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Foremost Study During the Investigation of Separation of Organic Dyes from Wastewater Using Low Cost Biomaterials: A Review

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Wastewater producing from different sources like textile industry, rubber industry, paper industry, leather industry, cosmetic industry etc. contains a large amount of organic and inorganic chemicals such as dyes, dye intermediates, heavy metal ions etc. This review article is about the removal of dyes from wastewater using plant materials. Among different methods used so far, adsorption by plant materials is inexpensive and easy alternative method for the removal of dyes from wastewater. Dyes have very harmful impact on the environment especially on aquatic life which in turn has adverse effect on human beings. Therefore, it is essential to classify these dyes and find some suitable, easy and inexpensive method for their removal. Utilization of adsorption has gained great importance for the protection of environment by removing dyes from wastewater. Some adsorbents are effective but expensive, harmful and nonbiodegradable while as some are ineffective. That is why, new effective adsorbents are needed which should be least expensive, energy efficient, biodegradable and easily available. Plant material is mainly composed of lignin and cellulose and has good adsorbent property, easily available, cheap, biodegradable and nontoxic. From the survey of research papers, it was found that agricultural wastes have outstanding capabilities for adsorption. In this article we have mentioned the key study during adsorption process and reviewed the suitability and efficiency of both raw and chemically modified agricultural products in the removal of dyes from wastewater.

Graphical abstract



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1. Introduction

Among various types of water consuming industries, dying industry is one of the largest industries in which dyes are used to color various substances like textile, leather, plastics, printing, food etc. The dyes used in these industries are classified into two main categories namely natural dyes and synthetic dyes. Synthetic dyes are manmade ones whose demand have been increasing for last few decades and therefore now are replacing natural dyes. Since these dyes are not completely utilized by the substances like textiles and a significant part of these dyes get waste and come out in the form of effluent containing harmful chemicals thus is in need of proper treatment before discharging into large water body [1, 2]. Dyes present in the water bodies strongly absorb sunlight and reduce photosynthesis and oxygen of phytoplankton thus increases COD [3]. Besides dyes are also hazardous to microbial population, human beings and animals. The adequate treatment of dyes is very difficult as they are highly variable in composition [4]. During the last few decades, a various physical, chemical and biological methods were studied like electrochemical adsorption, coagulation, photooxidation, ion exchange method etc. Among them adsorption method is considered as effective method in removing the dye concentration from wastewater. This is because of its high efficiency at low concentration, wide applicability i.e. can remove a variety of impurities, fast process, producing no harmful by-products, regenerable and selective ones, ecofriendly, and simple operation. In most of the situations, combined methods are used for the treatment of wastewater to eliminate all the contaminants from wastewater [5-7].

In this article, non conventional low cost adsorbents like agricultural waste have been reviewed. The main purpose of this review is to provide information regarding agricultural wastes like leaves, fruits, barks, roots and seeds of plants. Agricultural wastes have low economic value, inexpensive and abundantly available. Plant materials mainly contains cellulose, hemicelluloses and lignin which in turn contains functional groups like hydroxyl group, carboxyl group, phenols etc. making them effective adsorbents to form bond with pollutants.

Adsorbents are also processed to improve their efficiency. There are two methods namely physical and chemical for the processing of adsorbents. Physical methods involve heating of adsorbent in an oven while as chemical method involves activation by adding acid or alkali. The goodness of adsorbents can be compared with the help of their cost. In general an adsorbent abundant in nature and having in need of little processing with high adsorption capacity is considered to be low cost and efficient adsorbent [8]. This review article presents the usage of low cost adsorbents and their importance to remove dyes from wastewater.

2. Dyes and dye intermediates

Dyestuff is a common industry expression that includes both dyes and pigments in the language of chemistry. It is estimated that there are at least 600 types of dyestuffs formed in the India. Dye particles are much finer than pigment particles. Dyes are deep colored organic compounds or mixtures which are used to impart color various materials like cloth, paper, leather, plastics etc in an everlasting fashion. The aqueous solution of dye is generally used to impart colour to

the substances as dyes are generally soluble in water. However, sometimes a mordant (fixing material) is used to develop fastness of the dye on the substrate. The fundamental significance of dye is that after applying to substrate is resistant to washing. Pigments are usually insoluble in aqueous medium but play an important role in painting [9]. The dye intermediates are prepared from coal tar constituents by using number of chemical reactions and these dye intermediates are then easily transformed into commercial dyes [10]. By using number of methods, a representative example of dye intermediate like aniline can be synthesized from benzene which in can be converted into a various type of dyes by applying different methods of preparation. Therefore, any raw material used for the formation of organic dyes is known as dye intermediate. Among large number of dye intermediates used so far most of them are members of the aromatic series and these members in turn are mostly derivatives of three organic compounds namely benzene, naphthalene, and anthraquinone [11].

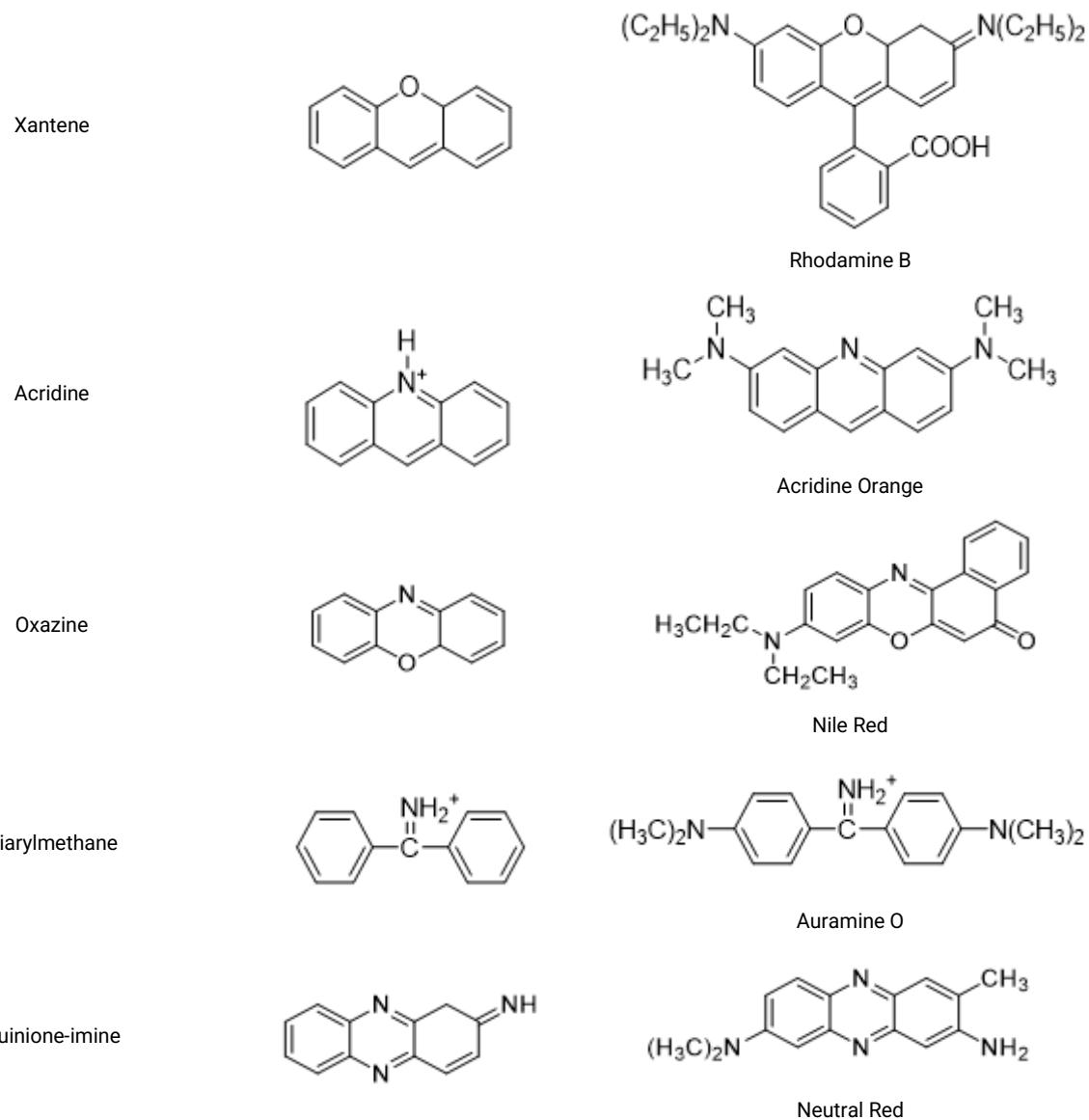
2.1. Classification and application of dyes

It is mandatory to classify dyes because of their enormous increase in kind and number. Therefore, the dyes are classified on the basis of their structure, color, source, solubility and application methods. However, the classification based on the application of dyes is often considered as more advantageous than that of the other parameters like color etc because of the complexities in nomenclature. Therefore, the main classification of dyes is based on the use or application of such dyes as adopted by the color index (C.I) and a five digit C.I number is given to every dye after knowing its chemical structure by manufacturer [12]. The dyes manufactured find their importance at various places as these dyes are widely used in textiles, paper printing, cosmetics and food. The textile industry is one of the largest users of such dyes and the number of these commercial dyes are more than 100,000 and their worldwide annual production is greater than 7×10^7 [13]. The dye obtained from natural sources like plants, minerals and animals are called as natural dyes and these natural dyes should be applied to the substances like textile with the help of mordant as these dyes are non-substantive [14]. The commonly used mordant is metallic salt as it has good affinity for both the dye and the substance such as fiber [14]. Synthetic dyes are chemically manufactured and are usually least expensive than that of natural dyes. These synthetic dyes are used in various industries to a great extent. Among large number of consumers of these synthetic dyes textile processing industries are chief users of such dyes [15]. The first man-made organic dye was prepared by Woulfe in 1871 by treating Indigo dye with nitric acid and prepared Picric acid. Since then a number of new synthetic dyes have been added to the continually growing list of dyes [16]. Textile dyes are generally classified on the basis of their chemical makeup, their use in fiber textiles and other uses related to colour [17].

On the basis of chemical structure of textile dyes, they are classified into (Nitro dyes, Indigo dyes, Azo dyes, Anthraquinone dyes, Phthalein dyes, Nitrated dyes, Triphenyl methyl dyes, etc.) or their industrial application. The classification of textile dyes on the basis of their chromophore are given in Table 1 [18, 19].

Table 1. Classification of dyes on the basis of chromophore.

Class	Chromophore	Example
Nitro dyes		 Disperse Yellow 14
Indigoid dyes		 C. I. Vat Blue 35
Azo dyes	-N=N-	 Acid Red 337
Phthalein dyes		 Phenolphthaleine
Anthraquinone dyes		 Reactive Blue 4
Triphenyl methyl dyes		 Basic Violet 2



2.1.1 Classification of dyes on the basis of chemical structure

Azoic dyes

It has been confirmed that about 1 million tons of azo dyes are formed yearly throughout the world and more than 2000 fundamentally different azo dyes are in use in the present time [20]. There may be one, two or three azo bond linkages ($-N=N-$) called monoazo, diazo or triazo dyes respectively. About 70% of dyes used in the industries are azoic one and azo dyes are one of the largest classes of man-made dyes [21, 22]. Azo dyes are used to impart colour of various substances such as textile, leather, paper, cosmetic, pharmaceutical, paint, and food industries.

Azo dye class nowadays in dyestuff chemistry represents the largest production volume and the importance of this class of dye goes on increasing with the passage of time [23]. The importance of this class of dye is ever increasing because of some important properties like their great possibility in structural change, high molar extinction coefficient, different uses and simplicity of coupling reaction. It has been reported that around 50,000 tons of textile dyes are released yearly throughout the world because of dyeing processes. Azo dyes

are predominantly used and contribute almost one-half of the total dyes synthesized [24].

It has been reported that aromatic compounds such as benzene and naphthalene are attached to both sides of azo groups and due to these aromatics same dye possesses different shades with different intensities of color [25-27]. Example of azo dyes is shown in Fig.1a.

Reactive dyes

The reactive dyes provide a broad range of shades with excellent clean and light fastness when applied to cotton and these dyes are the only textile dyes which on application to substrate form covalent bond [28]. Because of these properties the demand of this class of dyes in the market is good. It is also reported that these dyes can also be applied to cellulose, nylon and wool with increasing amount [29]. It is also confirmed that these types of dyes have poor fixation which is a long standing problem [30]. In the present time, large amount of reactive groups have been synthesized but the first commercial reactive dyes which were applied to cotton were based on the dichloro-s-triazine reactive group [31]. The example of reactive dyes is shown in Fig. 1b.

Vat dyes

It has been reported that these dyes find their use in cellulosic fibers particularly on which these dyes exhibit incomparable levels of fastness but most part of these dyes are water insoluble [32]. Vat dyes are not used so much for those substrates to which some other alternate dyes find their use. It has also been reported that the derivatives of indigo dye mainly containing halogens such as bromine give other classes of vat dyes like indigoid, thioindigoid, and anthraquinone which includes anthrimeide, indanthrone, dibenzanthrone, pyranthone, carbazole and flavanthrone [33]. Among various halogen substituents bromo substituents are major ones derived from natural indigo vat dye which in turn is obtained from number of species of indigofera plant. These vat dyes applied to those substrates which are in need of very high light and wet fastness. The typical chemical structures of vat dyes are given in Fig. 1c.

Sulphur dyes

Sulphur dyes are prepared by thionation of number of aromatic intermediates and involve different types of reactions like substitution, reduction, ring formation and oxidation. These sulphur dyes are very complicated in structure and are generally prepared from common aromatic compounds such as benzene, diphenylamine, naphthalene etc and these starting materials should possess at least one amino, nitro, nitroso, substituted amino, or hydroxy group. Sulfur dyes are used to colour silk, paper and leather but in limited quantities while as these dyes are mostly used for colouring textile containing cellulosic substances or applied to mixture of cellulosic and synthetic fibers [34]. Sulphur dyes are dullest in colour among all the man-made dyestuff classes and are enormously used because of some properties like cheap, show good washing, show good light fastness and their application mode is simple. Two commonly used sulfur dyes are shown in Fig. 1d.

Acid dyes

Acid dyes are usually sodium or ammonium salts of carboxylic, sulphuric or phenolic organic acids and are negatively charged therefore these dyes are also known as anionic dyes. These acid dyes are used to colour wool, silk, and nylon between pH range of 3.0 and 7.0 and their wet fastness varies from moderate to good. However, the light fastness of these dyes is usually in blue scale range i.e 5.0–6.0 [35]. The acid dyes have different fastness to washing and are commonly applied to substrate in acidic medium like formic acid or acetic acid. The concentration of acid that is to be used as medium depends upon the individual property of dye. The structure of dye molecules varies to a greater extent and the solubility of this group of dyes is because they contain sulphonated groups. This sulphonated group plays an essential role in bonding as it interacts with the ammonium group present on the wool fiber. In addition to this interaction, there is also presence of Van der Waals force of interaction. Acid dyes are soluble in water as these dyes use their one to four sulphonate groups for solubility and also contain azo chromophoric systems, triphenylmethane or anthraquinone. One of the examples of this dye is shown in Fig. 1e.

Disperse dyes

The disperse dyes are commonly insoluble or sparingly soluble in water and are applied to the water hating substances i.e hydrophobic fibers from an aqueous

dispersion [36]. These dyes are also used in huge quantities due to which large quantity of water is used during their application therefore releasing a great quantity of wastewater.

These disperse dyes are mainly applied to colour polyester fibers. In spite of using these dyes used on polyesters can also be used to colour other materials like nylon, cellulose acetate and acrylic fibers. However, the wet fastness property of this class of dye on these substances is poor. An example of dye from this class is shown in Fig. 1f.

Cationic dyes

Cationic dyes contain andazine, monoazoic and diazoic compounds and are also known as basic dyes because these dyes form coloured cationic salt on dissolution in water [37]. These dyes are used to colour textiles as they produce bright shades with great colouring value and are used for colouring of various materials such as silk, cotton, nylon, wool, and polyester. The chromophore of these salts contains positive charge which interacts with the negative site present on the substrate at the time of application of dye. These dyes are soluble in water and their solubility increases in the presence of glacial acetic acid. Basic dyes have also harmful effects as their exposure dermatitis, allergy, mutations, increased heart rate, shock, vomiting, skin irritation, jaundice, quadriplegia and may cause also cancer [38, 39]. Example of the basic dye is given in Fig. 1g.

Direct dyes

The direct dyes are also water soluble and can be used without any mordant. These dyes are classified on the basis of various parameters like chromophore, fastness and their application [36]. The main chromophoric groups they contain are azo, dioxazine, stilbene, and phthalocyanine. The other minor chemical classes may also be present like quinolone, formazan, anthraquinone, and thiazole. The application of direct dyes are easy and have also wide range of shade but their wash fastness is moderate and are therefore replaced somewhat by reactive dyes having high wet and washing fastness properties when applied to cellulosic substances. Example of the direct dye is given in Fig. 1h.

2.1.2 Adverse effects of dyes on humans, animals and plants

Most of the dyestuffs used in textile industries are not so dangerous for the workers working at that amount of exposure of dyes that are generally faced by the workers in the factories. However, dyes are very dangerous for a person if he gets exposed to greater amount by any means like accidental exposure, therefore workers should treat them with care. The respiratory problem is the most common health problem of workers handling reactive dyes because of inhalation of the particles of such dyes. Not only respiratory problems these dyes can cause other harmful effects like ones immune system. The symptoms of respiratory problems include itching, sneezing, watery eyes, wheezing and coughing [40]. The most leading health problem for the workers working in the industries related to dyeing and finishing is their irritation to chemicals acting as irritants like ammonia, formaldehyde-based resins, acetic acid, soda ash, some shrink-resist chemicals, caustic soda, some optical whiteners, and bleach. These irritants cause skin irritation, sneezing, itchy or blocked noses, and sore eyes. The skin sensitive dyes include certain vat, reactive and disperse dyes [40]. A significant amount of dye gets discharged into the wastewater as dye molecules are not fully fixed to the fabric thus wastewater contains both

organic and inorganic compounds [41, 42].

Examples of some dyes of the mentioned classification

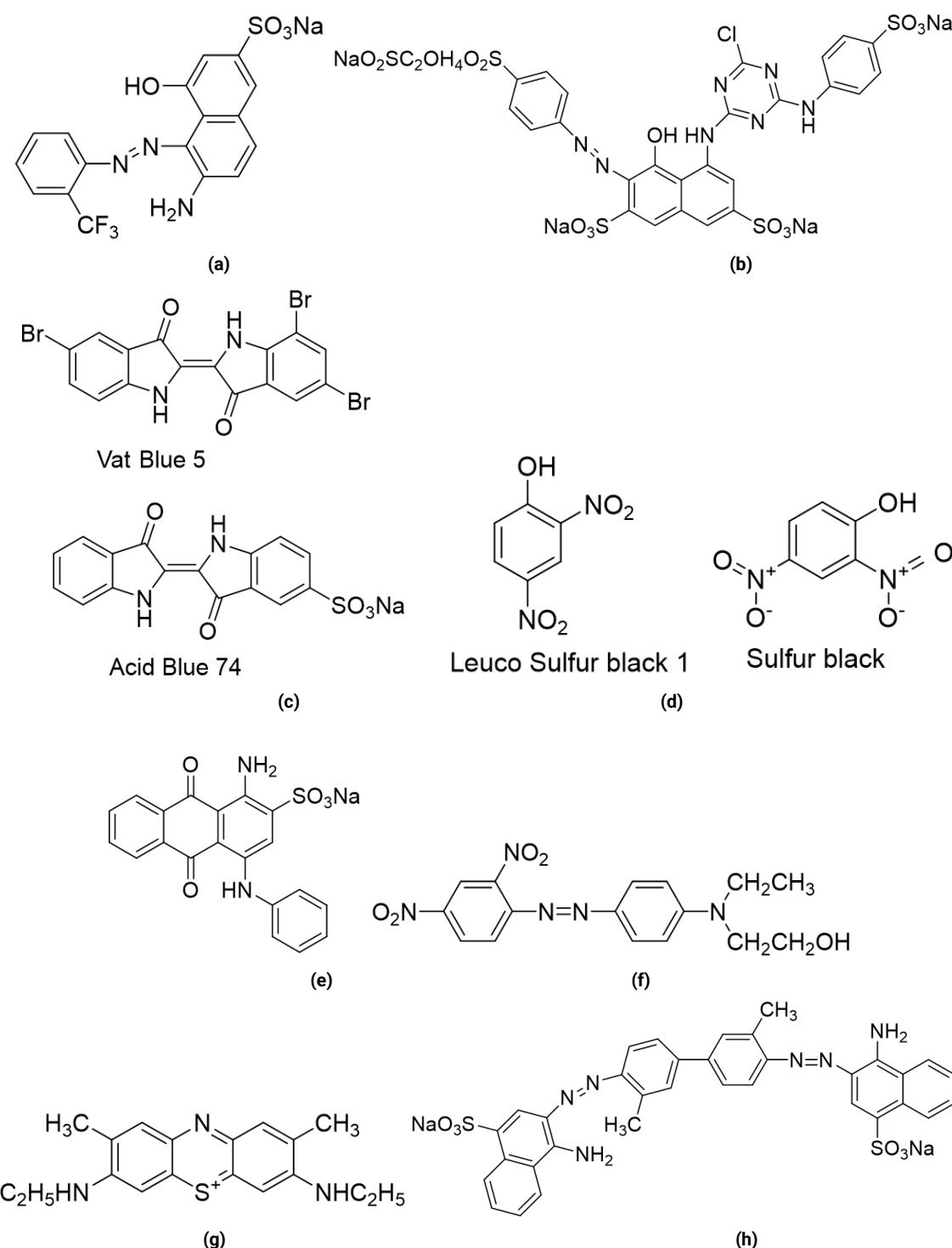


Fig. 1. Examples of some dyes of the mentioned classification. (a) Example of azo dye. (b) Dye (C.I. Reactive Red 198). (c) Chemical structures of vat dyes. (d) Two sulfur dyes very used. (e) C. I. Acid Blue 25. (f) C. I. Disperse Red 8. (g) Basic blue 22. (h) Direct Red 2.

The amount of water used in dyeing varies from fabric to fabric and it is estimated that about 1,000 to 3,000 m³ of waste water is released from textile industries after processing about 12 – 20 tons of textiles in a single day [43]. This waste water is rich in non-biodegradable and carcinogenic chemicals which cause a major risk to the human health and also the environment. The effluents also contain additional chemicals like trace metals (Cu, Cr, Zn, As etc) used in the

various processing stages and are very dangerous to the health causing nausea, dermatitis, hemorrhage, ulceration and irritation of skin [44]. Textile wastewater in addition to organic and inorganic substances also contains impurities of microbes. The azo dyes which are mainly used to colour cotton textiles in the industries are capable of changing the nature (physical and chemical) of soil, continuation of water bodies and also damage the plant and animal life in the

environment [45–52]. In addition to this azo dyes present in the water although in small amount (<1ppm) are highly visible and thus reduces transparency, visual property, and water-gas solubility. The photosynthetic activity of aquatic plants which get affected by dyes also reduces which in turn reduces the oxygen concentration of that region therefore harms the biological cycles of aquatic plants and animals [53]. Depending upon the exposure of time and azo dye concentration there are large number of azo dyes which are highly poisonous in nature and cause damage to the ecosystem. The compound 1,4-diamino benzene whose parent azo dyes can cause various health hazards like permanent blindness, skin irritation, chemosis, contact dermatitis, lacrimation, respiratory ailments, hypertension, vomiting gastritis, vertigo, swelling of the neck, face, tongue, pharynx, and larynx [54]. Aromatic amines can be absorbed by exposed areas like skin, mouth but their absorption is faster through ingestion [55].

3. Adsorption

The adsorption term was first given by Heinrich Keyser a German Physicist in 1881 [56]. Adsorption is a surface phenomenon in which adsorbate (atoms, ions or molecule in any form like gaseous, liquid or dissolved solid form) gets adsorbed on to the surface of the adsorbent. So, there is adhesion of adsorbate on to the adsorbents surface. The solid material to which adsorbate particles gets attached is known as adsorbent and the substances like dye molecules which get attached to the surface of the adsorbent is known as adsorbate. Adsorption process creates the film of the adsorbate on the surface of the adsorbent. Adsorption process is similar to that of surface tension phenomenon as the surface atoms of an adsorbent are not surrounded equally on all the sides, therefore attracts foreign particles. However, interior atoms of an adsorbent are equally attracted on all the sides thus fulfill their bonding requirements by their own constituent atoms. The adsorbate particles adsorbed from a particular type of solution are resistant to washing in the same solution for example dye adsorbed from aqueous solution cannot be washed from the adsorbent by the water but there is requirement of some other solvent for washing that particular dye. In general adsorption process is classified into two types namely Physisorption (weak van der Waals forces) and Chemisorption (characteristic of covalent bonding). However electrostatic interaction have been also reported [57]. Adsorption find its use in a wide range especially in industries such as heterogeneous catalysts and this adsorption phenomenon is present in natural, biological, physical and chemical systems [58, 59]. Activated charcoal by adsorption process plays a vital role for the purification of water. This adsorption process is described through various isotherms and among them some namely Langmuir, Freundlich, Temkin, and D-R isotherms are explained in this review article. The factors affecting adsorption are surface area of an adsorbent, particle size, temperature, pH, contact time, adsorbent dosage and their interactions. The reverse of adsorption process is known as desorption.

a. Factors affecting adsorption

There are a number of factors those affect the process of adsorption such as the effect of initial concentration, effect of adsorbent dosage, effect of pH, effect of contact time, effect of temperature and effect of agitation speed.

3.1 Initial Concentration

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The adsorption capability goes on increasing with increase in initial adsorbate concentration until reaches maximum value known as optimum value. After optimal value there is no further change in the case where adsorption and desorption rate are same. This is because at optimal value all the active sites get occupied and there is no further site for adsorption [60, 61].

3.2 Effect of adsorbent dosage

The amount of adsorbent that should be used at one time during a period of time is known as adsorbent dosage. Dosage is a considerable factor in the process of adsorption. Adsorption increases with the increase in adsorbent dosage but also depends upon the volume of solution to which adsorbent is added. High amount of dose of an adsorbent in little volume has no much role in percentage removal but has negative impact on the percentage removal of pollutant from the solution. The negative effect is because of aggregation of adsorbent particles in small volume due to which active sites of an adsorbent gets hidden. However, adsorption capacity depends upon the saturation limit of the adsorbent when aggregation of particles does not take place [60]. The study of effect of dose on the adsorption process gives an idea about the optimum and economical amount of dose of an adsorbent.

3.3 Effect of pH of the solution

The most important factor of the solution is its pH. It is the measure of acidity or basicity and controls the extent of electrostatic charges imparted by charged dye molecules in a solution [62]. Thus, when pH changes adsorption capacity also changes. By the addition of 0.1 N NaOH or 0.1 N HCl to the dye solution its pH can be adjusted. In basic pH the adsorption of cationic dyes is better than that of anionic dyes and vice versa [63]. Adsorption capacity changes with change of pH because H⁺ ion concentration changes which competes the cation groups on the dye for adsorption. As pH increases the competition between cation site of dye and H⁺ ion decreases, thus increases adsorption rate.

3.4 Effect of contact time

Contact time is another factor which should be considered mainly for adsorption. In general, the percentage removal of dye from its solution increases with increase in time but up to a certain extent. After this point, the adsorption is not affected at all as adsorption sites of adsorbent get occupied with adsorbate particles [64]. At this point there is equilibrium between adsorption and desorption. The time period at which equilibrium is attained is known as equilibrium time and the capacity of pollutant removal at equilibrium time is known as maximum adsorption capacity of that adsorbent under given sets of conditions. Contact time varies depending upon the type of adsorbent. It is because of the pores or the sites that are present in the adsorbent.

3.5 Effect of temperature

Temperature is also one of the important parameters that helps us to know whether the adsorption process is endothermic or exothermic. In endothermic process the adsorption capacity increases with increase in temperature while as adsorption capacity decreases with increase in temperature for exothermic process [65, 66].

3.6 Effect of agitation speed

Experimental results may vary by varying the agitation speed and thus different results can be obtained from the same adsorbate-adsorbent system in batch mode adsorption process [67, 68]. This effect can be measured by varying only this parameter and keeping all other parameters constant. It is observed that adsorption capacity increases with increase in agitation speed because of lowering the boundary layer resistance between adsorbate molecules in the bulk of solution and adsorbent's surface [63].

b. Mechanism of adsorption

Adsorption is a surface phenomenon where adsorbate is adsorbed by the adsorbent on to its surface. The adsorbate particles get adsorbed on to the adsorbents surface through bonding interactions and by diffusion of these adsorbate molecules like dyes into the interior pores from the surface of the adsorbent forming monolayer or multilayer of adsorbate molecules. An adsorption phenomenon depends up on various parameters such as dose of an adsorbent, pH, concentration of an adsorbate, contact time, temperature, size of an adsorbent and its surface morphology. It is assumed that the mechanism of adsorption process involves four steps [69]. These four steps are:

1. Movement of dye molecules from the bulk of its solution to the adsorbents surface.
2. Diffusion of dye molecules by crossing the boundary layer to the adsorbents surface.
3. Interaction between adsorbate molecules and active sites of adsorbent surface.
4. Intraparticle diffusion of dye molecules from surface to the interior pores of the adsorbent particle.

The increase in contact time reduces the resistance of adsorption by increasing the chances of adsorbate molecules to come in contact with the adsorbents surface. The adsorption speed is generally governed by two factors namely liquid phase mass transfer rate and rate of intraparticle mass transfer [70]. Intra-particle diffusion plot is one of the most commonly used kinetic models for identifying the mechanism of adsorption.

There are different types of adsorption forces involved in the adsorption processes like chemisorptions, ion exchange, hydrogen bonding, hydrophobic bonding and Van der waals forces. In many systems the net interaction of an adsorbate molecule with a surface may comprise more than one of the above types of interaction, especially if the molecule is polyatomic and contains functional groups of different types.

The adsorption mechanism also depends upon the following factors [71].

(a) Micellization: It means that above the critical micelle concentration of the solute (adsorbate) the activity increases only lightly with increase of concentration.

(b) Surface Potential: Greater the surface potential of adsorbent particles greater will be tendency for adsorbate molecules and vice versa.

(c) Heterogeneity: There are two types of surfaces namely homogenous and heterogeneous. The heterogeneous surfaces of adsorbents have two types of sites i.e low energy sites and high energy sites. Thus, adsorption process also depends upon the type of surface.

(d) Lateral Interaction: Lateral interaction between adsorbed molecules may be attractive or repulsive in character. For example, non-polar groups tend to lie flat on

non-polar surfaces at low coverage, but are forced to orient away from the surface at high coverage and their chemical potential is thereby increased or decreased depends on the molecular interaction. Where lateral interaction is strongly favourable the isotherm may even rise so markedly as to become a convex.

4. Adsorption Isotherms

Adsorption isotherm gives us relationship between the amount of adsorbate adsorbed on the adsorbent's surface and the equilibrium concentration of adsorbate in the solution at constant temperature. It is characteristic property and depends on adsorbent and adsorbate. Several factors govern the nature of isotherm so far. Therefore, it is difficult always to assign a single isotherm for a particular case. It may obey different isotherms. It is necessary then to find out best fit model so that nature of bonds between adsorbent and adsorbate can be ascertained. The adsorption isotherm describes the interaction between the adsorbate and the adsorbent thus has theoretical and a practical significance. As different kinds of forces play their role such as van der Waals forces, cohesive forces, electrostatic forces, buoyancy factor, chemical bonds, hydrogen bonds etc. From the parameters of different isotherm models various important information is obtained regarding the adsorption process like surface properties, adsorption mechanisms, and affinities of the adsorbent [72]. Among various isotherm models here only four important namely Langmuir, Freundlich, Temkin and Dubinin-Radushkevich (D-R) isotherm models were explained.

4.1 Langmuir Model

According to this model, adsorption occurs at particular homogeneous sites on the surface of the sorbent. This isotherm model considers same active sites on the adsorbents surface in terms of energy and adsorption process involves formation of monolayer coverage of adsorbate on the surface of adsorbent. According to this isotherm model there is no intermolecular interaction between neighboring molecules adsorbed. Thereby, Langmuir isotherm model is the equilibrium distribution of adsorbate molecules between the solid and liquid phases [73-75]. Based upon these assumptions, Langmuir represented the following equation:

$$q_e = \frac{q_{\max} b C_e}{1 + b C_e}$$

The linear form of Langmuir isotherm assuming monolayer adsorption is expressed by Eq. (1) [76].

$$\frac{C_e}{q_e} = \frac{1}{b q_{\max}} + \frac{C_e}{q_{\max}} \quad (1)$$

where C_e is equilibrium concentration of adsorbate, q_{\max} (mg/g) is the highest adsorption capability of the adsorbent due to the formation of monolayer of adsorbate. The b parameter is a coefficient associated with the adsorption energy. From the linear regression plot of $(1/q_e)$ versus $(1/C_e)$ the values of q_{\max} and b can be calculated from the values of slope and intercept. The important and fundamental feature of the Langmuir isotherm can be expressed in terms of a dimensionless constant called separation factor (R_L), and is given by [77]:

$$R_L = \frac{1}{1 + b C_0}$$

where C_0 (mg/l) and b (l/mg) represent the initial dye concentration and the Langmuir constant. For any adsorption

there are four possible R_L values:

$R_L > 1$ (unfavourable), $R_L = 1$ (linear), $R_L = 0$ (irreversible) and $0 < R_L < 1$ (favourable) [78].

4.2 Freundlich Isotherm

The Freundlich adsorption isotherm is generally applied for heterogeneous surfaces where adsorbate molecules form multilayer on the adsorbent [75, 79]. According to this model the active sites of an adsorbent having greater affinity for adsorbate molecules get occupied first and this affinity of binding sites goes on decreasing with the passage of time [77, 80, 81]. So initially there is greater affinity of adsorbent for adsorbate molecules and the non-linear form of Freundlich isotherm is given by the following equation [82]:

$$q_e = (K_F(C_e)^{1/n})$$

where K_F is the measure of sorption capacity or Freundlich isotherm constant (mg g^{-1}), $1/n$ is the sorption intensity, also known as the Freundlich coefficient, C_e is the equilibrium concentration of adsorbate (mg/L) and q_e is the amount of adsorbate adsorbed per gram of the adsorbent at equilibrium (mg/g). Linearizing above equation, the Eq. 2 is obtained.

$$\ln q_e = \ln K_F + 1/n \ln C_e \quad (2)$$

K_F and n are constants and depend up on the temperature [83]. Thus, the plot that is obtained from $\ln q_e$ vs. $\ln C_e$ should be a straight line with intercept $\ln K_F$ and slope $1/n$. The values obtained for intercept and slope are used to calculate the values of K_F and n respectively. If the values of $n > 1$ the adsorption conditions are considered favourable [84-86]. However, if the value of $n = 1$ then the partition between the two phases are independent of the concentration. The magnitude of the exponent $1/n$ tells the favorability and strength of adsorption in the adsorption process [87]. The value of $1/n$ more than 1 is the representation of cooperative adsorption while as the value of $1/n$ less than 1 is considered as normal adsorption [88]. The bigness of n value gives a measure of the appropriateness of adsorption and if its value is in between 1 and 10 the adsorption is considered as favourable [89, 90]. However, the constants n and K_F are characteristic of the adsorbate-adsorbent system and must be determined by linear regression of data fitting [91].

4.3 Temkin Isotherm

Temkin isotherm is one of the early models that describe the adsorption of hydrogen onto platinum electrodes in acidic solutions [92]. The isotherm contains a factor that explains the adsorbent-adsorbate interactions. It ignores the extremely low and high values of concentrations and assumes that the heat of adsorption (ΔH_{ads}) of all molecules in the layer would decrease linearly rather than logarithmically with coverage [93]. The non-linear form of Temkin equation is given

below [94]:

$$q_e = \frac{RT}{b} \ln K_T C_e$$

The linear form of the Temkin equation is represented as:

$$q_e = \left(\frac{RT}{b}\right) \times \ln K_T + \left(\frac{RT}{b}\right) \times \ln C_e \quad (3)$$

where $(RT/b) = B$, R is the universal gas constant, T is absolute temperature and b is another constant. K_T (g L^{-1}) and B (J mol^{-1}) are Temkin constants related to the equilibrium binding constant corresponding to the maximum binding energy (adsorption potential) and heat of adsorption respectively.

Temkin isotherm model is excellent for predicting the gas phase equilibrium and discusses the interaction between adsorbent and adsorbate. This model assumed that a uniform distribution of binding energy occurs [95]. From Eq. 3, slope RT/b and intercept $(RT \ln K_T)/b$ is obtained by plotting a graph between q_e versus $\ln C_e$.

4.4 Dubinin–Radushkevich Model

The Dubinin–Radushkevich isotherm takes into account Gaussian energy distribution onto a heterogeneous surface for the expression of adsorption mechanism [96, 97] and this model is often used for high and intermediate range of concentrations. With the help of this model the two main types of adsorption (physical and chemical) can be distinguished [98]. This isotherm is temperature dependent and is plotted between logarithm of amount adsorbed versus the square of potential energy [96]. This reason behind this model was to estimate the apparent free energy of adsorption and characteristic porosity [95]. The non-linear form of this equation is given by:

$$q_e = (q_m) \exp(-\beta \varepsilon^2)$$

The linear forms of D-R isotherm represented as:

$$\ln q_e = \ln q_m - \beta \varepsilon^2 \quad (4)$$

$$\varepsilon = RT \ln \left(1 + \frac{1}{C_e}\right) \quad E = 1/\sqrt{2\beta}$$

where, ε , q_m , C_e represent Polyani potential, monolayer capacity (mol g^{-1}), and equilibrium concentration (mol L^{-1}), respectively. Using above equations Polyani potential (ε) and mean free energy of adsorption ' E ' (kJ mol^{-1}) can be calculated. By plotting $\ln q_e$ vs ε^2 , the D-R model constant ($\text{mol}^2 \text{kJ}^{-1}$) or the porosity factor (β) can be obtained from the slope.

Whereas q_m in D-R model represent the maximum adsorption by adsorbent's total specific micropore volume.

It is generally expected that if the value of E is less than 8 kJ/mol the adsorption process is considered as of physical nature, between 8 and 16 kJ/mol the adsorption type is considered as ion exchange, and above 16 kJ/mol the adsorption process followed by chemical adsorption [99, 100].

Table 2. Linear and nonlinear forms of adsorption isotherm models

Isotherm	Nonlinear form	Linear form	Linear plot	Eq.	Ref.
Langmuir	$q_e = \frac{q_{\max} b C_e}{1 + b C_e}$	$\frac{C_e}{q_e} = \frac{1}{b q_{\max}} + \frac{C_e}{q_{\max}}$	$\frac{C_e}{q_e}$ vs C_e	1	[76]
Freundlich	$q_e = (K_F)(C_e)^{\frac{1}{n}}$	$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e$	$\ln q_e$ vs $\ln C_e$	2	[82]
Temkin	$q_e = \frac{RT}{b} \ln K_T C_e$	$q_e = \left(\frac{RT}{b}\right) \ln K_T + \left(\frac{RT}{b}\right) \ln C_e$	q_e vs $\ln C_e$	3	[94]
D-R	$q_e = (q_m) \exp(-\beta \varepsilon^2)$	$\ln q_e = \ln q_m - \beta \varepsilon^2$	$\ln q_e$ vs ε^2	4	[97]

Table 3. List of kinetic models and their linearized expression.

Kinetics	Nonlinear form	Linear form	Linear plot	Eq	Ref
Pseudo-first order	$q_t = q_e(1 - e^{-kt})$	$\log(q_e - q_t) = \log q_e - k_1 t / 2.303$	$\log(q_e - q_t) vst$	5	[104-106]
Pseudo-second order	$q_t = \frac{q_e^2 \times k_2 \times t}{1 + q_e \times k_2 \times t}$	$\frac{t}{q_t} = \frac{1}{k_2 \times q_e^2} + \frac{t}{q_e}$	$\frac{t}{q_t} vst$	6	[109-111]
Elovich	$q = \frac{1}{\beta} \ln(\alpha \beta t)$	$q_t = \frac{1}{\beta} \ln(\alpha \beta) + \frac{1}{\beta} \ln t$	$q_t vslnt$	7	[116]
Intra-particles diffusion	$q_t = K_{id} \sqrt{t}$	$q_t = K_{id} t^{0.5} + I$	$q_t vs \sqrt{t}$	8	[118,119]

5. Sorption kinetics

The sorption kinetics deals with the study of determination of uniqueness of sorption with respect to time. The kinetic model for adsorption is very important to understand the mechanism. Though there are various models suggested, but pseudo first order model is widely used. It is considered that either active surface sites or species present in solution govern the adsorption process. Initially it is observed that rate of adsorption is more and then it becomes exponential. This is obvious, because initially all active sites are available for adsorption and as time passes the number of active sites decreases, which reduces the rate of adsorption. Finally, all active sites are occupied by adsorbate and hence adsorption becomes steady. The percentage removal of CV dye with respect to time by different parts of banana plant showed that the percentage removal increases sharply during the beginning part of sorption (in part I) of the graph [101]. That part showed that initially there is sufficient availability of adsorption sites and also adsorbates. With the passage of time as shown in Part II of that graph both adsorbent sites and adsorbate concentration decreases and thus slows down adsorption process. The last part of that graph indicates equilibrium of sorption where sorption sites become saturated and there is no further sorption. So, suitable kinetic models are employed to examine the adsorption mechanism process such as mass transfer and chemical reaction.

5.1 Lagergren Pseudo – first order kinetic model

This model has been proposed by Lagergren and described as the below formula [102]:

$$q_t = q_e(1 - e^{-kt})$$

According to Lagergren the adsorption speed depends upon the adsorption capacity of adsorbent to adsorb the adsorbate from aqueous solutions thus is suitable model solid/liquid systems [103, 104]. Equation 5 gives the integrated linear form of this model [105-107].

$$\log(q_e - q_t) = \log q_e - k_1 t / 2.303 \quad (5)$$

where q_e (mg/g) is equilibrium amounts of adsorbate adsorbed, q_t (mg/g) is the amount of adsorbate adsorbed at time t and k_1 (min^{-1}) is the pseudo-first order equilibrium rate constant. The kinetics parameters are obtained from the plot $\log(q_e - q_t)$ against t .

5.2 Pseudo-second-order kinetic model

This model is based on the adsorption capacity of adsorbents and was proposed by Ho [108, 109]. It is based on the adsorbent concentration unlike other models based on adsorbate concentration. The Ho's pseudo-second-order model was expressed as [110-112]:

$$q_t = \frac{q_e^2 \times k_2 \times t}{1 + q_e \times k_2 \times t}$$

where k_2 ($\text{g mg}^{-1}\text{min}^{-1}$) is rate constant related to pseudo-second-order while as q_t and q_e have their usual meanings as in pseudo-first-order kinetic model. The above equation could be linearized as:

$$\frac{t}{q_t} = \frac{1}{k_2 \times q_e^2} + \frac{1}{q_e} \times t \quad (6)$$

The adsorption data is plotted as t/q_t versus t . The initial adsorption rate v_0 at $t = 0$ could be calculated from Eq. (6.1) [110, 113, 114].

$$v_0 = k_2 \times q_e^2 \quad (6.1)$$

5.5 Elovich kinetic model

This model is applicable to adsorbents having various chemical groups present on the surface and is applicable for chemisorptions [115]. However, this model was formerly used for the gaseous adsorption on the surface of solids without desorption and the adsorption rate decreases by surface coverage with time. Since, in chemisorptions adsorption mechanism is controlled by a chemical. The nonlinear Elovich kinetic model is represented by [116]:

$$q = \frac{1}{\beta} \ln(\alpha \beta t)$$

where the initial adsorption rate (mg/g min) represented by α and desorption constant (g/mg) represented by β are associated with the extent to which surface gets occupied and activation energy in case of chemisorptions.

The linear form is represented by equation 7 given by Wu et al. [117].

$$q_t = \frac{1}{\beta} \ln(\alpha \beta) + \frac{1}{\beta} \ln t \quad (7)$$

From the plot of q_t vs $\ln t$, the Elovich kinetic model parameters can be determined.

5.4 Intra-particles diffusion model

This is an important model which is used for the determination of adsorption mechanism [118, 119]. There are number of adsorption processes whose amount of adsorbate adsorbed by the adsorbent varies almost proportionately with \sqrt{t} rather than with the contact time [120] and is given by Weber and Morris, (1963) as:

$$q_t = K_{id} t^{0.5} + I \quad (8)$$

where q_t , K_{id} and $t^{0.5}$ determine amount of adsorbate adsorbed at time t , rate constant ($\text{mg/g min}^{0.5}$) and square root of time respectively. By plotting a graph between q_t vs $t^{0.5}$, the value of slope gives the value of K_{id} and the value of intercept gives intercept (I). I (mg/g) is a constant giving an idea about the boundary layer thickness. The value of I decides whether the adsorption process is controlled only by intraparticle diffusion ($I = 0$) or some other mechanisms are involved when I is not

zero [121]. In a batch reactor system intra-particle diffusion is generally rate limiting step and film diffusion is in continuous

6. Thermodynamic study

When the adsorption process is temperature dependent, it is useful and important to study the thermodynamic parameters like free energy change (ΔG°), enthalpy change (ΔH°), and entropy change (ΔS°) [123-127]. The below equation (9) is used for the calculation of Gibbs free energy change.

$$\Delta G = -RT\ln K_c \quad (9)$$

Using Van't Hoff equation as given below (equation 10), ΔH° and ΔS° of sorption process can be calculated.

$$\ln K_c = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (10)$$

were K_c , R, and T represent equilibrium constant, gas constant, and absolute temperature (K). From linear plot of $\ln K_c$ vs. $1/T$, the value of ΔH° and ΔS° can be calculated from slope and intercept respectively. To determine the value of K_c following relation is used.

$$K_c = \frac{q_e}{C_e}$$

where q_e (mg g⁻¹) is equilibrium amount of adsorbate adsorbed and C_e (mg L⁻¹) is the concentration of adsorbate at equilibrium in a solution.

In adsorption process the positive value of enthalpy change (H°) indicates endothermic adsorption as shown by malachite green dye (MG) onto DCB [128]. The endothermic adsorption was also reported by other adsorbents for the same MG dye like bentonite, activated carbon prepared from Tuncbilek lignite, and activated carbon prepared from de-oiled soya [129, 130]. The negative value of ΔG° indicates spontaneous adsorption as was also observed in the mentioned adsorbate-adsorbent system i.e MG onto DCB and ΔG° values decreased by increasing temperature 25 °C to 45 °C suggesting increase in adsorption capacity of adsorbent for MG with increasing temperature [131]. Also the entropy change value (ΔS°) is positive showing attraction of DCB towards MG [130, 132, 133].

7. Applications for dye removal

7.1 Plant leaves

Plant leaves are mainly composed of cellulose, hemicellulose, lignin, and pectins which in turn contains different functional groups such as hydroxyl, carboxyl, amino, carbonyl and nitro groups [134, 135]. These functional groups interact with the oppositely charged adsorbate molecules like dyes leading to the binding of such molecules onto leaves biomass [136, 137]. The plant leaves also contain saponins and alkaloids, when treated with some chemicals like formaldehyde, may get converted into resins and hence its adsorption capacity increases. Sometimes treatment with acids leads to formation of degraded product and surface area increases which ultimately lead to the increase in adsorption capacity. It is well known fact that carbon is charcoal and is the best adsorbent known so far. Therefore, plant materials are burnt to get charcoal, in order to get good adsorption. The charcoal of different plant materials exhibits different adsorption capacities. In most of the cases dry powder of plant materials are used as adsorbents. Different leaves based biomass have been utilized for the removal of different classes of dyes from which very promising results

flow system [122].

have been obtained. Some adsorbents like *Posidonia oceanica* (L) [138-140], Neem tree [141], Hyacinth leaves [142, 143], *Platanus orientalis* (L) [144], Coconut leaves [145], *Nerium indicum* (NI) and *Duranta erecta* (DE) leaves [146], *Platanus orientalis* (Chinar tree) [147], *Populus* leaves [46], *Juglans regia* leaves [148], *Salix alba* L (willow tree) [149], and *Morus nigra* L (mulberry tree) [150, 151] have been utilized for the adsorption of different dyes. Their applications with references are given below.

Table 4. Low-cost adsorbents (leaves) used for dye removal.

Adsorbents (leaves)	Applications (removal of dyes from wastewater)	references
<i>Posidonia oceanica</i> (L)	MB MV Astrazon Red	[138] [139] [140]
Neem tree	BG	[141]
Hyacinth leaves	Amaranth dye (AR27) BF-4B	[142] [143]
<i>Platanus orientalis</i> (L)	MB	[144]
Coconut leaves	MB	[145]
<i>Nerium indicum</i> (NI) and <i>Duranta erecta</i> (DE) leaves.	Crystal Violet	[146]
<i>Platanus orientalis</i> (Chinar tree)	Crystal Violet	[147]
Populus leaves	Methyl Orange	[46]
<i>Juglans regia</i> leaves	Crystal Violet	[148]
<i>Salix alba</i> L (willow tree)	Malachite green	[149]
<i>Morus nigra</i> L (mulberry tree)	Methylene blue, Crystal Violet	[150, 151]

7.2 Fruits

Adsorption of effluent dyes with the use of biological farm waste or skin/peels of fruits and vegetables is an excellent source for decolorization of the dyes from textile industries. However, it is dependent on the concentration of the dyes used. Some fruit peels namely Watermelon peels, sweet lime peels, Papaya peels [152], Orange peels [153, 154] and Banana peels [155, 156] and their application of adsorption of dyes are given below:

Table 5. Low-cost adsorbents (fruits) used for dye removal.

Adsorbents (Fruit peels)	Applications (removal of dyes from wastewater)	References
Watermelon (<i>Citrullus lanatus</i>)	MG, MB, MO, CV, CR	[152]
Sweet lime (<i>Citrus limetta</i>)	MG, MB, MO, CV, CR	[152]
Papaya (<i>Carica papaya</i>)	MG, MB, MO, CV, CR	[152]
Orange peels	Ramazol brilliant blue DR23, DR80 (DR=Direct red)	[153] [154]
Banana peels	MG, MB CR (Congo red)	[155] [156]

7.3 Barks

Bark is usually considered as a waste part of timber processing and its disposal is one of the major concerns. Barks are available in the forest industry and after in forestry operations as it includes 9 to 15 % of stem volume therefore are among the most abundant bio-resources in the world. Huge amounts of barks can be found around wood processing units like forest-processing industry, pulp mills and primary wood-processing mills. Since it is a solid waste and its disposal vary from country to country depending cost effectiveness and market availability. However recently, the adsorption process using barks as adsorbents has been gaining support for the removal of pollutants especially dyes from aqueous solutions. Some tree barks like Mangrove bark and Teak tree bark [157,158], Neem Tree bark and Mango tree bark [159], *Eucalyptus angophoroides* bark [160], *Coniferous pinus* bark [161] and Pineapple bark [162] and their applications are:

Table 6. Low-cost adsorbents (barks) used for dye removal.

Adsorbents (barks)	Applications (removal of dyes from wastewater)	References
Mangrove bark (<i>Rhizophora apiculata</i>), Teak tree bark (<i>Tectona grandis</i>)	Direct Red-23, Methylene blue	[157, 158]
Neem Tree (<i>Azadirachta indica</i>) & Mango tree (<i>Mangifera indica</i>)	Malachite green (MG)	[159]
<i>Eucalyptus angophoroides</i> bark	Solar red BA and Solar brittle blue A	[160]
<i>Coniferous pinus</i> bark	Crystal violet	[161]
Pineapple bark (<i>Ananas comosus</i>)	MB, Brilliant Green, Congo Red	[162]

7.4 Seeds

There are large number of fruits whose seeds are thrown as waste materials like mango, dates etc. In addition to this there are also cereal seeds having importance in adsorption. Some seeds namely Date seeds [163], *ziziphus jujube* seeds and Mango kernel [164], *Lathyrus sativus* seed husk [165], Rambutan seeds [166], Tamarind seed [167] and Husk of gram seeds [1] and their applications are:

Table 7. Low-cost adsorbents (seeds) used for dye removal.

Adsorbents (seeds)	Applications (removal of dyes wastewater)	references
Date seeds	Methylene blue, Methyl orange	[163]
<i>ziziphus jujube</i> seeds, Mango kernel	Methyl Red	[164]
<i>Lathyrus sativus</i> seed husk	Congo Red	[165]
Rambutan seeds	Malachite green	[166]
Tamarind seed (<i>Tamarindus indica</i> L)	Methylene blue	[167]
Husk of gram seeds	Methylene blue	[1]

7.5 Roots

Like above mentioned parts of plants, roots also play an important role in the removal of pollutants as some are mentioned here namely *Arundo donax* root [137], Tomato plant root [168], *Eichhornia crassipes* [169], Water hyacinth roots [170] and *Azolla filiculoides* [171]. The applications of these roots for the removal of different dyes from wastewater are:

Table 8. Low-cost adsorbents (roots) used for dye removal.

Adsorbents (roots)	Applications (removal of dyes from wastewater)	references
<i>Arundo donax</i> root	Malachite green	[137]
Tomato plant root	Methylene blue and Crystal violet	[168]
<i>Eichhornia crassipes</i>	Congo Red (CR)	[169]
Water hyacinth roots	Indosol Dark-blue GL dye	[170]
<i>Azolla filiculoides</i>	Malachite green	[171]

8. Conclusions

In this review article, the important aspects regarding adsorption phenomenon have been discussed like adsorption mechanism, factors effecting adsorption, isotherms, kinetics and thermodynamics. Various low-cost adsorbents prepared from plant biomaterials have been discussed playing a good role for the removal of contaminants especially dyes from wastewater. The plants of these biosorbents also grow in abundance thus biosorbents prepared from them are low cost and are therefore alternative for various traditional methods used for dye removal from wastewater. The plant biomass can be modified physically as well as chemically to increase the adsorbents capacity for adsorbate removal like dyes, metal ions, aromatic hydrocarbons, pesticides, explosives, radionucleotides and surfactants. So, this budding technology can be applied for the waste water treatment with various advancements making it more efficient, cost effective and ecofriendly.

Author Contributions

Fairooz Ahmad Khan: Investigation, Conceptualization, Formal analysis, Writing-original draft, Writing- review & editing; Shahid Shafi Shah and Bashir Ahmad Dar: Formal analysis, Software, Writing- review & editing; Mazahar Farooqui: Conceptualization, Supervision, Writing- review & editing.

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