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# Removal of Methylene Blue from Aqueous Solution Using Agricultural Wastes as Adsorbent

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**ABSTRACT:** Dye removal from wastewater has been a matter of concern, both in the aesthetic sense and as a potential health hazard. Dye removal from textile effluents on a continuous industrial scale has been given much attention in the last few years, not only because of its visibility problem, but also due to its potential toxicity. There have been various promising techniques for the removal of dyes from wastewater. Adsorption using activated carbon has been studied and widely used because of its high effectiveness. However, its prohibitively high costs has spurred a search for cheaper and effective alternatives. In this review, an extensive list of sorbent literature has been compiled. The review evaluates different agricultural waste materials as low-cost adsorbents for the removal of dyes from wastewater. The review also outlines some of the fundamental principles of dye adsorption on to adsorbents.

**KEYWORDS:** Adsorption, Dyes, Low-Cost Adsorbents, Peanut Hull, Rice Husk, Wastewater Treatment

### I. INTRODUCTION

Dyes are widely used in industries such as textiles, rubber, plastics, printing, leather, cosmetics, etc. to colour for beautifying and their products. Many industries like the textile uses dyes to colour their products and thus produces wastewater containing organics matter with strong colour. In the dyeing processes the percentage of dye lost with wastewater is 50% of the dye because of the low levels of dye-fibre fixation. There are more than 10,000 commercially available dyes with over  $7 \times 10^5$  tonnes of dye stuff produced annually.<sup>[5]</sup> Even just 1.0 mg/L of dye concentration in drinking water could impart a significant colour, making it unfit for human consumption. Furthermore, dyes can affect aquatic plants because they reduce sunlight transmission through water. Also dyes may impart toxicity to aquatic life and may be mutagenic, carcinogenic and may cause severe damage to human beings, such as dysfunction of the kidneys, reproductive system, liver, brain and central nervous system.

Dyes lead to number of environment and health hazards namely:

1. The greatest environment concern with dyes is their absorption and reflection of sunlight entering the water. Light absorption diminishes photosynthesis activity of algae and seriously influences the food chain.
2. Many dye and their breakdown product are carcinogenic, mutagenic and toxic to life. Dyes are mainly introduced into the environment through industrial effluent.
3. Triple primary cancers involving kidney, urinary bladder and liver of dye workers have been reported.

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4. Textile dyes can cause allergies such as contact dermatitis and other diseases, allergic reaction in eyes, skin irritation to mucous membrane and the upper respiratory tract.
5. Reactive dyes form covalent bond with cellulose, woollen and Poly Amide fibres. Certain reactive dyes have caused respiratory sensitisation of workers occupationally exposed to them.
6. The presence of very small amounts of dyes in the water, which are nevertheless highly visible, seriously affects the quality and transparency of water bodies like lakes, rivers and others, leading to damage to the aquatic environment.
7. The highly toxic and mutagenic dyes decrease light penetration and photosynthesis activity, causing oxygen deficiency and limiting downstream beneficial uses such as recreation, drinking water and irrigation.
8. Azo dyes have toxic effects, especially carcinogenic and mutagenic. They enter the body by ingestion and are metabolized by intestinal microorganism causing DNA change.

The removal of colour from waste effluents becomes environmentally important because even a small quantity of dye in water can be toxic and highly visible. Since the removal of dyes from wastewater is considered an environmental challenge and government legislation requires textile wastewater to be treated, therefore there is a constant need to have an effective process that can efficiently remove these dyes. Numerous techniques were used in the recent past for decolourisation of dyes. Among them adsorption technique has got maximum potential for the removal of dyes. Adsorption being a physical process, in-expensive and less time consuming, is widely accepted. [7]

Methods of dye removal from dye-containing industrial effluents have been discussed under three categories (Kayode Adesina Adegoke, et al, 2005):

1. Chemical
2. Physical
3. Biological

Currently the main methods of textile dye treatment are by physical and chemical means such as: Fenton Process, Photo/Ferri-Oxalate System, Photo - catalytic and electrochemical combined treatments, Photo-catalytic degradation using UV/TiO<sub>2</sub>, Sono - chemical degradation, biodegradation, Photo - fenton processes, Integrated chemical - biological degradation, Electrochemical degradation, Adsorption process, ozonation, oxidation, Nano-filtration, Chemical precipitation, Ion - exchange and Reverse osmosis (Mohamad Amran, et al, 2011).

**Table1. Existing and Emerging Processes For Dye Removal (Mohammed Et Al., 2014)**

S.N.	Physical/chemical Methods	Method description	Advantages	Disadvantages
1.	Fenton reagents	Oxidation reaction using mainly H <sub>2</sub> O <sub>2</sub> -Fe(II)	Effective decolorization of both soluble and insoluble dyes	Sludge generation
2.	Ozonation	Oxidation reaction using ozone gas	Application in gaseous state: no alteration of volume	Short half-life (20 min)
3.	Photochemical	Oxidation reaction using mainly H <sub>2</sub> O-UV	No sludge production	Formation of by-products
4.	NaCl	Oxidation reaction using Cl <sup>+</sup> to attack the amino group	Initial and acceleration of azo bond cleavage	release of aromatic amines
5.	Electrochemical destruction	Oxidation reaction using electricity	Breakdown compounds are non-hazardous	High cost of electricity

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6.	Activated carbon	Dye removal by adsorption	Good removal of a wide variety of dyes	Regeneration difficulties
7.	Membrane filtration	Physical separation	Removal of all dye type	Concentrated sludge Production
8.	Ion exchange	Ion exchange resin	Regeneration: no adsorbent loss	Not effective for all dyes
9.	Electro-kinetic coagulation	Addition of ferrous sulphate and ferric chloride	Economically feasible	High sludge production

Among treatment technologies, adsorption is rapidly gaining prominence as a method of treating aqueous effluent. Some of the advantages of adsorption process are possible regeneration at low cost, availability of known process equipment, sludge-free operation and recovery of the sorbate (Amit Bhatnagar, et al, 2010). Adsorption is a well-known equilibrium separation process and an effective method for water decontamination applications. 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. Activated carbon is the most widely used adsorbent for dye removal because of its extended surface area, micro-pore structures, high adsorption capacity and high degree of surface reactivity. However adsorption by activated carbon has some limitations such as the cost of the activated carbon, the need for regeneration after exhausting and the loss of adsorption efficiency after regeneration. This has led to search for cheaper alternatives (Mohd. Rafatullah, et al, 2010).

There have been many attempts to find inexpensive and easily available adsorbents to remove the pollutants such as the agricultural solid wastes where according to their physico-chemical characteristics and low cost may be factors to be considered as good potential adsorbents. Agricultural waste materials have little or no economic value and often pose a disposal problem. The utilization of agricultural waste is of great significance for treatment of wastewater. A number of agricultural waste materials are being studied for the removal of different dyes from aqueous solutions at different operating conditions (Amit Bhatnagar, et al, 2010). Agricultural wastes are renewable, available in large amounts and less expensive as compared to other materials used as adsorbents. Agricultural wastes are better than other adsorbents because the agricultural wastes are usually used without or with a minimum of processing (washing, drying, grinding) and thus reduce production costs by using a cheap raw material and eliminating energy costs associated with thermal treatment (Kayode Adesina Adegoke, et al, 2015). Agricultural waste includes (but not limited to) banana peel, rice husk, peanuts hull, orange peel, date pit, broad bean peel, almond shells, etc. The use of these low-cost bio-sorbents is recommended since they are relatively cheap or of no cost, easily available, renewable and show high affinity to dyes (K. S. Bharathi, et al, 2012).

## II. REMOVAL OF DYES BY LOW - COST SORBENTS

Wang et al. (2008) reported capacity and mechanism of adsorption of two basic dyes, namely Malachite green and Methylene blue by rice bran and wheat bran. The adsorption of both basic dyes was pH dependent. Both the dyes are basic in nature, which upon dissociation releases coloured dye cations into solution. The adsorption of these positively charged dye groups on to the adsorbent is primarily influenced by the surface charge on the adsorbent, which in turn is influenced by the solution pH. As the pH is increased, greater cation removal is facilitated. The kinetic experimental data were analyzed using four kinetic equations including pseudo-first-order equation, pseudo-second order equation, external diffusion model and intra-particle diffusion model. The experimental data were fitted with Langmuir and Freundlich isotherms and the results showed that the Langmuir isotherm exhibited a little better fit to the methylene blue adsorption data by both adsorbents. While, the Freundlich isotherm seemed to agree better with the Malachite green adsorption, the effects of particle size, adsorbent concentration and solution ionic strength on the adsorption of the two dyes were studied and it was found that the adsorption capacity decreased with an increase in the adsorbent

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concentration. This is due to the fact that some of the adsorption sites remained unsaturated during the adsorption process and inter-particular interaction.

Broad bean peel an agricultural waste chopped, sieved, washed and oven dried at 60 °C for 48 h was used by Hameed and El-Khaiary (2008) for removal of cationic dye (methylene blue) and adsorption capacity of 192.7 mg g<sup>-1</sup> was reported. It was noted that adsorption of dye decreased with an increase in the initial methylene blue concentration. The adsorption of dyes was reported to follow Langmuir adsorption model and pseudo-first-order kinetic model.

Agricultural solid waste such as seed hull has been used as adsorbents for the removal of dye from wastewater. These materials are available in large quantities and may have potential as adsorbents due to their physico-chemical characteristics and low cost. Sunflower seed hull activated with sulphuric acid has been used for the removal of Acid violet 17 by Thinakaran et al. (2008). The Langmuir adsorption capacity for the same was found to be 116.27 mg g<sup>-1</sup>. This material was used for the removal of methyl violet by Hameed (2008b), without any physical or chemical treatment. The equilibrium data were analyzed using the Langmuir, Freundlich and Temkin isotherm models and the equilibrium process was described well by the Freundlich isotherm model. The maximum sorption capacity was found to be 92.59 mg L<sup>-1</sup> at 30 °C. Pumpkin seed hull as low-cost adsorbent was investigated by Hameed and El-Khaiary (2008a), for the removal of basic dye (methylene blue) from aqueous solutions. Authors noted that adsorption of dyes was rapid on the outer surface, followed by slower adsorption inside. The removal of methylene blue was high at pH 6–9. The results obtained by the workers indicated that the multilayer adsorption isotherm was found to have the best fit to the experimental data, suggesting multilayer adsorption on a homogeneous surface. The adsorption capacity was determined from the parameters of adsorption isotherms and was found to be 141.92 mg g<sup>-1</sup>.

Various agricultural wastes for dye degradation includes coir pith (Namasivayam and Kavitha 2002), orange peel (Rajeswari et al. 2001), banana peel (Annadurai et al. 2002), rice husk (Malik 2003), straw (Kannan and Sundaram 2001), date pit (Banat et al. 2003a), oil palm trunk fiber (Hameed and El-Khaiary 2008), durian (*DurioZibethinus Murray*) peel (Hameed and Hakimi 2008), guava (*Psidium guajava*) leaf powder (Ponnusami et al. 2008), almond shell (Ardejani et al. 2008), pomelo (*Citrus grandis*) peel (Hameed et al. 2008), broad bean peel (Hameed and El-Khaiary 2008), peanut hull (Tanyildizi 2011), *Citrullus lanatus* rind, etc.(Bharathi and Ramesh 2013). The adsorption capacity of these sorbents is listed in Table 2.

**Table 2: Reported Adsorption Capacities Q<sub>m</sub>(mg/g) Of Different Agricultural Waste Materials (KS Bharathi, et al, 2013)**

Adsorbent	Dye	Maximum adsorption capacity (mg/g)
Peanut hull	Reactive dye	55.5
Rice husk	Acid yellow 36	86.9
Banana peel	Basic blue 9	20.8
Coir pith	Congo red	2.6
Straw	Basic blue 9	19.82
Date pit	Basic blue 9	17.3
Oil Palm Fiber: Activated carbon	Malachite green	149.35
Durian shell: based activated carbon	Methylene blue	289.26
Almond shell	Direct red 80	90.09

### III. ADSORPTION ISOTHERMS

Adsorption is usually described through isotherms, that is, the amount of adsorbate on the adsorbent as a function of its pressure (if gas) or concentration (if liquid) at constant temperature. The quantity adsorbed is nearly always normalized

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by the mass of the adsorbent to allow comparison of different materials. To date, 15 different isotherm models were developed. Two adsorption isotherms are widely used namely Langmuir and Freundlich isotherms.

### Langmuir Isotherm

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. The Langmuir isotherm is expressed as

$$\frac{X}{M} = \frac{1}{q_{max}} + \frac{1}{q_{max} \cdot b \cdot C_e}$$

where  $b$  is the constant that increases with increasing molecular size,  $q_{max}$  is the amount adsorbed to form a complete monolayer on the surface ( $\text{mg g}^{-1}$ ),  $X$  is the weight of substance adsorbed ( $\text{mg}$ ),  $M$  is the weight of adsorbent ( $\text{g}$ ),  $C_e$  is the concentration remaining in solution ( $\text{mg L}^{-1}$ ).

### Freundlich Isotherm

Freundlich proposed an empirical relationship between amount of gas adsorbed by unit mass of adsorbent and pressure at a particular temperature. The following equation was proposed for the Freundlich Adsorption Isotherm:

$$\frac{X}{M} = k \cdot C^{1/n}$$

Where,  $X$  is the mass of the gas adsorbed,  $M$  is the mass of the adsorbent,  $C$  is the concentration,  $k$  and  $n$  are constants which depends on the nature of the adsorbent and the fluid at a particular temperature.

## IV. FACTORS AFFECTING ADSORPTION

Main factors affecting the adsorption process is listed below,

### Initial Dye Concentration

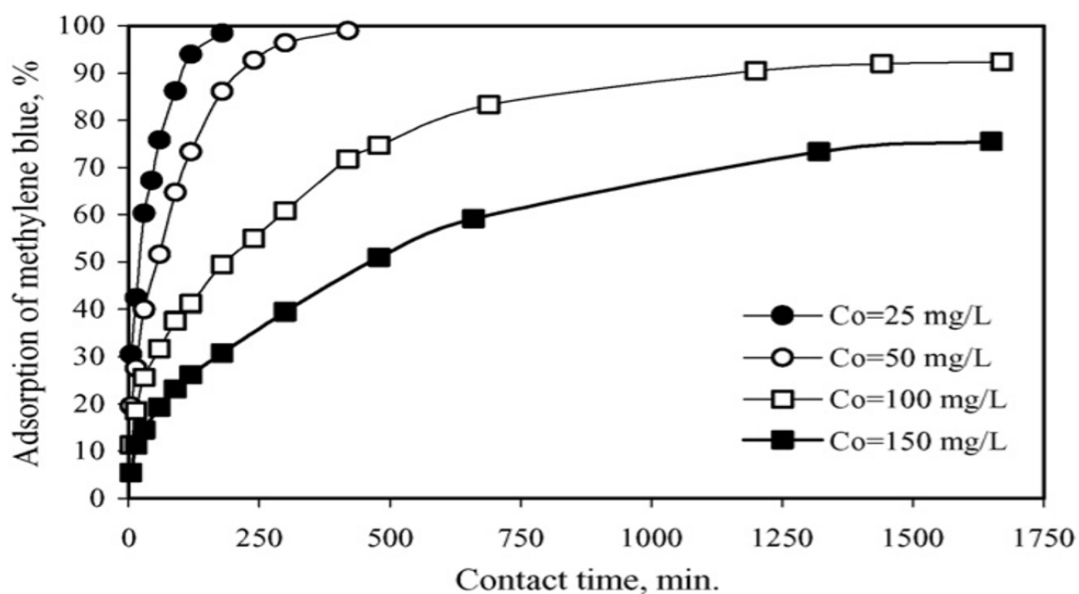


Fig. 1 Effect of initial dye concentration on the adsorption of methylene blue on the dehydrated peanut hull (Dursun Ozer et al., 2007)

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The initial dye concentration of an effluent is important since a given mass of sorbent material can only adsorb a fixed amount of dye. The effect of the initial of dye concentration depends on the immediate relation between the concentration of the dye and the available binding sites on an adsorbent surface. Normally the dye removal will decrease with increase in initial dye concentration. This is because for a given mass of adsorbent; the amount of dye it can adsorb is fixed. The higher the concentration of the dye, the smaller the volume it can remove. At a low concentration there will be unoccupied active sites on the adsorbent surface, and when the initial dye concentration increases, the active sites required for adsorption of the dye molecules will be lacking. It is also reported that the time taken to reach equilibrium increased with increasing concentration.

Ozer et al. (2007) carried out of an experiment by taking different initial MB concentration (25-150 mg/l) at temperature 25 °C; pH 3.5; adsorbent dosage=1gdm<sup>-3</sup>; and RPM=150 to find effect of dehydrated peanut hull on removal of methylene blue. They reported that the adsorption capacity increases from 73 to 98 % with increases initial concentrate ion from 25 to 150 mg/l as in figure above.

### Effect Of Adsorbent Dose On Adsorption Of Methylene Blue Dye

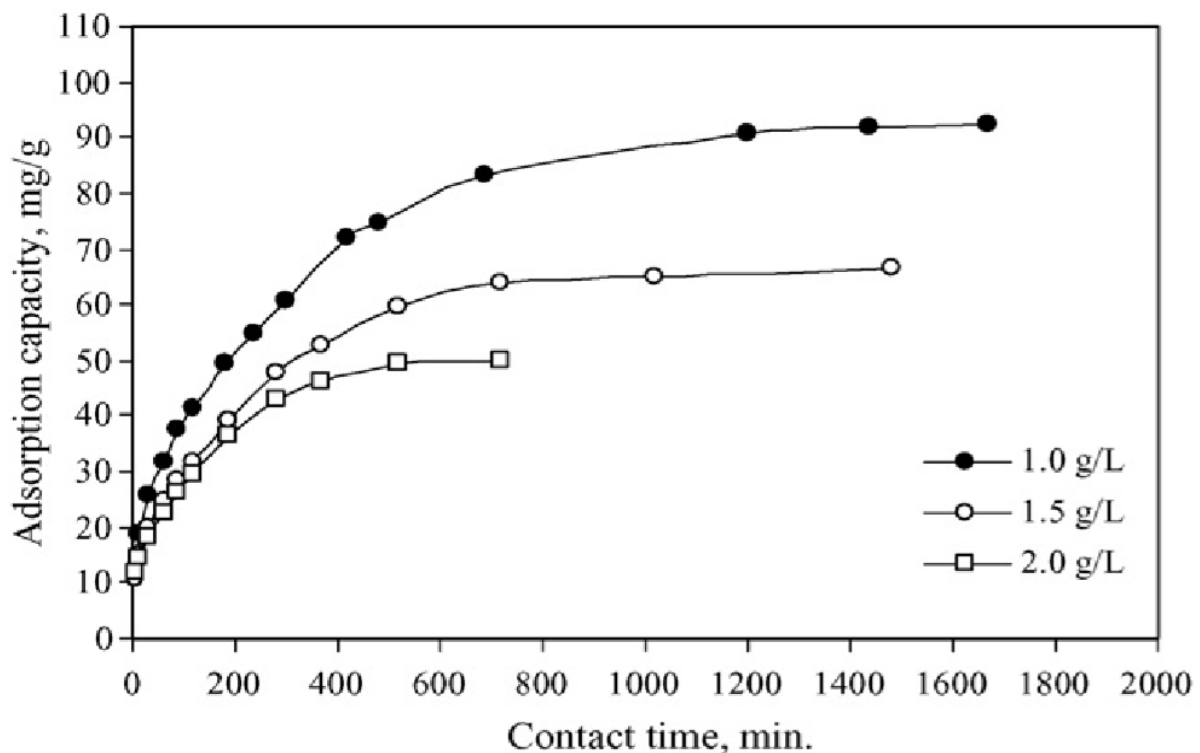


Fig. 2 Effect of adsorbent dosage on the adsorption of MB on dehydrated peanut hull (Dursun Ozer et al., 2007)

The effect of adsorbent dosage on the adsorption process was carried out by preparing adsorbent-adsorbate solution with different amounts of adsorbents added to fixed initial dye concentration and shaken together until equilibrium time (Dursun Ozer, et al, 2007). It was observed that rate of dye removal increased with increasing adsorbent dosage. Initially the rate of percent dye removal was found to be rapid which slowed down as the dose was increased. This phenomenon can be explained, based on the fact that at lower adsorbent dose the adsorbate (dye) is more easily accessible and because of this, removal per unit weight of adsorbent was higher. With rise in adsorbent dose, there was less commensurate increase in adsorption, resulting from many sites remaining unsaturated during the adsorption. But after a certain dosage the increase in removal efficiency was insignificant with respect to increase in dose.

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Ozer et al. (2007) carried out of an experiment by taking different adsorption dosage at using initial concentration of  $100\text{mg}\cdot\text{dm}^{-3}$ ; temperature of  $25^\circ\text{C}$  to find effect of dehydrated peanut hull on removal of methylene blue dye. It was observed that the adsorption capacity decreased from 84 to 49.85 % with increase in adsorbent dose from 1 to 2 g/L as shown in Fig. 2.

### Effect of pH on Adsorption of Methylene Blue Dye

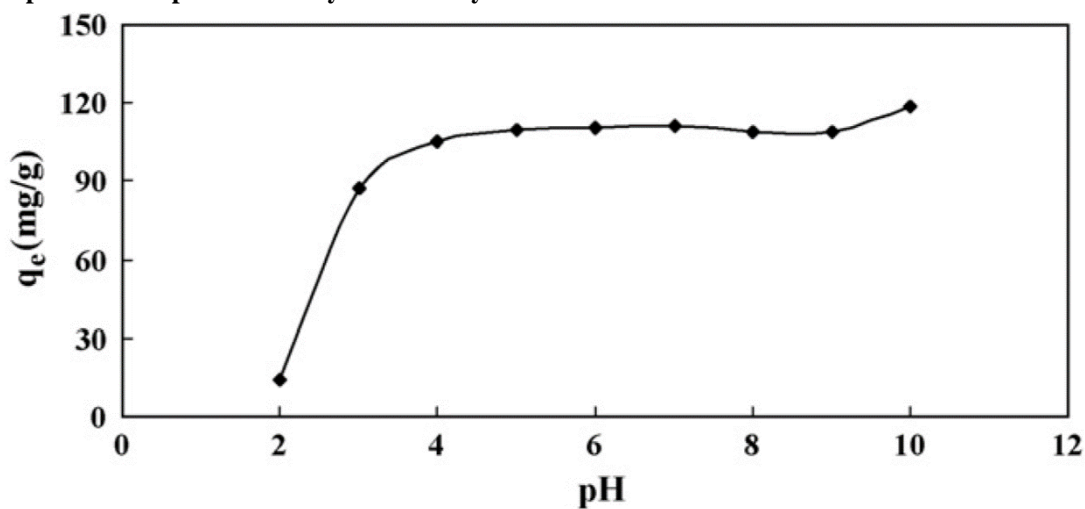
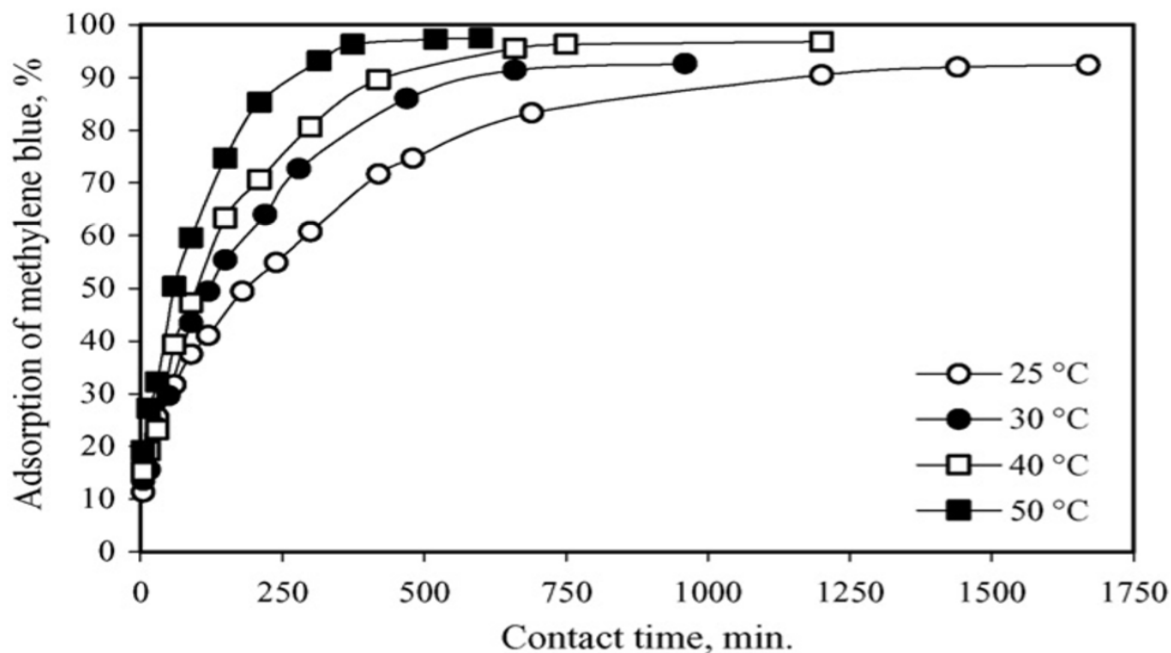


Fig. 3 Effect of pH on Methylene Blue biosorption using pineapple stem (Hameed, et al., 2009)

The pH factor is very important in the adsorption process especially for dye adsorption. The pH of a medium will control the magnitude of electrostatic charges which are imparted by the ionized dye molecules (KS Bharathi, et al, 2013). As a result, the rate of adsorption will vary with the pH of an aqueous medium. Generally, at low pH solution, the percentage of dye removal will decrease for cationic dye adsorption, while for anionic dyes the percentage of dye removal will increase (Mohamad Amran, et al, 2011). In contrast, at a high pH solution the percentage of dye removal will increase for cationic dye adsorption and decrease for anionic dye adsorption. For cationic dyes, lower adsorption of dye at acidic pH is probably due to the presence of excess  $\text{H}^+$  ions competing with the cation groups on the dye for adsorption sites. As surface charge density decreases with an increase in the solution pH, the electrostatic repulsion between the positively charged dye and the surface of the adsorbent is lowered, which may result in an increase in the extent of adsorption. With an increase in the solution pH, the electrostatic repulsion between the positively charged cationic dyes and the surface of adsorbent is lowered and consequently the removal efficiency is increased.

Hameed, Krishni and Sata (2009) carried out experiment by taking different pH of solution at initial concentration  $250\text{mg/L}$ , temperature of  $30^\circ\text{C}$ , agitation rate  $120\text{rpm}$  and fixed adsorbent dosage at  $0.3\text{g}$  to find out the effect of pH on the removal of Methylene Blue using pineapple stem as adsorbent. They reported that the removal yield increased from  $13.29$  to  $104.5\text{mg/g}$  with increase in pH from 2 to 10 as shown in Fig. 2.

**Effect of Temperature on Adsorption of Methylene Blue Dye**



**Fig. 4 Effect of temperature on the adsorption of MB on dehydrated peanut hull (Dursun Ozer et al., 2007)**

Temperature is an indicator for the adsorption nature whether it is an exothermic or endothermic process. If the adsorption capacity increases with increasing temperature then the adsorption is an endothermic process. This may be due to increasing the mobility of the dye molecules and an increase in the number of active sites for the adsorption with increasing temperature.

Ozer et al. (2007) carried out an experiment by maintaining different temperature at initial concentration  $100 \text{ mg/dm}^3$ , pH of 3.5; adsorbent dose of  $1 \text{ g/dm}^3$ ; and RPM of 150 to evaluate the effect of dehydrated peanut hull on the removal of methylene blue dye. It was observed that the adsorption capacity increased from 92 to 98.6 % with increase in temperature from 25 to  $50^\circ\text{C}$ (Fig.4).

Other factors which affect the adsorption process are contact time, speed of agitation and ionic strength.

Batch wise experiment was conducted by Hameed, Krishni and Sata (Hameed, et al., 2009) at  $30^\circ\text{C}$  to study the effect of initial concentration of methylene blue, contact time and pH on dye adsorption. It was found when the initial dye concentration increased from 25 to  $300 \text{ mg/L}$ , the loading capacity of PS increased from 18.33 to  $116.95 \text{ mg/g}$  and the percentage removal decreased from 96.70 to 54.50%. also the dye up-take was found to increase from 13.29 to  $104.50 \text{ mg/g}$  for an increase in pH from 2 to 10. A consistent increase in adsorption capacity of the PS was noticed as the pH increased from 2 to 4, but further increase in pH from 4 to 9 seemed not affect the sorption in any wise. Equilibrium data agreed well with Langmuir isotherm model with monolayer adsorption capacity of  $119.05 \text{ mg/g}$  at  $30^\circ\text{C}$ .

Similar trade of result was also shown by Ozer, Dursun and Ozer (Ozer et al. 2007) for adsorption of methylene blue using dehydrated peanut hull (DPH) at different set of experimental conditions. It was found that for the pH variation from 2 to 10, adsorbent dosage of 1-2 g/l and 100 – 400 mg/l initial dye concentration, the percentage removal of methylene blue by DPH was between 49.85 – 98 %.

Same trade of results was shown by all the studies conducted by various researcher using various types of dye and different agricultural waste for adsorption process. Table 3 compares results obtained by various authors and researchers



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Dye	Absorbent	Operating condition							Ref.
		pH	Initial dye conc.(mg/l)	Adsorbent dosage(g/l)	Temperature (°C)	Adsorbent size	Contact time (hr)	Removal (%)	
Methylene Blue	Pineapple Stem	2-10	25-350	1.5	30	355-500 micron	5:30	54.5 - 96.7	[17]
Methylene Blue	Dehydrated Peanut Hull	2-10	100-400	1-2	25-50	50-100 mesh	5	49.85 - 98	[14]
Methylene Blue	Rice Husk	3-8	10-124	0.67-4	32	60 – 85 mesh	4	28.5 – 88.7	[19]
Amaranth (Am), Sunset Yellow(SY), Fast green FCF(FG)	Peanut Hull	2-11	10-100	1-20	22	20-100 mesh	2	98.7–72.83(Am), 99.57-70.42(SY), 98.2-74.31(FG)	[11]
Direct Red and Acid Brilliant Blue	Banana pith	3-11	10-40	1-10	30	53 microns	0:40	83.3 (DA) 90.0 (ABB)	[22]
Methylene Blue	Activated Carbon(CAC) and Indigenously Prepared Activated Carbon(IPAC)	2-10	100-400	8-11.6	30	90-250 micron	2	99.2	[24]
Methylene Blue	Rice husk	5-9	35-100	1-6	32	0.5mm	5	71-86	[25]
Direct Red 23(DR 23) Direct Red 80(DR 80)	Orange peel	2	50-125	0.5-2	25	3.36mm	8	89-76 (DR 23) 91-76 (DR 80)	[26]
Reactive green (RG) Reactive Orange (RO) Reactive Yellow (RY)	Peanut hull	2	25-250	0.5-9	25	80 mesh	3	84.2 (RG) 87.36 (RO) 88.49 (RY)	[27]
Methylene blue	Rice husk	2	100	3	29	40 mesh	5	98.95	[28]
Methylene blue	Corn husk	6.2	5-30	0.5-10	25-28	NA	2	90	[29]

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Methylene blue	Parthenium Hystrophorous	2-10	10-400	2-20	25	1 mm	2:20	90	[31]
Reactive Orange 12 (RO) Reactive Red 2 (RR) Reactive Blue 4 (RB)	Coir pith	1-3	20	2-2.5	20	400-500 micron	7	82(RO) 77(RR) 72(RB)	[32]
Methylene Blue	Cashew nut shell	2-10	20-100	5-30	30-60	30-200 mesh	1	97.57	[36]
Methylene Blue	Cashew nut shell	2-10	50-250	0.5-3	30	NA	2	99.17-55.48	[39]
Acid Violet (AV) Congo Red (CR) Rhodamine B (RB)	Banana pith	2-11	20-200	2.5-30	30	53-1000 micron	4	57-80	[15]
Rhodamine B	Banana pith	4.81-9.23	20-100	10	30	53-1000 micron	1:30	87-54	[13]

### V. CONCLUSION

In this paper, a wide range of agricultural waste materials used by various authors as low-cost adsorbents is reviewed. The use of these low-cost biosorbents increase its usefulness as they are relatively cheap or of free of cost, easily available, renewable and shows high affinity for dyes. Various materials used by various authors include peanut hull, rice husk, banana pith, coir pith, etc. Removal efficiency was found to vary between 28% to 99% for a pH of range 3 - 11. Similarly write effects of other parameters in one sentence each. The process of biosorption requires further investigation in the direction of modelling, regeneration of biosorbent and immobilization of the waste materials for enhancing the efficiency and recovery. Further, more emphasis should be on predicting the performance of the adsorption process for dye removal from industrial effluents.

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