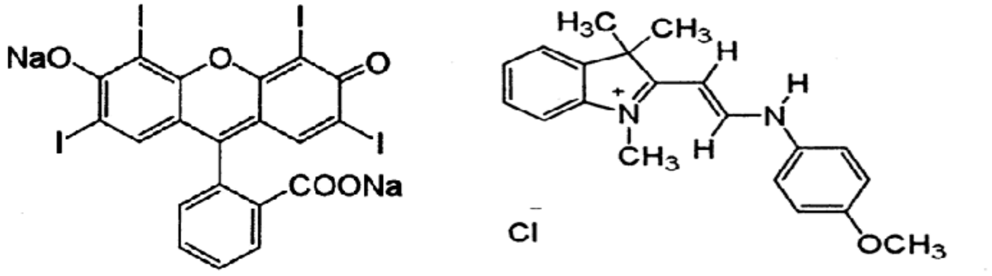
3.1 Collection of adsorbents

Adsorbents such as sugarcane bagasse, corn stalk, garlic peel, tapioca peel, sunflower stalk, sea foam, tea waste, orange peel, pea shell, egg shell were selected for pilot study. The selected adsorbents were collected from local market. The collected biomaterial is cleaned to remove soil and dust. The cleaned biomaterial is oven dried at 80^0c for 4 hours and crushed into particles. The obtained adsorbent samples were directly used without further processing.

3.2 Preparation of dye solution

Basic yellow 13 (BY13), chemical formula $C_{20}H_{23}ClN_2O$ and molecular weight 342.86g mol^{-1}, Acid red 51 (AR51), chemical formula $C_{20}H_6I_4Na_2O_5$ and molecular weight 879.86g mol^{-1} were procured from Anmol chemicals, Tirupur and kept at room temperature. The dye solutions were prepared at a concentration of 0.01%.

Chemical structure:


a. Acid red 51

b. Basic yellow 13

Figure 1: Chemical structure of dyes

3.3 Screening of different adsorbents for decolourizing basic yellow 13 and acid red 51

About one gram of each adsorbent was added separately into a series of beakers containing 0.01% of basic yellow 13 and acid red 51. The colour intensity of basic yellow 13 and acid red 51 was measured at 432nm and 509nm respectively using UV-visible spectrophotometer. The percent decolouration was calculated as

$$\% \text{ Decolourization} = \frac{\text{Initial absorbance- Final absorbance}}{\text{Initial absorbance}} \times 100$$

3.4 Characteristics of adsorbent

3.4.1 Determination of pH

Adsorbents may contain chemical groups on their internal surfaces of an acidic or alkaline nature and such groups often have a profound effect on the adsorption / precipitation of both organic and inorganic pollutants in wastewater. The suspension of adsorbent is allowed to settle and the pH of the supernatant liquor measured (Kay, 2006).

The One gram of powdered sunflower stalk was transferred into beaker containing 100ml of distilled water and mixed well. The sample was allowed to stabilize for one hour and the pH was measured.

3.4.2 Determination of moisture content

The moisture content does not adversely affect the adsorbent performance, but merely acts as a diluents, so that an additional quantity of adsorbent is required to provide the desired dry weight (Thobanoglous et al., 2003).

Ten grams of powdered sunflower stalk was dried in an oven for 1 hour. The dried adsorbent was kept at room temperature for 1 hour and weighed. The weight loss percent was calculated as

$$\% \text{ Weight loss} = \frac{\text{Initial weight - Final weight}}{\text{Initial weight}} \times 100$$

Plate I

Selected adsorbents for the decolourization of selected dyes.

1. Sea foam, 2. Orange peel, 3. Garlic peel, 4. Corn stalk, 5. Egg shell,
6. Tapioca peel, 7. Pea shell, 8. Tea waste, 9. Sugarcane bagasse,
10. Sunflower stalk.

3.5 Optimization of various parameters for decolourization of selected dyes

3.5.1 Effect of adsorbent dosage

To determine the optimum quantity of adsorbent to decolourize the selected dye solutions, 1g, 2g, 3g, 4g and 5gm of powdered sunflower stalk was added separately to beakers containing 0.01% of basic yellow 13. The dye solution was incubated at room temperature for 8 hrs. The same procedure was followed for acid red 51. The absorbance of dye solution was measured in UV-visible spectrophotometer and the percent decolourization was calculated.

3.5.2 Effect of initial concentration of the dye

To determine the optimum dye concentration of basic yellow 13, 0.01, 0.02, 0.03, 0.04, and 0.05 percent of the dye was added individually to beakers containing optimum adsorbent dosage. The absorbance of dye solutions were measured using UV-Visible spectrophotometer.

3.5.3 Effect of temperature

To determine the optimum temperature for decolourization of basic yellow 13 and acid red 51, the beakers containing 0.01% of dye with optimum concentration of sunflower stalk was incubated at different temperatures such as 30^0C, 40^0C, 50^0C, 60^0C and 70^0C. The percent decolourization was determined.

3.5.4 Effect of time

To determine the optimum time for decolourization of basic yellow13 and acid red51, the dye solution at 0.01% concentration was incubated at various time intervals such as 2 hrs, 4 hrs, 6 hrs, 8 hrs and 10 hrs at optimum temperature and adsorbent dosage. Percent decolourization was determined.

Control and decolourized solution of Basic Yellow 13

Control and decolourized solution of Acid Red 51

Plate II

Decolourization of Selected Dye Solution Using Selected Adsorbent Under Optimized Conditions

3.5.5 Effect of pH

To evaluate the optimum pH for decolourization, the initial pH of dye solution was adjusted to 5,6,7,8 and 9 using 1N HCL and 1N NaOH. Percent decolourization was determined.

3.6 Decolourization of selected dye solution under optimized conditions

Basic yellow 13 dye solution was decolourized using four grams of powdered sunflower stalk at an optimum dye concentration of 0.01% at pH 7 with incubation period of 4 hours at 40^0C. Acid red 51 dye solution was decolourized at optimum concentration of sunflower stalk (4%) at pH 5 with an incubation time of 8 hrs at 40^0C. The decolourized solution was reused for dyeing.

3.7 Reuse of decolourized solution for dyeing

Reuse of wastewater involves environmental benefits, because it decreases discharge of pollutants and collection of high quality water from ground and surface aquifers. Recycled water can satisfy most water demands, as long as it is adequately treated to ensure water quality appropriate for the use. (Ben, et al.,2012). Reuse provides an excellent, environmentally-preferred alternative to other waste management methods, because it reduces air, water and land pollution. Hence in the present study, an attempt was made to reuse the decolourised dye solution. The decolourized dye solution was used to dye cotton fabric with basic yellow 13 and acid red 51 dyes.

3.8 Selection of fabric

Cotton has been used for over 7000 years and consists of mainly cellulose, natural waxes and proteins. The large number of hydroxyl groups on cellulose provides a great water absorption capacity (Alcantara, 2012). Cotton today is a very large industry, the most common material for our clothing and furnishings, a widely traded commodity as well as the source of the means of living for million of cultivators, workers and large and small traders (Riello, 2013). The cotton fibre is an elongated cell, constructed from millions of cellulose molecules. Cotton cellulose is the purest form, which consists of 90% cellulose (Mishra, 2005). Cotton is the back bone of the world's textile trade. Many of our everyday textile fabrics are made from cotton fabrics that are hard wearing and capable of infinite variety of weave and colouring (Cook, 2003). Hence, plain woven fabric was chosen for the present study. It was procured from NTC, Murugan mills, Coimbatore.

3.8.1 Desizing of the fabric

It has been found that pre-treatment of cotton before dyeing can offer a simple and effective method of improving dye-fibre affinity. Desizing is the process in which the size applied to the warp yarn before weaving is removed to facilitate the penetration of dyes and chemicals in the subsequent wet processing operations (Aishwarya, 2012). Desizing of cotton fabric was carried out with following procedure:

Table 1

Desizing of cotton fabric

Cotton material	1 meter
Material liquor ratio	1:20
Detergent	2%
Wetting agent	0.5%
Temperature	80^0C to 100^0C
Time	1 hour

3.9 Dyeing

Dyes may be defined as substances that, when applied to a substrate provide color by a process that alters, at least temporarily, any crystal structure of the colored substances. The dyes can adhere to compatible surfaces by solution, by forming covalent bond or complexes with salts or metals, by physical adsorption or by mechanical retention. (Melih, 2013). The dyeing process is one of the key factors in the successful trading of textile products. In addition to the design and beautiful color, the consumer usually looks for some basic product characteristics, such as good fixation with respect to light, perspiration and washing, both initially and after prolonged use (Guaratini, 2012).

Dyeing is the application of colourant to the substrate in order to enhance the appearance by the attraction of hue. The selected cotton fabric was desized and then dyed with the basic yellow 13 and acid red 51 dyes using decolourized and soft water. The dyeing of cotton fabric with selected dyes was carried out with the following procedure:

Table 2

Procedure for dyeing with acid red 51

Percentage of shade	2%
Acetic acid	3%
Nacl or Na2so4	15%
Water	1:50
Temperature	900C
Time	60 mins

Table 3

Procedure for dyeing with basic yellow 13

Percentage of shade	2%
Acetic acid (40%)	6%
Nacl or Na2so4	5%
Water	1:50
Temperature	900C
Time	90 mins

The dyeing was done using fresh water and decolourized water.

3.10 Scanning Electron Microscopic (SEM) Analysis

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electrons-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties. Areas ranging from approximately 1cm to 5 microns in width can be imaged in a scanning mode using conventional SEM techniques (magnification ranging from 20x to approximately 30,000x, spatial resolution of 50 to 100nm).

The SEM is also capable of performing analyses of selected point locations on the sample; this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions.

3.11 UV-Vis Spectrophotometer analysis

UV spectroscopy is a type of absorption spectroscopy in which light of ultra-violet region (200-400 nm.) is absorbed by the molecule. Absorption of the ultra-violet radiations results in the excitation of the electrons from the ground state to higher energy state. The energy of the ultra-violet radiation that are absorbed is equal to the energy difference between the ground state and higher energy.

Many molecules absorb ultraviolet or visible light. The absorbance of a solution increases as attenuation of the beam increases. Absorbance is directly proportional to the path length and the concentration of the absorbing species.

Different molecules absorb radiation of different wavelengths. An absorption spectrum will show a number of absorption bands corresponding to structural groups within the molecule.

The analysis was carried out for the control and decolourized solution.

3.12 FTIR analysis

FTIR (Fourier Transform Infra-red Spectroscopy) is a sensitive technique particularly for identifying organic chemicals in a whole range of applications although it can also characterise some in-organics. FTIR relies on the fact the most molecules absorb light in the infra-red region of the electromagnetic spectrum. This absorption corresponds specifically to the bonds present in the molecule. The frequency ranges are measured as wave numbers typically over the range 4000 – 600 cm-1.

3.13 Adsorption isotherm

The equilibrium data obtained experimentally were analysed by two well-known adsorption isotherms with the help of nonlinear curves. Langmuir and Freundlich isotherms were employed to the experimental data, and graphs were plotted between Q_e

and C_e. Description, analysation and modelling of the experimental data were done by applying the obtained experimental data to Langmuir and freundlich isotherms. Nonlinear regression method was used to speculate the best fitted isotherm model.

3.13.1 Langmuir isotherm

Langmuir isotherm describes the homogenous adsorption over the surface of adsorbent. It is assumed that once a dye molecule occupies a site, no further adsorption can take place at the site views (Mall et al.,2006). The langmuir adsorption isotherm is calculated using the following formula;

$$X/m = (k_1{}^1 C_e /1+k_1C_e)$$

where, x - Amount of dye adsorbed (mg)

m – Weight of adsorbent (mg)

Ce – Concentration of dye at equilibrium

K - Adsorption capacity

K_1 – Energy of Adsorption

The essential characteristics of Langmuir adsorption isotherm can be expressed in terms of a dimensionless constant. R_L a dimensionless separation factor in Langmuir isotherm indicating the shape of the isotherm can be expressed as

$$R_L = 1/ (1+bc_i)$$

Where, C_i= initial concentration of the dye in mg/l

b = Langmuir constant (K1)

Table 4

Langmuir Adsorption Isotherm

R_L value	Type of Isotherm
$R_L > 1$	Unfavourable
$R_L = 1$	Linear
$R_L < 1$	Favourable

3.13.2 Freundlich isotherm

Freundlich gave an empirical expression representing the isothermal variation of adsorption of a quantity of gas adsorbed by unit mass of solid adsorbent with pressure. Freundlich adsorption Isotherm can be calculated by the following formula;

$$X / m = KF\, C_e^{1/n}$$

$$\text{Log } x/m = \log KF + 1/n \log Ce$$

Where, X/m = Amount of dye adsorbed on unit weight of adsorbent at equilibrium.

Ce = Concentration of solute in aqueous solution.

The freundlich parameters KF and 1/n are indicators of adsorption capacity and adsorption intensity respectively. The magnitude of exponent n gives an indication of favourability.

3.14 Assessment of physical properties

The physical and mechanical properties of the fabrics are affected by the fabric type, yarn construction and fabric structure, as well as any treatment that have been applied to the materials. A range of fabric performance parameters are assessed for different end-use applications (Jinlian,2008).

3.14.1 Fabric weight

Weight measurement of a fabric is often prerequisite for subsequent tests of other fabric properties.

Weight can be determined by a mass per unit area or a mass per unit length of fabric. Specimens of known dimensions are taken by a cutting device(GSM cutter) or a template, to obtain a consistent specimen size. It is a device to cut circular specimen of 100 square centimeters of a fabric very accurately. It has 4 blades that cut the fabric, when the hand wheel is rotated by applying light pressure. Five specimens were selected from each fabric sample. Specimen selection should avoid taking samples from the fabric selvedge or close to the ends of a fabric piece. Care should be taken to avoid the loss of fibres/threads during weighing. Each specimens are weighed on an electronic balance as per the standards of ASTM D3776-96. The samples were weighed for five times and the mean value was calculated and recorded. The weight of original, fresh water dyed and decolourized water dyed and decolourized water dyed fabrics were determined

3.14.2 Fabric Thickness

The thickness of a fabric is one of its basic properties, giving information on its warmth, weight and stiffness. Thickness measurements are very sensitive to the pressure and sample size used in the measurement (Raul, 2005). Fabric thickness is defined as the distance between lower and upper surface of the material measured under a standard pressure, using Shirley's thickness tester with an accuracy of 0.01mm. Fabric thickness guage was used to measure thickness of the sample. It has 2 parts the anvil and pressure foot. Pressure was given at the foot to make the guage zero. The sample was placed between the cleaned pressure foot and anvil. The reading shown by the dial was noted. For each sample, thickness was determined at 5 different places away from 2 inch of the selvedge.

3.14.3 Fabric Strength

The strength of the fabric is important for all textile users. Measurement of tensile stress- strain properties is the most common mechanical measurement on fabrics. It is used to determine the behaviour of a sample while under an axial stretching load. The tensile properties measured are generally considered arbitrary rather than absolute. Results depends on specimen geometry, the fibre type and arrangement, as well as the fabric structure.

The original and dyed fabrics were tested for tensile strength using Eureka Cloth tensile strength tester. 12 inch x 2 inch specimen from each samples were cut both in warp and weft direction of the fabric, 2 inches apart from selvedge. The specimen was placed between the upper and lower clamp. The dial reading was set to zero by adjusting the pendulum over the quadrant scale. The elongation pointer was checked for its position in zero. Before starting the machine the pendulum lock was released and machine was stitched to run. At certain point the fabric starts to break, the machine was switched off and the dial reading in kg was noted and recorded.

3.14.4 Fabric Elongation

Elongation is defined as the change in length of a material due to stretching. When a fabric of original length is stressed along its axis, it extends an amount. Strain is a dimensionless quantity, often reported as a percentage.

Elongation at load is the amount, that a fabric stretches in length after a fixed load is applied. This is commonly used to define the level of stretch within the fabric. The elongation is noted as the increase in inches (Chen, H.,2005) Elongation measures the extent of deformation along the axis of a material under a tensile stress, and expressed in units of length of the fabric when loaded.

Elongation reading was noted from the elongation scale when the strength of the fabric is tested. The specimen was removed and the machine positioned back to original and the five specimens of both directions from each samples were tested and readings were noted.

3.14.5 Fabric Stiffness

A stiffness test measures the severity of the flexing action of a material. The bending length is a measure of the interaction between fabric weight and fabric stiffness in which a fabric bends under its own weight. It reflects the stiffness of a fabric when bent in one place under the force of gravity and is one component of drape. Thus bending length is also called drape stiffness.

The Shirley's stiffness tester, a rectangular strip of fabric 6 inch ×1inch is mounted on a horizontal platform in such a way that it overlays, like a cantilever and bends downwards. The length of fabric that will bend under its own weight to a definite extent (Saville, 2002). The sample was placed on the platform with the template at the top of it, so that the leading edges coincide. Both were slowly pushed forward until the leading edges of the sample and the template project beyond the edge of the platform. The sliding of the sample was stopped when it cut both the index lines. Then the bending length of the sample read from the scale opposite a datum line engraved on the side of the platform. Four readings were taken for sample A. Mean values of the bending length in warp and weft wise direction was calculated. Similarly other samples were determined.

3.14.6 Absorbency tests

The pressure required to force water through a fabric may be determined and the information used is the assessment of the fabrics ability to do a particular job (Raul, 2005).

3.14.6.1 Fabric Sinking

This involves a simple test of wettability of fabric. In this test, a small square specimen about 1inch × 1inch was cut and dropped in to the surface of water in a beaker. The time taken for the specimen to sink below the surface was observed. The shorter the time, the greater the wettability

3.14.6.2 Fabric Wicking

The wicking or capillary travel method measures the rapidity of absorption. 5 pieces of a sample were cut measuring 15 cm length and 2.5 cm width. One end of the sample strip was pasted with a glass rod which was placed on heavy wooden blocks and, at the other end two grams weight was attached to keep the sample straight. At the weighed end 2 cm of the sample was allowed to immerse in a tray of distilled water. The rise of the water level in the strip was noted by keeping time as constant (1 minute). The same procedure was repeated for all the samples and the mean value was calculated and recorded.

3.13.7 Colour Fastness Test

Colour fastness is a term used in the dyeing of textile materials, meaning resistance of the material's colour to fading or running. The term is usually used in the contact of clothes. In general, clothing should be tested for colour fastness before using bleach or other clearing product (Oger, 2010).

Colour fastness properties, of the dyed textile material depend upon the nature of dye, dyeing procedure, washing methods and after treatment methods. The grey scale employed for colour fastness test is 1-5 grade. In this scale one means poor fastness and 5 represent excellent colour fastness reports.

In this study, 4 colour fastness tests were carried out. They are colour fastness to sunlight, washing, wet and dry crocking, wet and dry pressing.

3.13.7.1 Fastness to Sunlight

The colour fastness of textile material to day light is very important property. Colour fastness determine the resistance of a material to change its colour characteristics as a result of exposure light source views AATCC (2008).

To test the fastness to sunlight the specimens of 16 cm x 5 cm were cut from each sample of dyed fabrics and divided into 8 equal parts measured as 20m each, the specimens were covered with black chart. For the successive 7 days the specimens were exposed to direct sunlight. First day first portion of the specimens were exposed and accordingly seven portions are exposed to Sunlight. The first portion was exposed from 7 days and the 7th portion was exposed for a day. The last portion was not exposed to sunlight and considered as standard. Comparisons were done using grey scale and the specimens were rated.

3.13.7.2 Wet and Dry Crocking

Crocking is the rubbing fastness of dyes. Crocking is the transfer of colourant from the surface of the coloured fabric to an adjacent area of sample fabric or to another surface, principally by rubbing action. Fastness to crocking is important in both apparel as well as upholstery. crocking test determine the extent to which colour may be transferred from the surface of the dyes fabric to another by rubbing.

Sasmira Crock meter was used to determine the fastness to crocking. Each of dyed samples was cut in the measurement of 25 cm x 20 cm and mounted on flat base. The desized white cotton fabric was mounted in a ring on rubbing finger. Each sample was given 10 rubs based on standardization. The colour transfer from the dyed sample to the white material was used for wet crocking. The procedure adopted was same as that of dry crocking. The color transfer from the dyed sample to the white material was assessed using grey scale.

3.13.7.3 Fastness to washing

Major loss of colour from the fabric is due to washing and results in staining over the adjacent fabric. Test sample of the dyed fabric measuring 5*10cm size were cut. Each of them was sandwhiched between the undyed white cloth which was desized well. Specimen were completely soaked in the soap solution about 5g/l for 30 minutes at 40^0C, after that the samples were removed, rinsed in cold water thoroughly, squeezed well and dried. Evaluations of staining on the white adjacent fabrics were found using a grey scale. The same procedure was carried out for other dyed samples.

3.13.7.4 Wet and dry Pressing

Colour fastness of the samples to pressing was measured following the specifications of Bureau of Indian Standards (2000). Two specimens measuring 10 cm x 10 cm from each dyed samples were cut and one set of specimens covered at either side with 5 cm x 5 cm of desized white fabric. The prepared specimens were pressed for 5 seconds to assess its color fastness to dry pressing, while the others were covered with wet white cloth and pressed for 5 seconds to assess its color fastness to wet pressing. The same procedure was repeated for 5 specimens. The colour change in the dyed fabrics was graded using grey scale.

4. RESULTS AND DISCUSSION

4.1 Screening of different adsorbents for decolourization of selected dyes

4.2 Optimization of various parameters for the decolourization of selected dyes

- 4.2.1 Optimization of adsorbent dosage
- 4.2.2 Optimization of initial dye concentration
- 4.2.3 Optimization of temperature
- 4.2.4 Optimization of time
- 4.2.5 Optimization of pH

4.3 UV-Vis spectral analysis

4.4 Isotherm studies

- 4.4.1 Langmuir isotherm
- 4.4.2 Freundlich adsorption isotherm

4.5 SEM (Scanning Electron Microscopic) analysis

4.6 FTIR (Fourier Transform Infra Red) analysis

4.7 Evaluation of dyed fabric

- 4.7.1 Fabric Weight
- 4.7.2 Fabric Thickness
- 4.7.3 Fabric Strength
- 4.7.4 Fabric Elongation
- 4.7.5 Fabric Stiffness
- 4.7.6 Absorbency test
  - 4.7.6.1 Fabric Wicking
  - 4.7.6.2 Fabric Sinking
- 4.7.7 Colour fastness tests

4.1 Screening of different adsorbents for decolourization of selected dyes

The decolourization efficiency of different adsorbents such as sea foam, sugarcane bagasse, corn stalk, garlic peel, sunflower stalk, tea waste, tapioca peel, pea shell, orange peel, egg shell were screened and the results are presented in Table 5 and Figure 2.

Table 5

Screening of selected adsorbents for the decolourization of selected dyes

Adsorbents	**Percent decolourization**	
	Basic yellow 13	**Acid red 51**
Sea foam	2.43	46.12
Sugarcane bagasse	79.44	52.15
Corn stalk	83.62	61.20
Garlic peel	40.41	39.22
Sunflower stalk	**85.71**	**74.13**
Tea waste	65.15	33.62
Tapioca peel	78.39	44.82
Pea shell	17.07	41.81
Orange peel	6.24	4.56
Egg shell	12.54	42.67

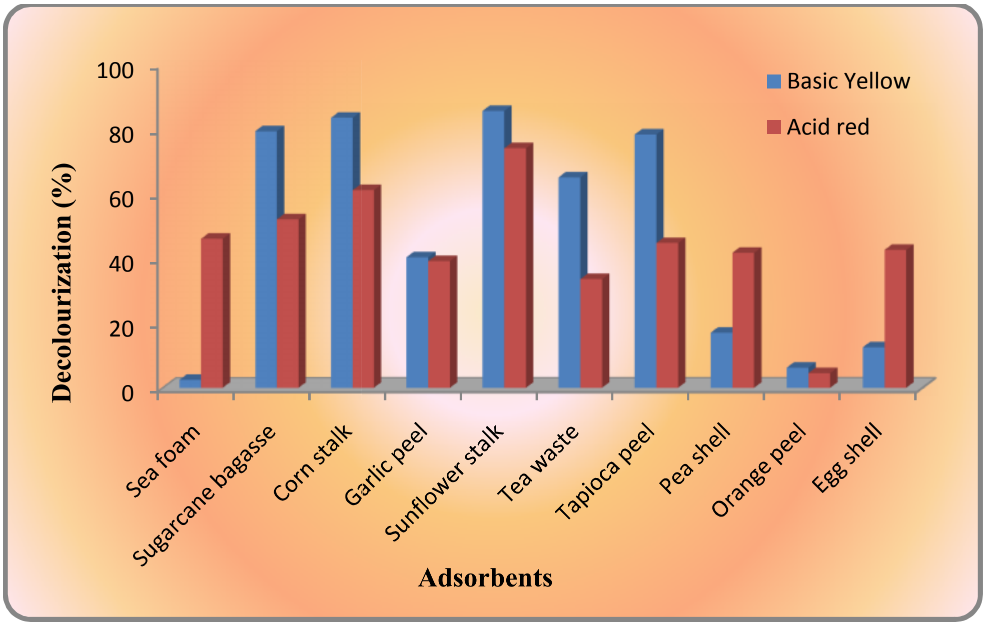


Figure 2

Screening of selected adsorbents for the decolourization of selected dyes.

Among the selected adsorbents, sunflower stalk powder showed maximum decolourization percentage for basic yellow 13 and acid red 51. The percent decolourization observed for basic yellow 13 is 85.71 and acid red 51 is 74.13 respectively. Hence sunflower stalk was selected as potent adsorbent for the decolourization of selected dyes.

4.2 Optimization of various parameters for the decolourization of selected dyes

4.2.1 Optimization of adsorbent dosage

The adsorbent dosage is an important parameter in order to determine the adsorbent's capacity for a given amount of the adsorbate at the operating conditions. The effect of adsorbent concentration for the decolourization of selected dyes was studied and the results are presented in Table 6 and Figure 3.

Table 6

Optimization of Adsorbent dosage

Adsorbent concentration (%)	Percent decolourization	
	Basic yellow 13	Acid red 51
1	81.88	75
2	83.62	77.15
3	86.06	78.44
4	**88.50**	**82.32**
5	87.10	81.03

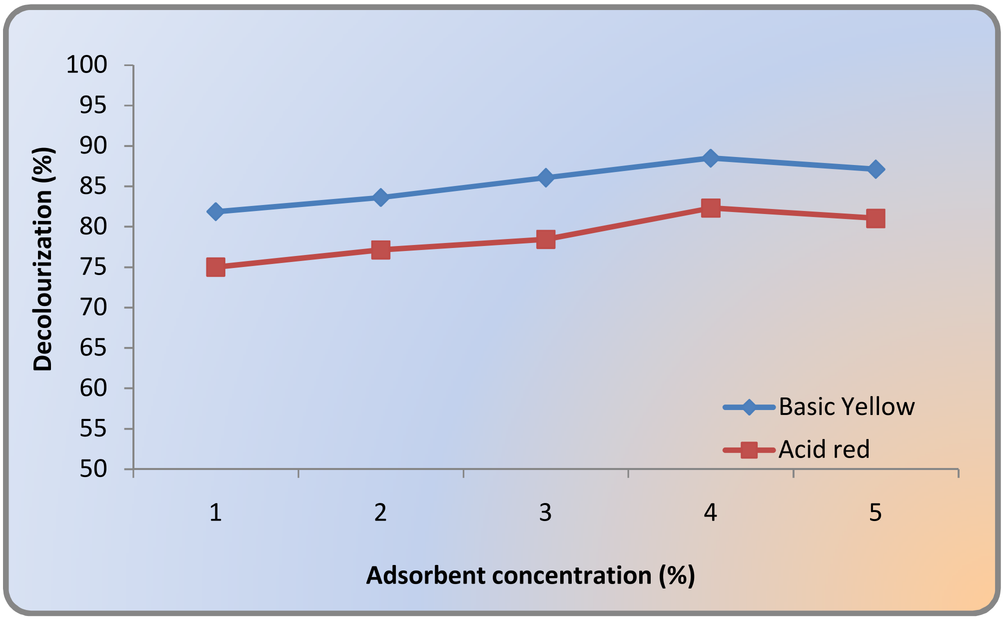


Figure 3

Optimization of Adsorbent dosage

Table 6 and Figure 3 clearly shows that the percent dye decolourization, increase with increase in adsorbent concentration from 1% to 5%. Maximum percent decolourization was observed at 4% for both basic yellow 13 (88.50%) and acid red 51 (82.32%). Hence adsorbent concentration of 4% was selected as optimum adsorbent dosage for decolourization and fixed for further experiments.

The increase in percent dye removal with increasing adsorbent dosage, might be due to increase in the amount of sorption sites at the surface of adsorbent with increasing dose of adsorbent (Ofomaja, 2008). Hassani et al.(2015) have reported that increasing adsorbent dosage will provide more surface area, thereby leading to more binding sites for the adsorption of dyes.

4.2.2 Optimization of initial dye concentration

The dye removal efficiency is highly dependent on the initial dye concentration. The effect of initial dye concentration on the decolourization of selected dyes was studied and the results are presented in Table 7 and Figure 4.

Table 7

Optimization of Initial Dye concentration

Initial Dye concentration (%)	**Percent decolourization**	
	Basic yellow 13	**Acid red 51**
0.01	**97.90**	**89.65**
0.02	94.07	88.36
0.03	84.66	86.20
0.04	80.13	84.48
0.05	78.04	81.03

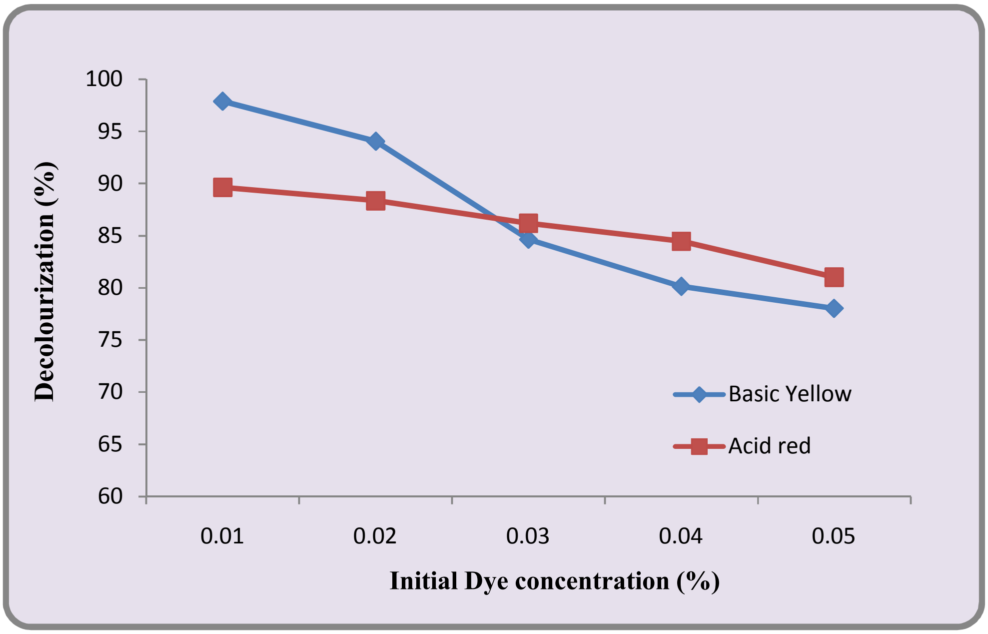


Figure 4

Optimization of Initial Dye concentration

The Table 7 and Figure 4 clearly shows that increase in initial dye concentration lowers the dye decolourization percentage. Maximum dye removal was observed at an initial dye concentration of 0.01% for both basic yellow 13 (97.90%) and acid red 51 (89.65%) respectively. Hence an initial dye concentration of 0.01% was fixed as optimum and used for further experiments.

The effect of initial dye concentration relies on the immediate relation between the dye concentration and the available binding sites on the adsorbent surface. The removal efficiency will decrease with an increase in the initial dye concentration due to the saturation of adsorption sites on the adsorbent surface. There will be unoccupied binding sites on the adsorbent surface at a low dye concentration, and when the initial dye concentration increases, there will be insufficient sites for the adsorption of dye molecules, thus decreasing the dye removal efficiency (Salleh et al, 2011). On the other hand, the increase in initial dye concentration will cause an increase in the loading capacity of the adsorbent and this may be due to the high driving force for mass transfer at a high initial dye concentration (Bulut et al, 2006).

4.2.3 Optimization of temperature

The effect of temperature on decolourization of selected dyes were examined at various temperatures such as 30^0C, 40^0C,50^0C,60^0C and 70^0C and the results are presented in Table 8 and Figure 5.

Table 8

Optimization of Temperature

Temperature (ºC)	Percent decolourization	
	Basic yellow 13	**Acid red 51**
30	72.12	91.37
40	**94.77**	93.53
50	94.03	92.24
60	92.33	**89.22**
70	91.63	87.5

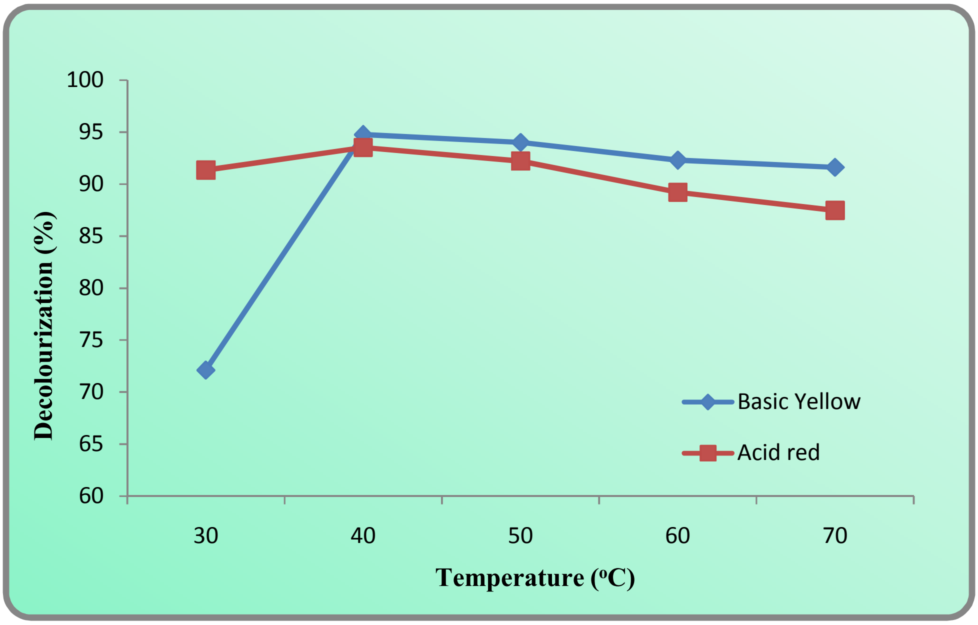


Figure 5

Optimization of Temperature

The Table 8 and Figure 5 clearly shows that both exothermic and endothermic processes occurs based on the dye molecules. Basic yellow 13 dye showed exothermic process with maximum dye removal at 40^0C, and the percentage of removal was found to be 94.77. Acid red 51 dye exhibited endothermic process with maximum dye removal at 60^0C, and the percentage of removal was 89.22. Hence, an optimum temperature of 40^0C and 60^0C was selected for basic yellow 13 and acid red 51 respectively for decolourization studies.

Temperature is an important factor that serves as an indicator as to whether the adsorption is an exothermic or endothermic process. The process of exothermic or endothermic occurs based on the dye molecule. If the adsorption is an endothermic process, the adsorption capacity will increases with increasing temperature. This may possibly due to the increase in the number of active sites and the mobility of the dye molecules at higher temperature. In contrast, if the adsorption is an exothermic process, the adsorption capacity will decrease with increasing temperature. In this case, higher temperature may decrease the adsorptive forces between the dye molecules and the active sites on the adsorbent surface (Ofomaja, 2008).

4.2.4 Optimization of time

In adsorption studies, contact time plays a vital role. Effect of contact time on decolourization of selected dyes was examined at various time interval such as 2, 4, 6, 8 and 10 hrs and the results are presented in Table 9 and Figure 6.

Table 9

Optimization of Time

Time (hrs)	**Percent decolourization**	
	Basic yellow 13	**Acid red 51**
2	73.17	87.06
4	**94.77**	88.79
6	88.50	91.8
8	85.36	**93.96**
10	80.13	90.08

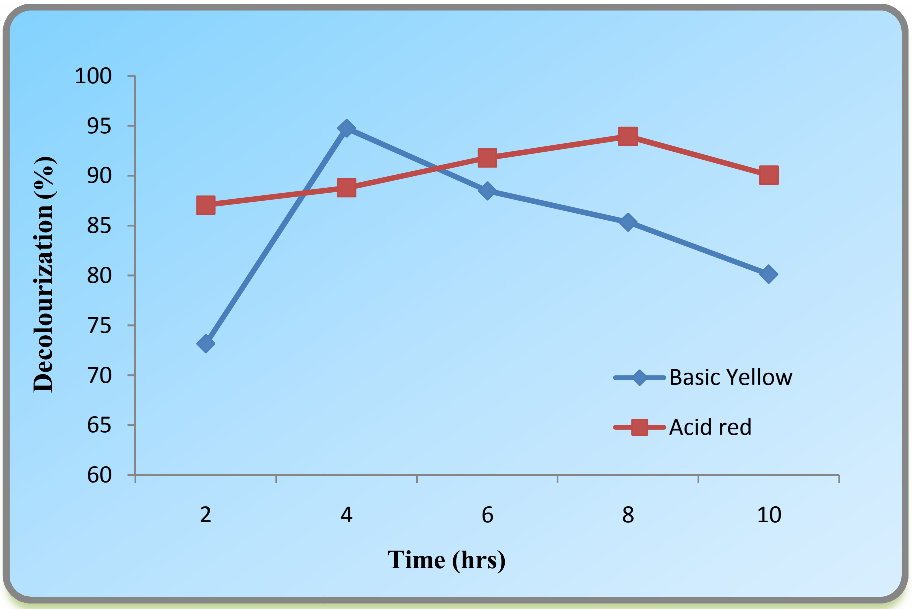


Figure 6

Optimization of Time

Table 9 and Figure 6 clearly indicates that maximum percent dye removal was obtained when incubated for 4 hrs for basic yellow 13 and 8 hrs for acid red 51 respectively. When the contact time was increased above 4 hrs, with respect to basic yellow 13 the percent decolourization was found to be reduced from 94.77% to 80.13%. In acid red 51 reduction in percent decolourization was found to be from 93.66% to 90.08%. Hence, an optimum time of 4 hrs and 8 hrs was selected for basic yellow 13 and acid red 51 respectively for decolourization studies.

The observed findings correlated with Jirekar et al.,(2014), who have reported that, at initial stage percentage removal of dye was rapid and becomes slow and gets stagnated with increase in time.

4.2.5 Optimization of pH

pH is a important parameter in the adsorption process, particularly for dye adsorption. Effect of pH on decolourization of selected dyes was examined at various pH such as 5, 6, 7, 8 and 9 and the results are presented in Table 10 and Figure 7.

Table 10

Optimization of pH

pH	Percent decolourization	
	Basic yellow 13	Acid red 51
5	81.53	**91.37**
6	91.63	89.65
7	**93.37**	88.79
8	92.68	86.20
9	92.33	85.34

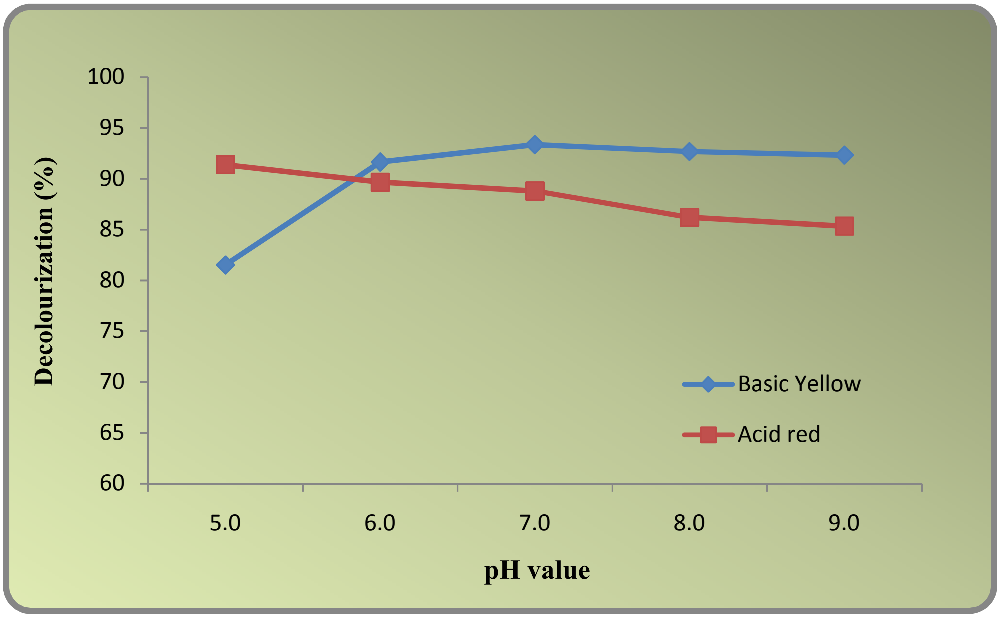


Figure 7

Optimization of pH

From Table 10 and Figure 7 it is clear that, percent decolourization was maximum at pH 5 for acid red 51 (91.37%). This might be due to the anionic dye adsorption which increases at low pH because the adsorbent is acting as a positively charged surface. Hence, an optimum pH of 5 was selected for acid red 51. The percent decolourization was found to be maximum at pH 7 for basic yellow 13 (93.37%). An optimum pH of 7 was selected for basic yellow 13.

Dawood and Sen (2012) studied the effect of solution pH on the adsorption of Congo red by pine cone and they noticed that the adsorption was maximum at pH 5. Another study conducted by Aksu and Isoglu (2006) reported the effect of solution pH on the adsorption of Gemazol turquoise blue-G a reactive dye using sugar beet pulp and they noticed that the adsorption was at maximum at pH 2 where the adsorption capacity was 83.7 mg/g and then decreased with a further increase in pH and reached zero at pH 6.

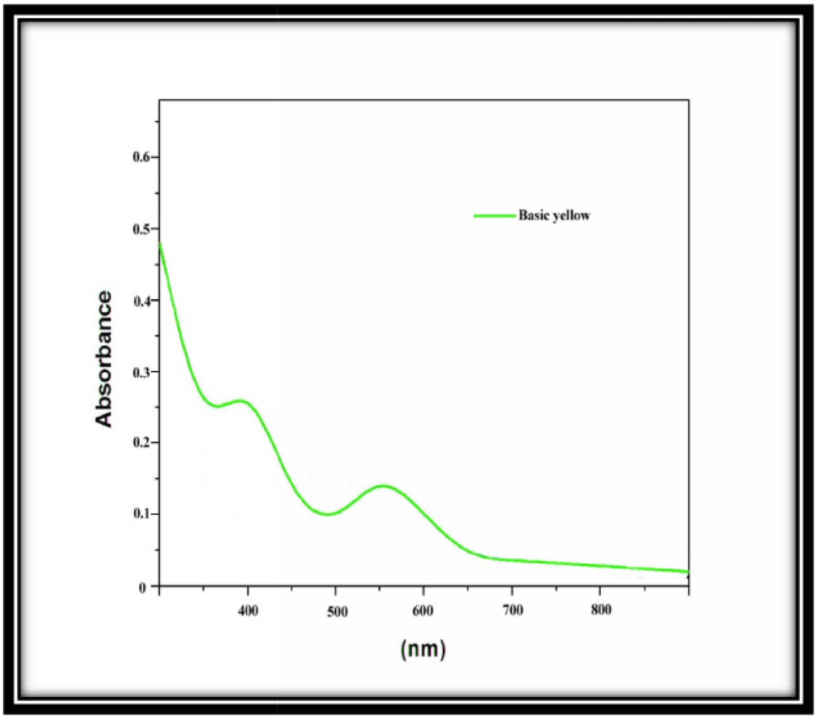
The magnitude of electrostatic charges which are imparted by the ionised dye molecules is controlled by the solution pH. As a result the rate of adsorption will vary with the pH of the medium used. At low solution pH, the percentage of dye removal will decrease for cationic dye adsorption, while for ionic dyes the percentage of removal will increase. In contrast, high solution pH is preferable for cationic dye adsorption but shows a lower efficiency for anionic dye adsorption. At high solution pH, the positive charge at the solution interface will decrease while the adsorbent surface appears negatively charged. As a result, the cationic dye adsorption will show an increase and the anionic dye adsorption will decrease. At low pH solution, the positive charge on the solution interface will increase and the adsorbent surface will appear positively charged, which results in decrease in cationic dye adsorption and an increase in anionic dye adsorption (Salleh et al, 2011).

4.3 UV-Vis spectral analysis

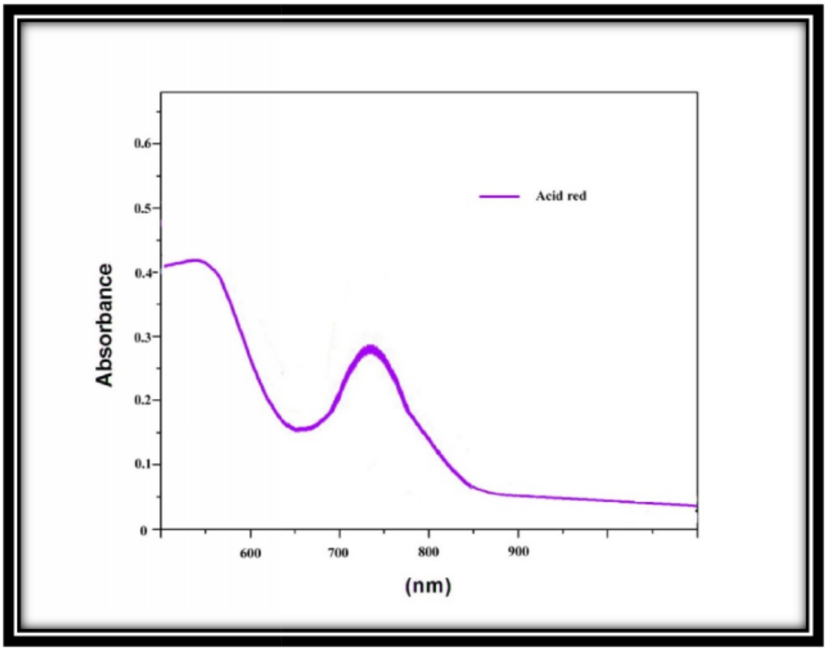
UV-Vis spectral analysis is used to determine the optical density and to confirm the degradation processes of dye that was due to decolourization. Spectrophotometric analysis of dye solution showed maximum absorbance at visible range of 432 nm in basic yellow 13 dye (Figure 8. a) and 509 nm in acid red 51 dye (Figure 8. b). The decolourized dye solution showed maximum adsorbance in UV range 127 nm in basic yellow 13 and 230 nm in acid red 51. The dye removal is attributed to the degradation when the major visible light absorbance peak would be completely disappear or a new peak will appear (Battacharya et al.,2005). Results indicate that the peaks in the visible region have been shifted to UV region which rules the degradation of the dye by the adsorbent.

Figure 8

UV-Vis spectral Analysis



a.UV-Vis spectral analysis of basic yellow 13



b. UV-Vis spectral analysis of acid red 51

4.4 Isotherm studies

Two types of isotherms have been investigated, namely Langmuir and Freundlich isotherm.

4.4.1 Langmuir isotherm

Langmuir adsorption isotherm model was applied to estimate the adsorption capacity of the adsorbents. Hence the model was studied and the results are presented in Table 11.

TABLE 11

LANGMUIR ADSORPTION ISOTHERM

Adsorbent Dosage (g)	Time(hrs)	Dye left (C_e)	$1/C_e$	m/x	Slope	Intercept	Separating factor R_L
Basic yellow 13	2	7.31	0.136	292.7			0.981
	4	9.47	0.105	379.12			0.216
	6	8.85	0.112	354.04	2754.1	666.31	0.111
4	8	8.53	0.117	341.48			0.989
	10	8.01	0.1247	320.56			0.986
Acid red	2	1.23	0.772	348.24			0.752
51	4	1.12	0.892	355.16			0.778
	6	0.81	1.221	367.24	29.958	328.11	0.827
4	8	0.60	1.655	375.84			0.867
	10	0.99	1.008	360.32			0.798

The R_L Value indicates the shape of isotherm. The R_L value obtained was below one i.e. $R_L<0.99$.This proves the feasibility of the adsorbent for decolourization and indicate favourable adsorption isotherm.

4.4.2 Freundlich adsorption isotherm

The Freundlich parameters K_F and 1/n indicates the adsorption capacity and values are evaluated from the slope and intercept respectively. With the log C_e and log x/m, the slope and intercept are calculated. Hence the model was studied and the results are presented in Table 12.

TABLE 12

FREUNDLICH ADSORPTION ISOTHERM

Adsorbent Dosage (g)	Time (hrs)	log C_e	log x/m	Intercept	Slope 1/n	n
Basic yellow 13 4	2	0.863	0.533	0.992	1.396	5.13
	4	0.976	0.421			
	6	0.946	0.450			
	8	0.930	0.466			
	10	0.903	0.494			
Acid red 51 4	2	0.110	0.458	0.637	0.522	6.17
	4	0.049	0.898			
	6	0.091	0.425			
	8	0.221	0.424			
	10	0.004	0.443			

The n values range between 2 and 10 indicates favourable adsorption. The n value obtained was 5.13 for basic yellow 13 and 6.17 for acid red 51 indicating the applicability of freundlich isotherm is favourable for the present study.

4.5 Scanning Electron Microscopic analysis

The control sunflower stalk and sunflower stalk obtained after decolourization with basic yellow 13 and acid red 51 dyes were analyzed for structural morphology using Scanning Electron Microscope (SEM) and the results are presented in Figure 9.

Figure 9

SEM analysis

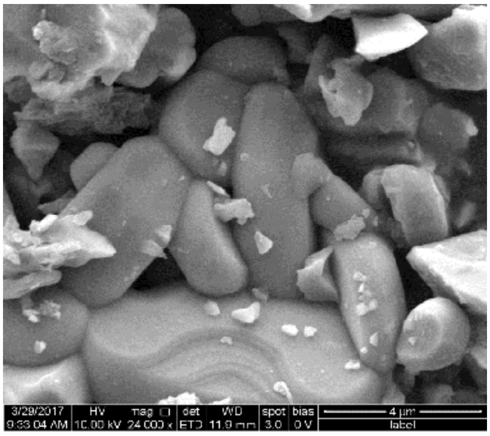


Figure 9. a. Sunflower stalk.

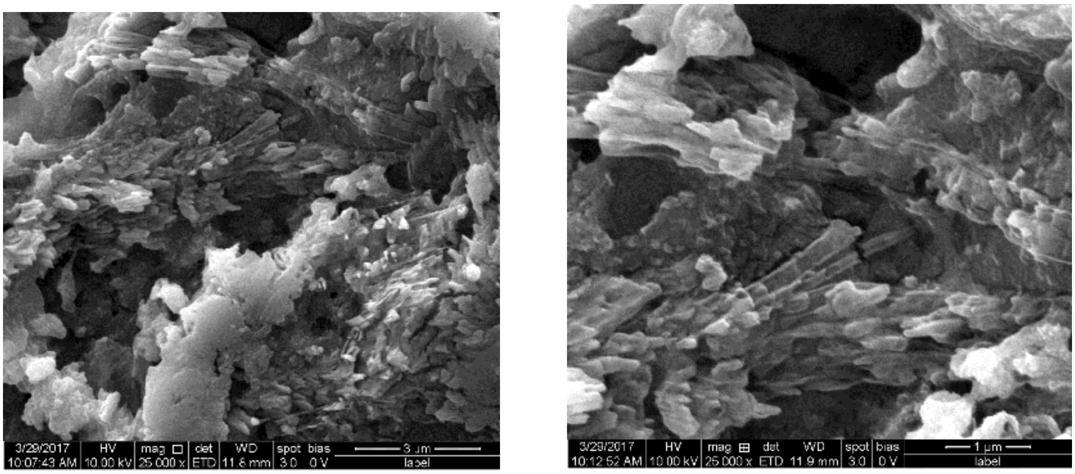


Figure 9.b Sunflower stalk (after decolourization of basic yellow 13 dye).

Figure 9.c Sunflower stalk (after decolourization of acid red 51).

It is clear from the Figure 9, that sunflower stalk after decolourization in both dyes show very distinguished dark spots and change in surface morphology which can be effectively taken as a sign for effective adsorption of dye molecules in the cavities and pores of this adsorbent.

4.6 FTIR (Fourier Transform Infra Red) analysis

FTIR adsorption spectrum of the dried *Helianthus annuus* stalk before and after decolourization can offer information regarding the chemical change of the functional groups involved in adsorption. FTIR spectrum shows different major absorbance band peaks positioned at 3452.58064, 2322.29446, 1365.60173, 763.81 in raw sunflower stalk (before decolourization). Among them the absorbance band 3452.58064 was associated with O-H stretch, H- bond with functional group of alcohols and phenols, band 2322.29446 shows -C=C- stretch with functional group of alkynes, band 1365.60173 shows C-H rock with functional group of alkanes, band 763.81 shows C-Cl stretch with functional group of alkyl halides showed in Figure 10 (a).

After decolourization there is a shift in band ranges in basic yellow 13 dye. The major absorbance band peaks was positioned at 3196.04811, 1535.33754, 1388.74752, 675.0856. Among them, the absorbance band 3196.04811 was associated with -C≡C-H: C-H stretch with functional group of alkynes (terminal), band 1535.33754 was associated with N-O asymmetric stretch with functional groups of nitro compounds, band 1388.74752 was associated with C-H bend with functional groups of alkanes, band 675.0856 was associated with C-Br stretch with functional groups of alkyl halides showed in Figure 10 (b).

After decolourization there is a shift in band ranges in acid red 51 dye. The major absorbance band peaks was positioned at 3381.21445, 3026.3123, 1614.41899, 1394.53397, 908.47234, 704.0178. Among them, the absorbance band 3381.21445 was associated with N-H stretch with functional group of 1*, 2* amines and amides, band 3026.3123 was associated with =C-H stretch with functional groups of alkenes, band 1614.41899 was associated with N-H bend with functional groups of 1* amines, band 1394.53397 was associated with N-O symmetric stretch with functional groups of nitro compounds, band 704.0178 was associated with -C≡C-H: C-H bend with functional groups of alkynes showed in Figure 10 (c). Thus there is a change in absorbance bands of sunflower stalk after decolourization of dyes. Band intensity decreases after decolourization which proves the degradation of the selected dyes.

Figure 10

FTIR analysis

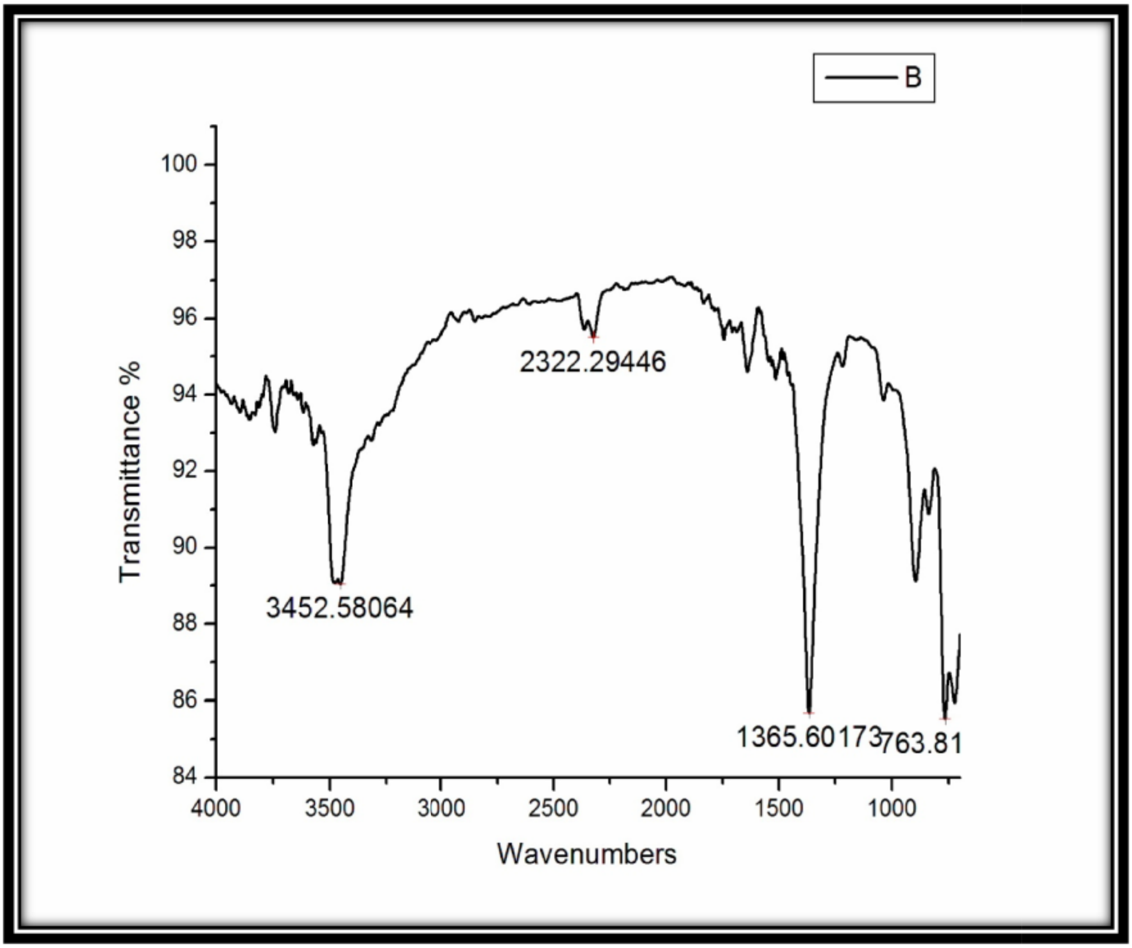


Figure 10 (a). FTIR analysis of sunflower stalk powder

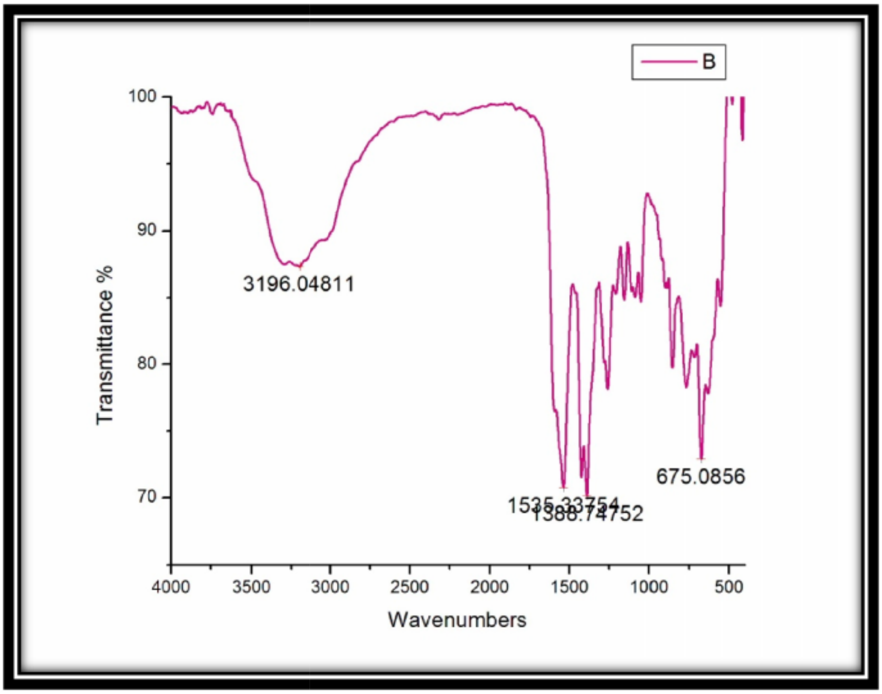


Figure 10(b). FTIR analysis of sunflower stalk powder after decolourization of basic yellow 13

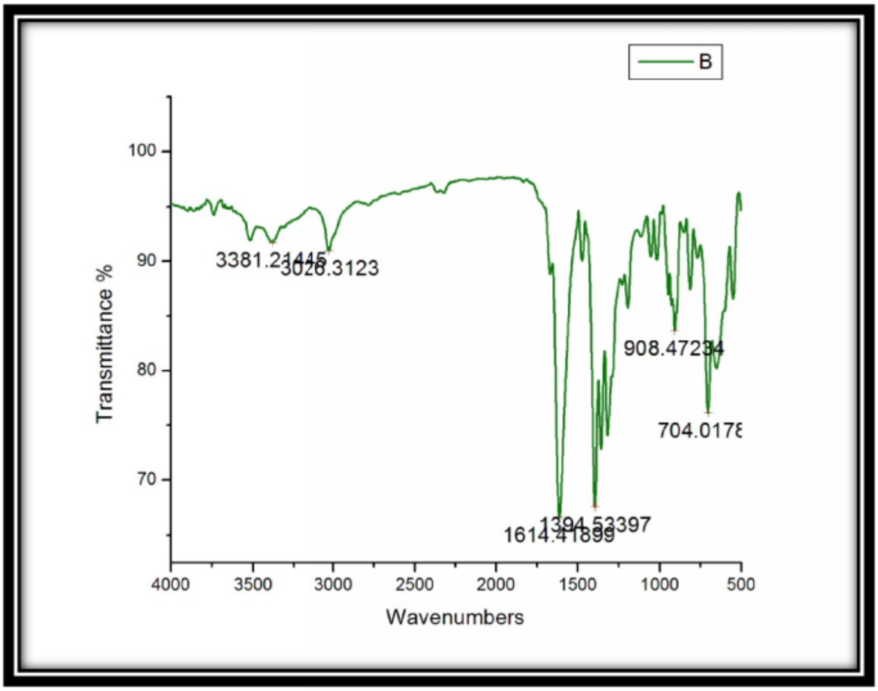


Figure 10(c). FTIR analysis of sunflower stalk powder after decolourization of acid red 51

4.7 Evaluation of dyed fabric

4.7.1 Fabric weight

The fabric weight and analysis of variance of the desized fabric (DF), fabric dyed with basic yellow 13 using fresh water (YDF), fabric dyed with acid red 51 using fresh water (RDF), fabric dyed with basic yellow 13 using decolourized water (YDD) and fabric dyed with acid red 51 using decolourized water (RDD) are given in Table 13 and Figure 11.

Table 13

Fabric Weight

S.No	Samples	Mean (GSM)	Loss /gain	Loss / gain %	F value
1	DF	1.19			22.406**
2	YDF	1.31	0.12	10.08	
3	YDD	1.36	0.17	14.28	
4	RDF	1.33	0.14	11.76	
5	RDD	1.42	0.23	19.32	

Values are mean of five readings

** - Significant at 1% level (p<0.01)

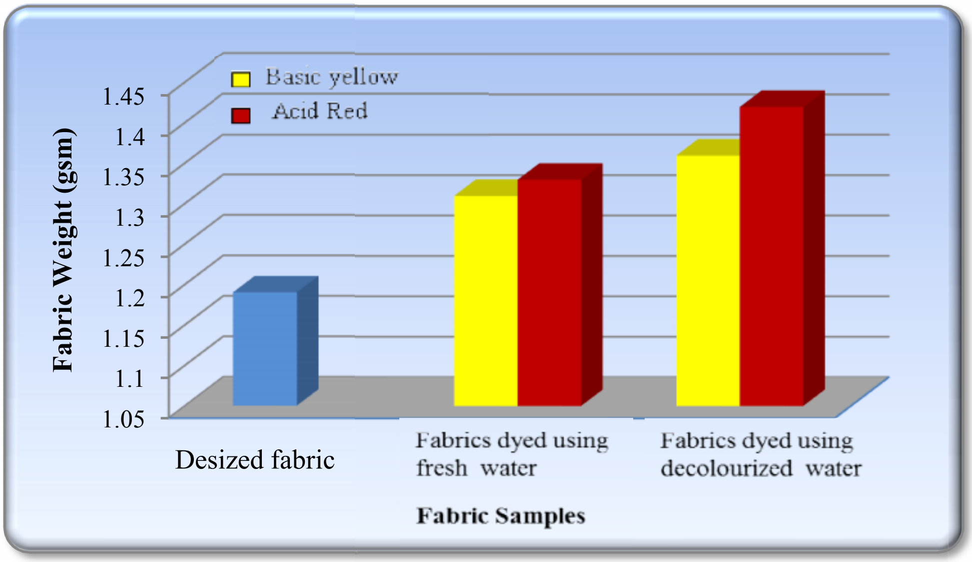


Figure 11

Fabric Weight

From Table 13 and Figure 11, it is clear that the weight of all the dyed samples increased after dyeing when compared to their desized fabric. The fabrics dyed using decolourized water exhibited higher fabric weight when compared to fabric dyed using fresh water. Statistical analysis proved that there was a significant difference at 1% level when compared between the dyed samples. The observed results support that the treated water can be effectively used for dyeing cotton fabric.

4.7.2 Fabric Thickness

Thickness and analysis of variance of the samples DF, YDF, RDF, YDD and RDD are presented in Table 14 and Figure 12.

Table 14

Fabric Thickness

S.No	Samples	Mean (mm)	Loss/ gain	Loss/gain %	F value
1	DF	0.32			16.036**
2	YDF	0.39	0.07	21.87	
3	YDD	0.5	0.18	56.25	
4	RDF	0.42	0.1	31.25	
5	RDD	0.5	0.18	56.25	

Values are mean of five readings

** - Significant at 1% level (p<0.01)

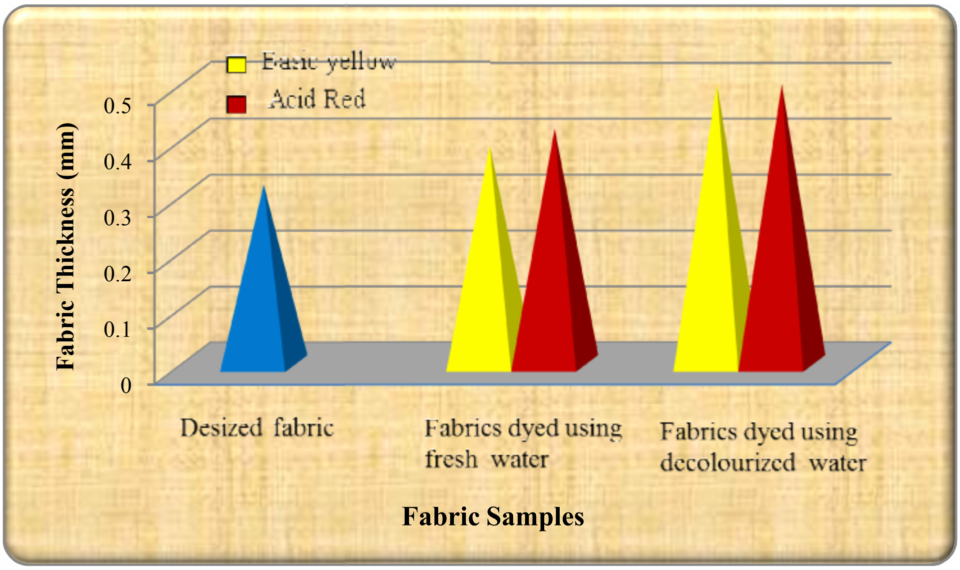


Figure 12

Fabric Thickness

From Table 14 and Figure 12, it is clear that the thickness of the dyed samples increased when compared to the original fabric. The percent increase in thickness was found to be maximum in fabrics dyed using decolourized water. The increase in thickness may be attributed to increased uptake of dye. Statistical analysis proved that there was a significant difference at 1% level when compared between the dyed samples.

4.7.3 Fabric strength

Strength and analysis of variance of the samples DF, YDF, RDF, YDD and RDD are presented in Table 15 and Figure 13.

Table 15

Fabric Strength

S.No	Samples	Mean (kg)	Loss/gain	Loss/gain %	F value
1	DF	55.6			12.882**
2	YDF	55	0.6	1.07	
3	YDD	54.6	1	1.79	
4	RDF	54.3	1.3	2.33	
5	RDD	55.4	0.2	0.35	

Values are mean of five readings

** - Significant at 1% level (p<0.01)

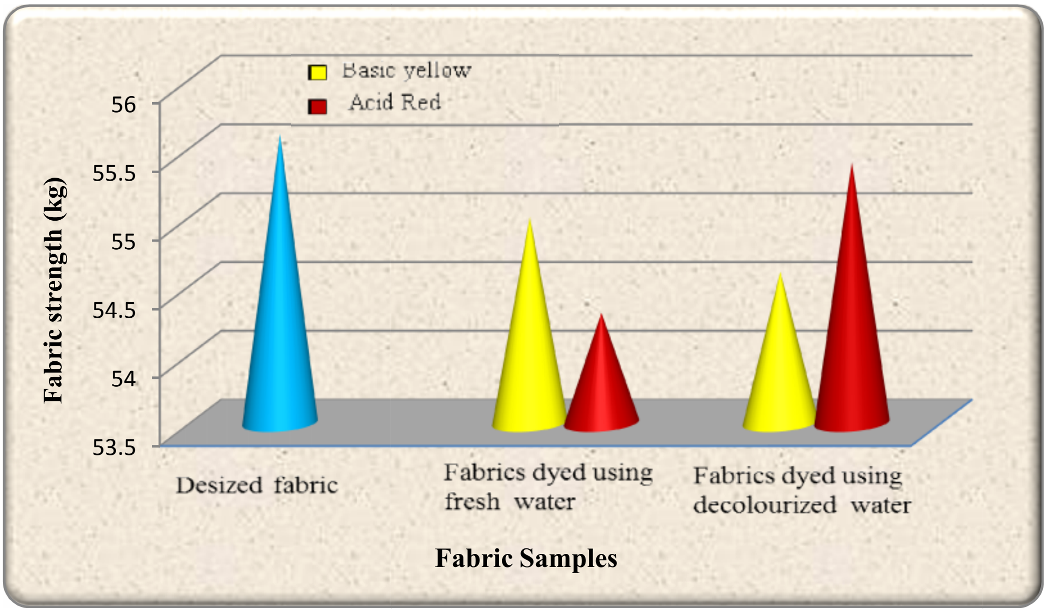


Figure 13

Fabric Strength

From Table 15 and Figure 13, it is clear that the strength of all the dyed samples decreased when compared to their desized fabric. Maximum decrease was found to be in sample RDF followed by YDD.

Statistical analysis proved that there was a significant difference at 1% level between the dyed samples. The obtained results showed that treated water could be effectively utilized for dyeing.

4.7.4 Fabric Elongation

Elongation and analysis of variance of the samples DF, YDF, RDF, YDD and RDD are presented in Table 16 and Figure 14.

Table 16

Fabric Elongation

S.No	Samples	Mean (inches)	Loss/gain	Loss/gain %	F value
1	DF	2			6.084**
2	YDF	2.4	0.4	20	
3	YDD	2.5	0.5	25	
4	RDF	2.9	0.9	45	
5	RDD	3.2	1.2	60	

Values are mean of five readings

** - Significant at 1% level (p<0.01)

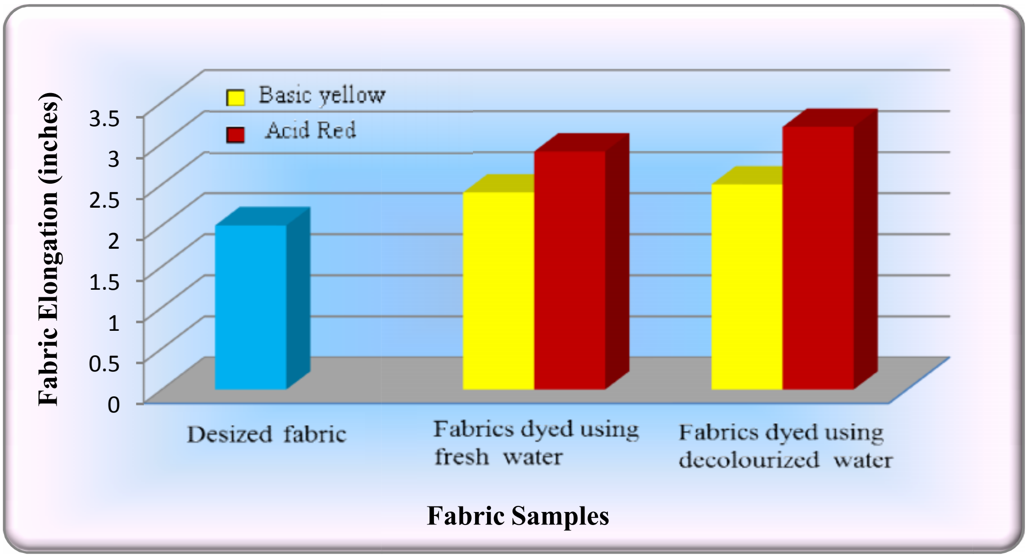


Figure 14

Fabric Elongation

From Table 16 and Figure 14, it is clear that the elongation of the dyed samples increased when compared to their original fabric. The percent increase in elongation for fabric dyed using fresh water was 20% in YDF and 45% in RDF, where as in fabrics dyed using decolourized water the percent increase is 25% in YDD and 60% in RDD samples.

Statistical analysis showed that there was a significant difference at 1% level between the dyed samples.

4.7.5 Fabric Stiffness

Fabric stiffness and analysis of variance of the samples DF, YDF, RDF, YDD and RDD are presented in Table 17 and Figure 15.

Table 17

Fabric Stiffness

S.No	Samples	Mean (cm)	Loss/gain	Loss/gain %	F value
1	DF	3.53			72.745**
2	YDF	3	0.53	15.01	
3	YDD	3.27	0.26	7.36	
4	RDF	2.46	1.07	30.31	
5	RDD	2.97	0.56	15.86	

Values are mean of five readings

** - Significant at 1% level (p<0.01)

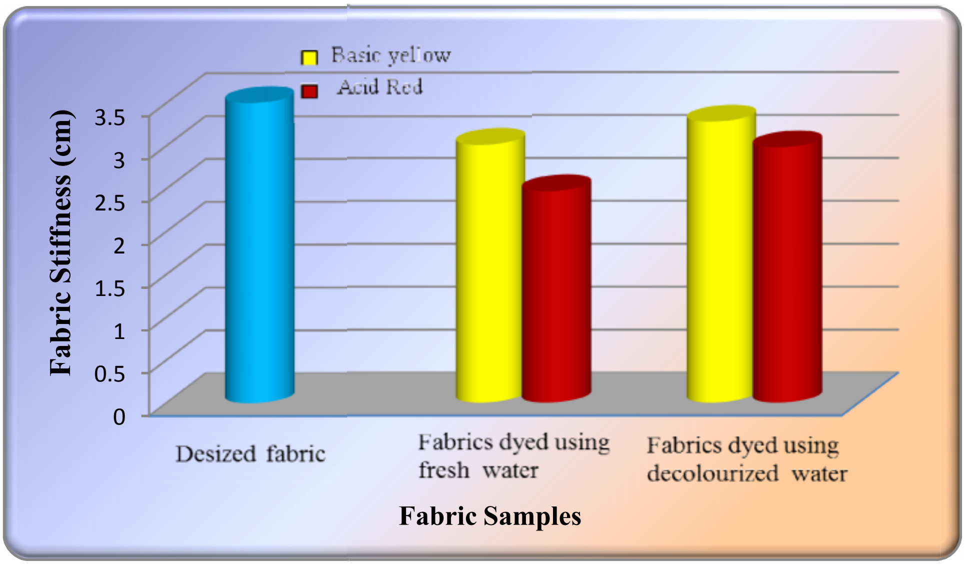


Figure 15

Fabric Stiffness

From Table 17 and Figure 15, it is clear that the stiffness decreased in all the dyed samples when compared to their original fabric. Maximum decrease was found to be in sample RDF (30.31%) followed by RDD (15.86%). There was a significant difference at 1% lavel when compared between the samples.

4.7.6 Absorbency test

4.7.6.1 Fabric Wicking

Wicking and analysis of variance of the samples DF, YDF, RDF, YDD and RDD are presented in Table 18 and Figure 16.

Table 18

Fabric Wicking

S.No	Samples	Mean (cm/min)	Loss/gain	Loss/gain%	F value
1	DF	3.4			5.975**
2	YDF	3.45	0.05	1.47	
3	YDD	3.53	0.13	3.82	
4	RDF	3.48	0.08	2.35	
5	RDD	3.5	0.1	2.94	

Values are mean of five readings

** - Significant at 1% level (p<0.01)

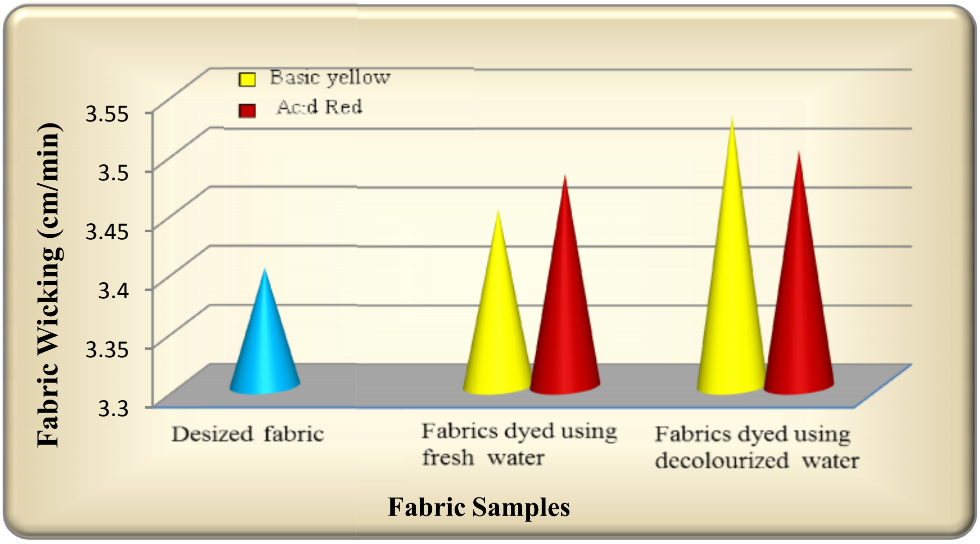


Figure 16

Fabric Wicking

From Table 18 and Figure 16, it is clear that the wicking of the dyed samples increased when compared to their desized fabric. Fabrics dyed using treated water exhibited higher absorbancy (wicking) of 3.82% in YDD and 2.94% in YDF and 2.35% in RDF when compared to fabrics dyed using fresh water.

4.7.6.2 Fabric Sinking

Sinking and analysis of variance of the samples DF, YDF, RDF, YDD and RDD are presented in Table 19 and Figure 17.

Table 19

Fabric Sinking

S.No	Samples	Mean (sec)	Loss/gain	Loss/gain %	F value
1	DF	3.45			6.674**
2	YDF	3.4	0.005	0.14	
3	YDD	3.41	0.04	1.15	
4	RDF	3.34	0.11	3.18	
5	RDD	3.33	0.12	3.47	

Values are mean of five readings

** - Significant at 1% level ($p<0.01$)

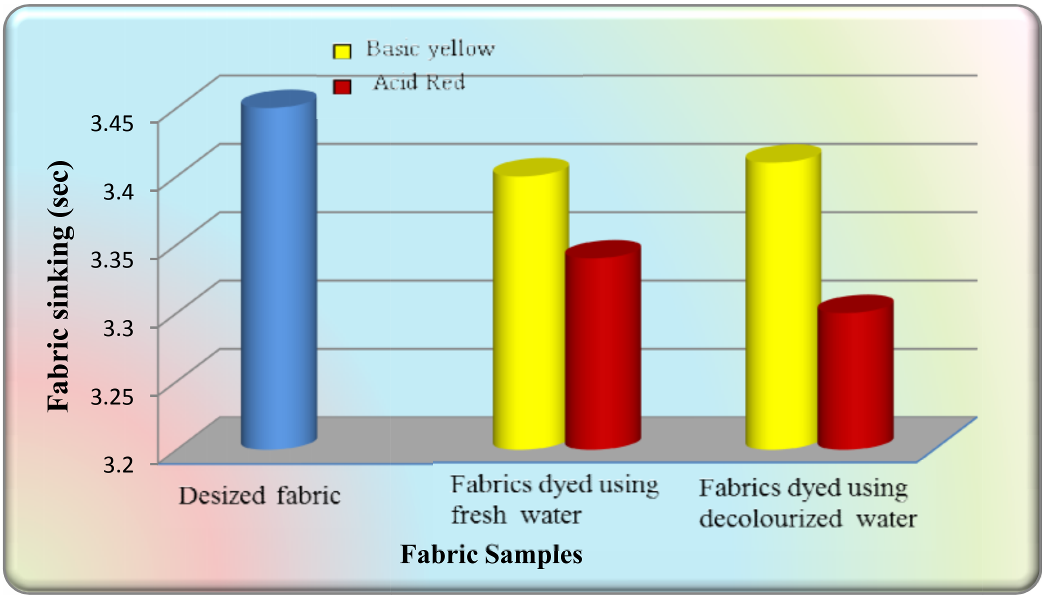


Figure 17

Fabric Sinking

From Table 19 and Figure 17, it is clear that the time taken for sinking of the dyed samples decreased when compared to their original fabric. Maximum decrease was found to be in sample RDD (3.47%) followed by RDF (3.18%).

Statistical analysis proved that there was a significant difference at 1% level between dyed samples. From wicking and sinking tests, it could be concluded that the absorbance of the fabrics dyed using treated water is on par with fabrics dyed using fresh water. Hence the decolourized water can be reused for dyeing.

4.7.7 Colour fastness tests

The results of colour fastness tests to washing, sunlight, pressing and rubbing of dyed fabric are presented in Table 20.

Table 20

Colour Fastness Tests

Samples	Washing	Sunlight	Rubbing		Pressing	
			Dry	Wet	Dry	Wet
YDF	4	4	5	4	5	4
RDF	4	5	4	4	5	5
YDD	5	4	5	4	5	4
RDD	4	4	5	4	5	4

5- Excellent 4- Good 3- Fair 2- Poor 1- Very poor.

From Table 20, it is evident that all the dyed samples showed good fastness to washing and sunlight. With regard to pressing all the samples recorded excellent to dry and good fastness to wet pressing. All the samples were found to exhibit good fastness to rubbing.

5. SUMMARY AND CONCLUSION

The Indian textile industry is one of the largest in the world with a massive raw material and textiles manufacturing base. Our economy is largely dependent on the textile manufacturing and trade in addition to other major industries. The textile industry accounts for 21% of the total employment generated in the economy. Around 35 million people are directly employed in the textile manufacturing activities. Indirect employment including the manpower engaged in agricultural based raw-material production like cotton and related trade and handling could be stated to be around 60 million. India's textile industry is also significant in a global context, ranking second to china in the production of both cotton yarn and fabric and fifth in the production of synthetic fibers and yarns.

Textile effluent includes a large variety of dyes and chemicals that create ecological challenge not only as liquid waste but also as chemical composite. Main pollutants in textile effluent comes from dyeing and finishing processes. Also dye baths could have high level of BOD/COD, colour, toxicity, surfactants, fibres and turbidity and may contain heavy metals. Major pollutants in textile wastewaters are high suspended solids, COD, heat, colour, acidity, and other soluble substances. Colour in the effluent is one of the most obvious indicators of water pollution. The discharge of highly coloured synthetic dye effluents is aesthetically displeasing and can damage the receiving water body by impending penetration of light.

Many synthetic dyes are harmful to human being, so the removal of colour from waste effluent is environmentally important. Several colour removal methods have been used for the decolourization of dye. Many of these methods are cost prohibited and suffered with one or other limitations. In recent years, there has been considerable interest in the use of biological by-products and residues from solution by adsorption.

Adsorption methods employing solid sorbents are widely used to remove certain classes of chemical pollutants from wastewater. Among these materials, agricultural by-products and biomass are relatively cheap and exhibit very high adsorption capacities. Agricultural by products are available in large quantities and are one of the most abundant renewable resources in the world. These waste materials have little or no economic value and often present a disposal problem. Therefore, utilisation of these low cost by-products for decolourization would help in their disposal problem.

The mechanism of dye adsorption on the agro-waste in colour removal process is similar to dyeing textile material which involve the following steps such as diffusion of dye molecules from the solution to the surface of adsorbents, adsorption of dye molecules on the surface of the materials through molecular interactions and diffusion of dye molecules from the surface into the interior of the adsorbent materials.

Many researches are inventing the new technologies to make a life to live easy and that makes a life easy but results in environmental hazards and polluting the lands with chemicals. Keeping that in mind, to reduce the pollution in environment the present study, “Decolorization of selected dyes in aqueous solution using *Helianthus annuus*” has been designed with following objectives.

- To screen various adsorbents for their efficiency in decolourizing the textile dye solution
- To optimize various parameters such as adsorbent dosage, initial dye concentration, time, temperature and pH for efficient removal of dye from aqueous solution
- To reuse the treated dye solution for dyeing the selected fabric
- To compare the properties of the fabrics dyed with fresh water and decolourized water

Experimental procedure

- Different adsorbents such as sea foam, sugarcane bagasse, corn stalk, garlic peel, sunflower stalk, tea waste, tapioca peel, pea shell, orange peel, egg shell were screened for their decolourization capacity for selected dyes
- Based on percent decolourization, sunflower stalk was selected for decolourization of basic yellow 13 and acid red 51 dyes
- Various experimental conditions like adsorbent concentration, contact time, pH, temperature and dye concentration were optimized for effective decolourization of selected dyes using sunflower stalk
- The colour intensity was measured using UV-Visible spectrophotometer
- Scanning Electron Microscopy was done for characterizing the surface morphology and fundamental physical properties of the adsorbent surface

- Fourier transform infrared spectrum analysis was carried out to determine the functional groups present in the adsorbent
- Adsorption isotherm studies were carried out using two adsorption models to describe how adsorbents interact with adsorbents
- In order to assess the reusability of decolourized water, it was utilized for dyeing selected fabric. Subjective and objective evaluation was carried out for the dyed samples

Findings

- Among different adsorbents screened, sunflower stalk was observed to be more efficient for the decolourization of selected dye
- An adsorbent concentration of 4% was found to be optimum for the decolourization of both the dyes basic yellow 13 and acid red 51
- An initial dye concentration of 0.01% was found to be optimum for both the selected dyes
- The optimum pH for decolourization of acid red 51 and basic yellow 13 was found to be 5 and 7 respectively
- Maximum percent decolourization was noticed at a temperature of 40^0C for both the dyes
- A contact time of 4 hours for basic yellow 13 and 8 hours for acid red 51 dye resulted in maximum decolourization
- Decolourization was carried out under optimized conditions. The maximum decolourization percent was found to be 97.90 in basic yellow 13 dye and 93.96 in acid red 51 dye
- After decolourization, the decolourized water was reused for dyeing the selected fabric
- Samples dyed using decolourized water was rated as good, bright and evenly dyed when compared with fresh water dyed sample
- Fabric weight of the samples, dyed using fresh and decolourized water increased when compared over original. It may be due to the result of dye uptake by the samples

- Increased thickness was observed in decolourized water dyed samples than when compared to fresh water dyed sample
- The strength of the dyed samples decreased in all samples when compared to their original fabric. Maximum decrease was found to be in sample RDF followed by YDD.
- Elongation of the dyed samples increased when compared to their desized fabric. The percent increase in elongation for fresh water dyed fabric was found to be 20% in YDF and 45% in RDF. The fabric dyed using decolourized water showed 25% and 60% increase in YDD and RDD sample respectively
- Dyeing has decreased the stiffness in both fresh water dyed and decolourized water dyed samples when compared over original sample
- Absorbance nature of the decolourized water dyed sample was found to be on par with fresh water dyed sample
- Samples subjected to colour fastness test exhibited excellent colour fastness properties. Fastness to sunlight, pressing and rubbing was rated as excellent for both the samples
- Scanning Electron Microscopic analysis showed that there was numerous pores in the selected adsorbent which enhances the entrapment of dye molecule
- Fourier Transform Infrared showed the presence of functional groups in the adsorbent
- Equilibrium studies fitted very well with both the Langmuir and freundlich isotherm model and they are in favourable level

Conclusion

The present study proved that sunflower stalk could be effectively utilized for decolourization of selected dyes and the decolourized water could be reused. Decolourization of dyes is essential to create eco friendly environment. Adsorption follows both the Langmuir and Freundlich isotherm studies. From Scanning Electron Microscopic analysis it could be concluded that the dye molecules are entrapped by the adsorbent. Hence sunflower stalk powder, a low-cost adsorbent could be effectively utilized for decolourization of textile dye effluent.

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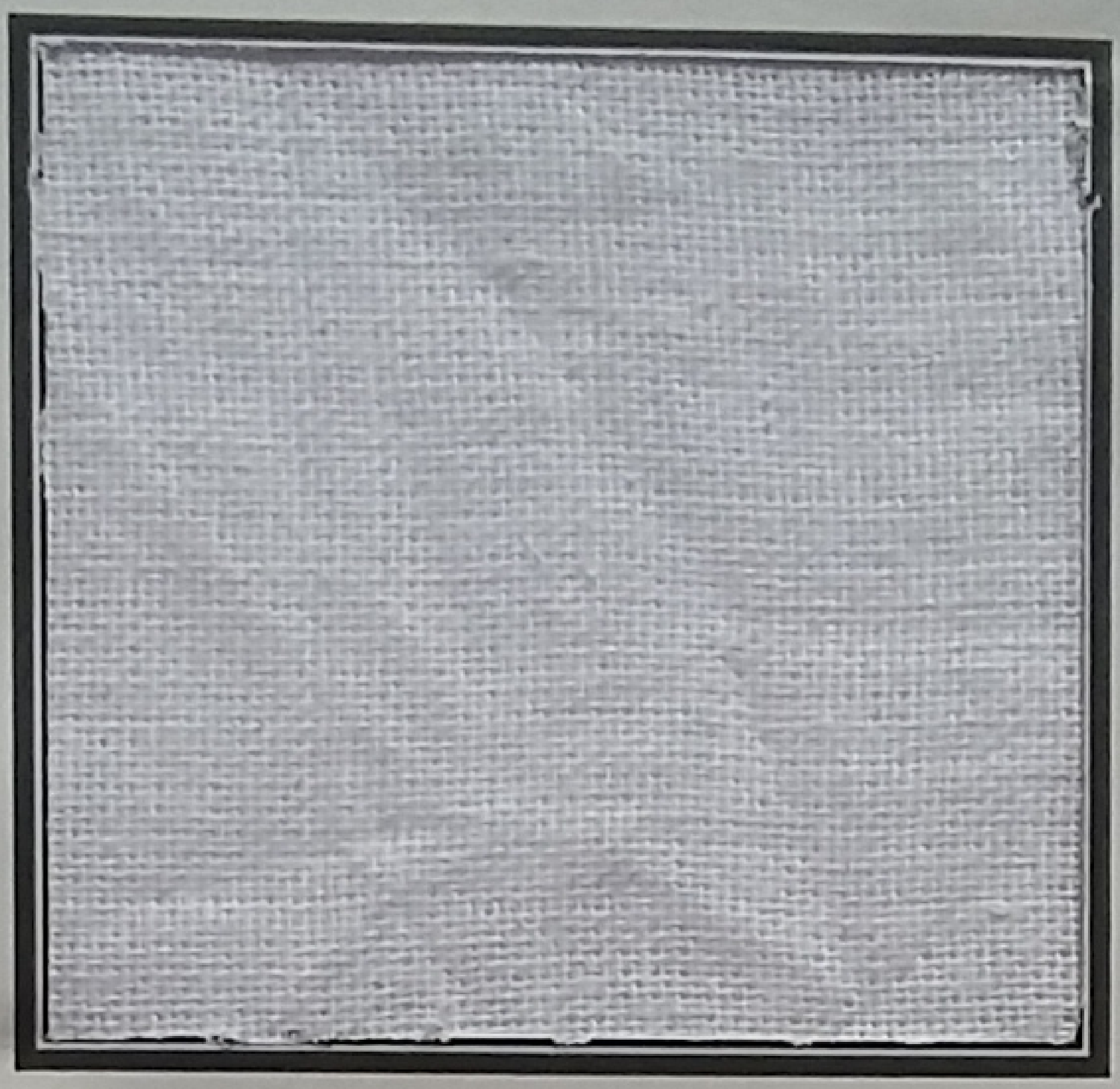
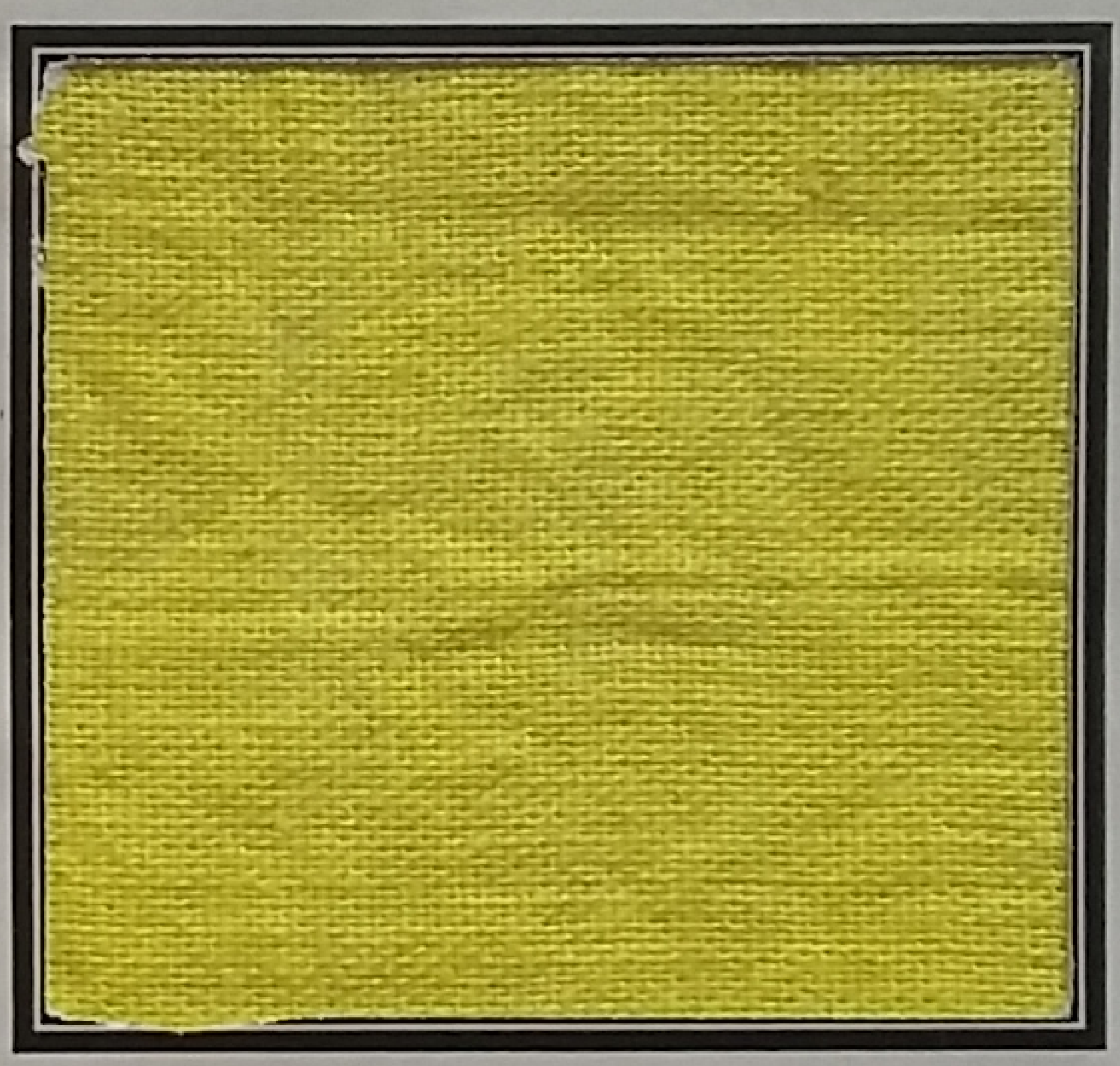
APPENDIX

ORIGINAL AND DYED FABRIC SAMPLES

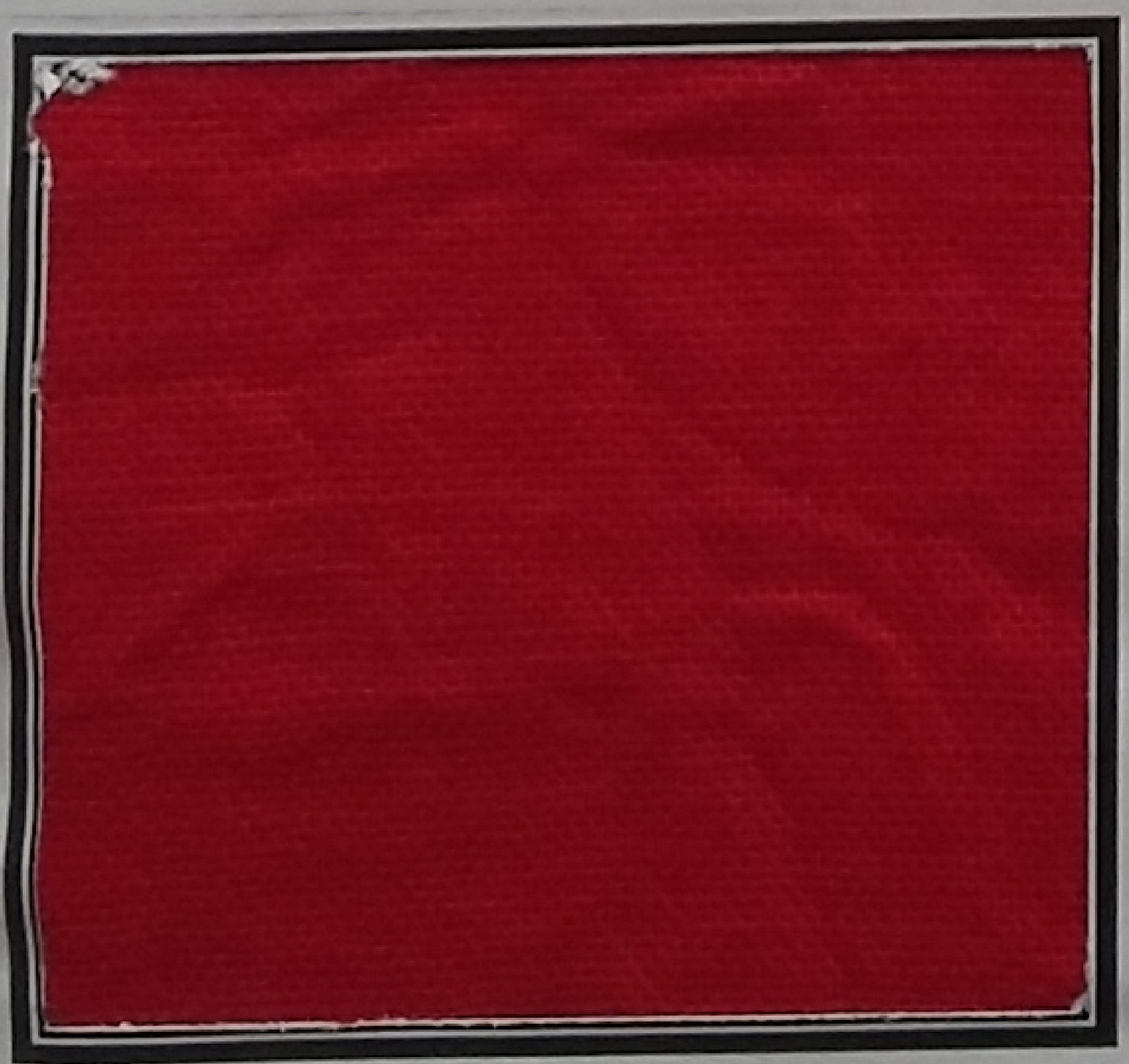


Desized Fabric

Fabric dyed with basic yellow 13 using fresh water



Fabric dyed with basic yellow 13 using decolourized water



Fabric dyed with acid red 51 using fresh water

Fabric dyed with acid red 51 using decolourized water