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# Adsorption Efficacy Of Limonia Acidissima Fruit Shell Based Adsorbent For Methyl Red

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***Abstract:***
*The toxic dye methyl red pose harmful effects on human and aquatic ecology. The focus of this work is to use different adsorbents prepared from Wood apple (Limonia acidissima) fruit shell for removing methyl red from aqueous solutions. The prepared adsorbents LAS1; chemically activated adsorbents LAS2 and LAS3; were tested for their adsorption potentials with respect to the dye. The batch adsorption experiments were carried out by using 100 mg $L^{-1}$ dye solution with adsorbent dose of 0.030 g with a contact time of 90 min at 300 rpm. The adsorbents prepared by chemical activation showed significant enhancement of dye removal efficiency compared to the raw material, indicating the effectiveness of chemical activation in improving adsorption performance.*

***Keywords:*** *Adsorption, Methyl red, Limonia acidissima fruit shell*, *Adsorption potential*

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## I. Introduction

Water plays an essential role in sustaining biological systems. Water quality gets impacted due to discharge of different toxicants into the water streams [1]. Scarcity of pure water affect different service sectors. The main cause of water pollution is due to effluents discharged from industries like paper, textile, leather etc. Various acids, bases, toxic organic and inorganic solids and colors are present in this effluent [2]. Increased population increases demand of pure water. But, decrease in water quality, due to huge urbanization, increase in industrial area, change in climate and environment may lead to crisis of pure water [3]. For domestic purposes and drinking, only 3% of groundwater is sufficient [4]. The pure water resources are decreasing due to entry of number of new pollutants in water bodies [5]. Some of the contaminants include biological, pharmaceutical, inorganic, plastic particles and radioactive wastes [6]. The waste water released from industries like paper, plastic, leather, food, textile, paints and varnish, pulp, pharmaceutical etc. mainly includes dyes like methylene blue, rhodamine B, methyl orange, Congo red, disperse violet 26, methyl red, crystal volet etc. The effects of these dyes on human include respiratory problem, irritation, teratogenicity and carcinogenicity. The presence of dyes in water restricts light penetration affecting photosynthesis and destruction of aquatic ecology. Among all the wastes, dyes are responsible for creating unwanted colors to the water [7, 8]. Methyl red dye is responsible to cause sensitization of eyes, skin. It's inhalation or swallowing cause irritation of digestive tract [9].

The term adsorption is a mass transfer process related to transfer of water pollutants onto a solid adsorbent [10]. Physical or chemical processes use a wide variety of adsorption forces which binds to particular pollutant [11]. Adsorption is most widely used method of removal of dyes from industrial effluents [12].

Rosemary root adsorbent prepared with use of $H_3PO_4$ adsorb 50 mg $g^{-1}$ methyl red dye at adsorbent dose of 15 mg. The process of adsorption is best described as Freundlich isotherm with a pseudo second order kinetics [13]. Prepared activated carbon of Lemongrass leaf adsorbs methyl red at pH 2. The process of adsorption is endothermic following physisorption [14]. At pH 6, about 99.2 % of methyl red dye with 70 mg $L^{-1}$ concentration was removed in 40 min equilibrium time using biochar prepared from *Rumex abyssinicus* [15]. Methyl red was degraded with hyacinth and *Tinospora cordifolia* based bioadsorbent at pH 4. The study incorporated the effect of interfering ions during adsorption [16]. Biosorbent prepared from sacred flowers specially like marigold and rose petals showed 61.16 % and 56.08 % methyl red dye decolorization [17]. Leaf and bark-based adsorbent of *Leucaena leucocephala* remove 81.2 to 90 % methyl red with a contact time of 90 min [18]. The Langmuir adsorption model and monolayer adsorption of methyl red dye was effected by use of adsorbent powder made from Guava bark [19]. The adsorption of methyl red on the surface of *Shorea robusta* sawdust occurs at time of 3 to 4 h [20]. Biochar of *Delonix regia* was able to carry out adsorption/desorption cycle for six times with negligible loss in the adsorption and desorption process of methyl red dye [21].

The biochar prepared by physical and chemical activation processes of fennel seeds removes methyl red dye with adsorption capacity ranging from 26-135 mg $g^{-1}$ [22]. A novel hyper cross linked polyamine resin (BPA-PEA) showed maximum adsorption capacity of 230.6 mg $g^{-1}$ methyl red following a chemisorption way,

achieving equilibrium within 6 hours [23]. NiO@Hyphaene thebaica seed derived porous carbon adsorb methyl red by a chemisorption process following pseudo second order kinetic model and has good reusing capability [24]. Egg shell-based cost-effective adsorbent removes 82.2 % methyl red dye with a monolayer dye adsorption capacity of 1.66 mg $g^{-1}$ at pH 2 [25]. Biocarbon obtained from waste carbon digest have adsorption capacity 31-113 mg $g^{-1}$ for methyl red from aqueous solutions [26]. Agricultural waste material Pomelo peel at pH 6.5 is highly selective bioadsorbent for removal of methyl red by Langmuir adsorption isotherm model [27].

The aim of this work is to prepare adsorbent material from the shells of Wood apple fruit (*Limonia acidissima*) and to study its efficiency for removal of methyl red dye from aqueous solutions. The study was explored to compare adsorption efficiency of prepared bioadsorbent activated by use of chemical activators like KOH and $H_2SO_4$ with acetic acid.

## II. Experimental

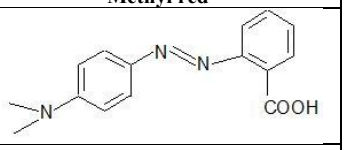
**Chemicals and Materials**:

**a) Adsorbate and other chemicals**:

AR Grade reagents were used throughout the experiment. They were used as such without further purification.

A stock solution of methyl red dye of 1000 mg $L^{-1}$ was prepared by dissolving 1 g in minimum amount of alcohol, adding two drops of 0.1 M NaOH to remove turbidity and then diluting with distilled water. The test solutions were prepared by required dilution of stock solution. The properties of dye are as shown in Table no 1.

**Table no 1:** Adsorbate properties

| **Dye** | **Methyl red** |
|---|---|
| Structure |   |
| Molecular formula | $C_{15}H_{15}N_3O_2$ |
| λmax | 430 nm |
| Molecular weight | 269.3 g $mol^{-1}$ |

**b) Preparation of adsorbents**:

Wood apple shell having botanical name *Limonia acidissima* [28] was used for preparation of adsorbent. Wood apple was collected from local fields. The shell was separated, cut into small pieces and washed thoroughly number of times with double distilled water for removal of dirt and other soluble impurities. It was sundried and crushed using mechanical grinder and subjected to further washing. It was kept in an oven at 383 K for 24 h. This material was then passed through sieves and used as adsorbent. It was named Limonia acidissima shell adsorbent, LAS1. The synthesis of chemically activated adsorbents is desired from LAS1. The dried LAS1 powder was kept for soaking in the solutions of KOH and concentrated $H_2SO_4$ respectively at 1:2 ratio (weight of LAS1 to volume of activating solution) for 24 h. The materials were washed with double distilled water, dried and then kept in muffle furnace at 600 K for 1 h. Then washed number of times with distilled water till neutral pH, dried, sieved. The adsorbent prepared by the use of impregnating agent KOH was named LAS2. The obtained dried powder after $H_2SO_4$ treatment was again mixed with $CH_3COOH$ for further treatment, then filtered, washed, dried, sieved and named as LAS3 (**Fig no. 1**).

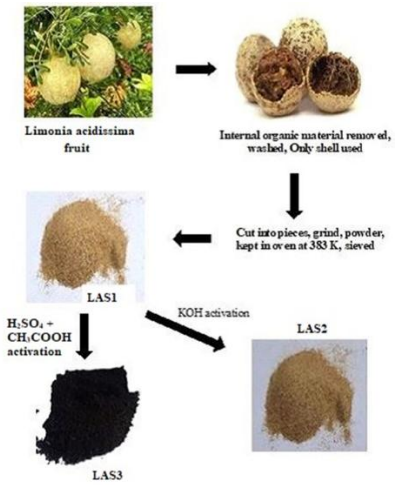



**Fig. no. 1**: Preparation of adsorbent

**c) Batch adsorption procedures**:

To study adsorption, a technique of batch adsorption was carried out.

A 25 mL 100 mg $L^{-1}$ dye solution was taken in 250 mL conical flask and added with 0.030 g of adsorbent. The flask was shaken for 90 min maintaining agitation speed of 300 rpm at 25°C. The contents were filtered through a Whatman filter paper. The concentration of methyl red dye in the effluent was determined using a Systronics digital spectrophotometer model 104. The absorbance measurements were carried out at 430 nm after verification of Beer's law.

The percentage removal of dye was determined using Eqn. (1). Adsorption capacity (qe), was calculated using Eqn. (2).

$$\% \text{ Removal of Dye} = \left[\frac{Co - Ce}{Co}\right] \times 100 \quad \text{Eqn. (1)}$$

Where, Co = Initial dye concentration
Ce = Equilibrium dye concentartion

$$q_e = \left[\frac{Co - Ce}{M}\right] \times V \quad \text{Eqn. (2)}$$

Where, $q_e$ = Equilibrium adsorption capacity in mg $g^{-1}$
M = Mass of adsorbent in g
V = Volume of dye in lit

## III. Results And Discussion

**Comparative study of adsorbent potential of LAS1, LAS2 and LAS3**:

The adsorption potential of three different adsorbents in the terms of % dye removal and adsorption capacity, qe was studied. The results are summarized in Table no. 2.

**Table no 2:** Adsorption potential of adsorbents

| Dye → | Methyl Red | |
|---|---|---|
| Adsorbent ↓ | Dye Removal (%) | qe (mg/g) |
| LAS1 | 79.60 | 66.33 |
| LAS2 (KOH Activated) | 87.84 | 73.20 |
| LAS3 ($H_2SO_4$ + $CH_3OOH$ Activated) | 99.57 | 82.97 |

It was observed that the adsorption behavior is following the trend in the order LAS3 > LAS2 > LAS1 with respect to percentage of dye removal and equilibrium adsorption capacity for the dye, methyl red. Chemical activation with respect to $H_2SO_4$ + $CH_3COOH$ shows almost 99 % of dye removal. This may be due to improvement in adsorption surface, porous nature and more availability of functional groups. The activating agents introduce number of oxygenated surface groups like -COOH, -C=O, -OH on the surface of adsorbent [29-30]. This is responsible for creation of H-bond sites. A strong pi-pi interaction between aromatic rings of methyl red and that on carbon surface of adsorbent occurs and dye get adsorbed. Raw adsorbent LAS1 show lowest adsorption efficiency due to unmodified surface area of adsorbent.

**b) Effect of amount of adsorbent**:

The adsorbent experiments were carried out by using different quantities of adsorbents LAS1, LAS2 and LAS3 for methyl red. If the amount of adsorbent is increased, the percentage removal of dye is increased (Fig. 2) due to availability of extra surface area. But it was observed that keeping the initial concentration of dye constant and increasing the adsorbent dose, decrease the adsorption capacity (Table no 3). This is due to increase in adsorption sites in excess of dye molecules which are adsorbed. The chemical activation of adsorbent materials using KOH and $H_2SO_4$ + $CH_3COOH$ are responsible for improvement of adsorption. Chemical activation process improving the porous nature of adsorbent improving the removal efficiency.

**Table no 3:** Effect of variation of amount of adsorbent on adsorption capacity for methyl red dye

| Amount of adsorbent added, mg | Adsorption capacity, mg $g^{-1}$ | | |
|---|---|---|---|
| | LAS1 | LAS2 | LAS3 |
| 0.01 | 182.58 | 202.04 | 235.42 |
| 0.02 | 94.67 | 107.38 | 123.93 |
| 0.03 | 66.33 | 73.20 | 82.97 |
| 0.04 | 51.53 | 56.04 | 62.23 |
| 0.05 | 43.62 | 44.91 | 49.78 |
| 0.06 | 37.30 | 37.46 | 41.36 |
| 0.07 | 31.97 | 32.36 | 35.30 |
| 0.08 | 27.98 | 28.31 | 31.20 |
| 0.09 | 24.87 | 25.16 | 27.60 |
| 0.1 | 22.38 | 22.65 | 24.95 |

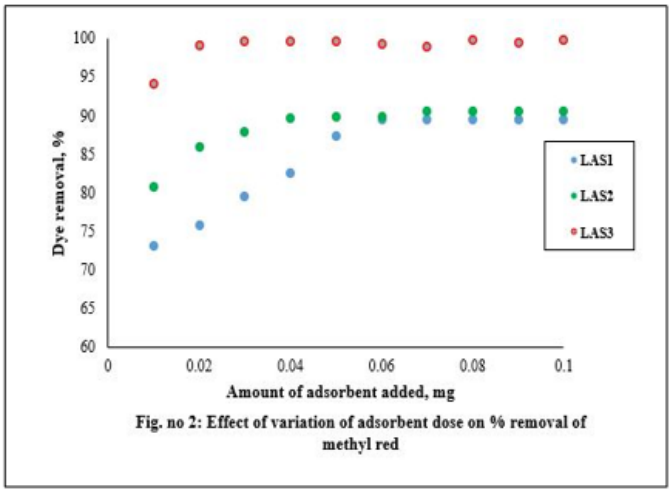



**Fig. no 2: Effect of variation of adsorbent dose on % removal of methyl red**

## IV. Conclusion

Different adsorbents prepared from wood apple fruit shell were utilized for adsorption of methyl red dye. The study of adsorption potential reveals LAS3 was the best adsorbent for removal of methyl red dye with its 99.57 % removal and maximum adsorption capacity of 82.97 mg $g^{-1}$. In conclusion, the prepared adsorbent effectively removes methyl red dye from aqueous solutions.

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