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RESEARCH ARTICLE

THE UTILIZATION OF BIO-ADSORBENTS FOR REMOVAL OF TEXTILE DYE (INDIGO CARMINE) AT AQUEOUS SOLUTION

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ABSTRACT

Exclusion of Indigo carmine color by adsorption method on eggshell (ESP), date palm seeds (DPS) and papaya seeds powder (PSP) from watery arrangement was researched. The examination done by following group mode strategies, for example, portion sum mg/g, pH, focus, contact time and speed rpm separately. The surface reading examined through; FTIR-Fourier change electron microscopy, SEM-Scanning electron microscopy and EDS-Energy dispersive x-beam investigation. Best adsorption follows dynamic pseudo - second request motor plots. Adsorption isotherms best fitted in Langmuir and Freundlich plots. Results pronounced that most extreme expulsion of color was accomplished at powerful portion for ESP is 500mg, DPSP 100mg while for PSP is 80mg at enhanced pH3, utilizing 50mg/l convergence of color, contact time and 100speed rpm study performed at room temperature.

KEYWORDS

Indigo carmine dye, removal, textile wastewater, adsorption, use of adsorbents.

1. INTRODUCTION

In many industries using stuff of dyes vi textile, leather cosmetics, plastics, paper etc (Safi et al., 2006; Gieetha et al., 2009). Dyes are widely used to colour their products. These pigments of dyes are very harmful to mankind. Colors have complex molecular structure, and they are stable to oxidizing agent resistant to aerobic digestion and so recalcitrant and difficult to degrade and remove (Mittal et al., 2010). IUPAC name of dye Indigo Carmine is (2E)-3oxo-2(3oxo-5-Sulfonato- 1H-Indol-2-ylidne0-1H-Indoll-5sulfonate (Ramya et al., 2008).

Table 1: Dye chemical structure and properties

Dye	CAS number	Chemical formula	Molecular weight	Wavelength nm
Indigo carmine	86-22-0	$C_{16}H_8N_2Na_2O_8S_2$	466.35	615:00nm

Water soluble with di sulphate derivate of the indigo carmine usually is blue colored. Dye major application is used in textile industrial application as a color agent. The dye has been employed as agent in confectionary food items besides cosmetic and as additive in pharmaceutical medicine and capsules. Additional it is also used as redox indicator in analytical chemistry and in diagnostic Aid in Kidney function test. In a biology also used as microscopic stain (Ojuz et al., 2005). Indigo carmine is an acidic dye has been used to color protein wool fiber and silk (Ojuz et al., 2005; Chen et al., 2003). At this present time various combinations and methods are utilized to treat dye waste fluid, like organic treatment, chemical coagulation, electrocoagulation and ozonation (Sonwane and Shrivastav, 2008; Seleuk, 2005; Kasaoz, 2005; Lacase and Bauman, 2004). The capability of adsorption technique has been proved an excellent efficiently on broad level removing range of adsorbent (Ramya et al., 2008). Adsorption developed a good value by exclusion of colors. Owing to their biologically and chemically strength on water-based techniques. Previously reported A limited quantity of technology indigo carmine dye by adsorption method studies have been conducted away from textile industrial wastewater by using activated carbon from aqueous solution using PWCAC carbon. In this paper authors made efforts in study removal of indigo carmine dye from aqueous solution using agriculture raw material following as chicken Eggshell, date palm seeds and papaya seeds powder.

2. MATERIAL AND METHODS

2.1 Preparation method and selection of Adsorbent

The precursor material eggshell and papaya seeds were collected from local market Hyderabad. While date palm seeds were collected from Date Palm Research Institute, Shah Abdul Latif University, Khairpur, Sindh Pakistan. Adsorbent's materials were thoroughly washed with tap water then double distilled water. Then ESP and PSP were placed in Electric oven at 105℃ temperature for 4 hours. While for DPSP temperature was 105℃ for the 24hours after completely drying of material then crushed with the help of herb crushing bowl using 20mints. Then grinding time 15mins after crushing material sieve with medium sieve size 12.5cm (Medium sieve size). No further chemical used. Then material placed in airtight bottle for the experimental use.

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2.2 Adsorbate

The stock solution of 1000ppm required concentration of Indigo carmine dye using 100mg/g dye powder. The wavelength of dye is 615:00nm. Its is supplied by sigma Aldrich Pakistan. see Figure 1.

Figure 1: Chemical structure of indigo carmine dye

Synonym: Acid blue 74, indigo-55 disulphonic acid disodium salt indigo carmine Table 1.

2.3 Experimental method

Batch experimental techniques following at room temperature in aqueous lab solution of dye indigo carmine. In individual flask of 50mg/l adsorbent dose were added. Total dye stock solution of 1000ml prepared stock using 100mg/g of indigo carmine dye powder dilute up to 100ml/l desired concentration using 1000ppm. Study performed in acidic pH initial pH was control using 0.1% Hydrochloric acid (Hcl) and 0.1% sodium hydroxide (NaoH). Adsorption best uptake in acidic pH. Determination of absorbance was analyzed using UV-Vis 1800 Spectrophotometer (Shimadzu). Study was performed following experimental parameters. Such as dose mg/g, pH, concentration, contact time speed rpm and desorption of dyes. The selected affecting factors remain same for all adsorbent's ESP, DPSP and PSP. Result were tested and analyzed using isotherms and kinetic graphs using appropriate equation following as. Equation: no1

$$\text{Adsorption} = \frac{CO-Ce}{M} * V$$

Where Co= is initial concentration, Ce= concentration at equilibrium, V= volume, M= mass of adsorbents. Figure 2 prepared stock solution of Indigo carmine dye 50mg/l.

3. Result and Discussion

3.1 Characterization of adsorbents

Surface characterization of selected material ESP, DPSP, PSP for the structural and morphological analyzed using FTIR, and EDS of adsorbent have been used.

3.2 SEM analysis

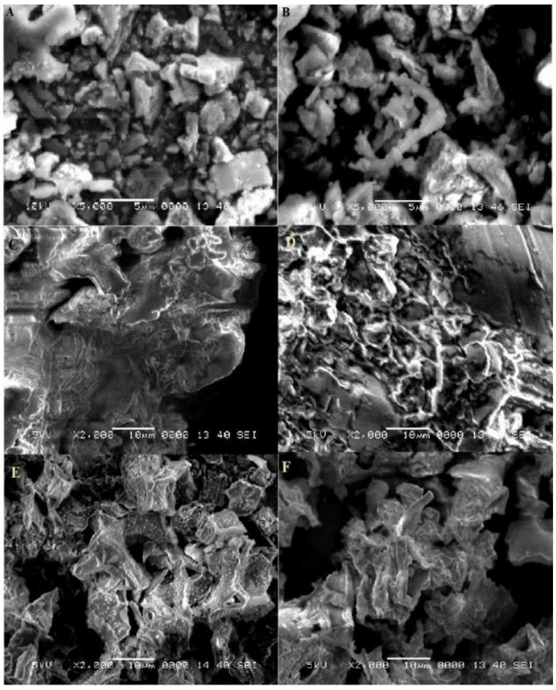


Figure 2: Before and after removal of IC by ESP, DPSP and PSP (A)shows before ESP (b) IC-loaded ESP (C) show before DPSP (D) DPSP- loaded IC (E) beofre PSP (F) PSP-loaded IC

Widely applied practices scanning electron microscope (SEM) for the adsorbents for morphological future and surface characteristics of the sorbent powder. It is very useful for the determination of surface structure shape, appropriate size and porosity supply of adsorbent mate. See figure 3 (A) shows before surface crystal agglomerate structure of ESP. (B) shows after removal of IC on ESP is porous and rough surface. (C) before images of DPSP tell about nature of adsorbent was brittle surface. D after removal of Ic on DPSP Shown adhesive and irregular morphological images. (E) give the structure of before PSP some pores appear on surface and (F) shows PSP overlaps on IC dye. These images give information about topography of surface adsorbents was very helpful in analysis changes appear in after study.

3.3 Fourier transform electron Microscope

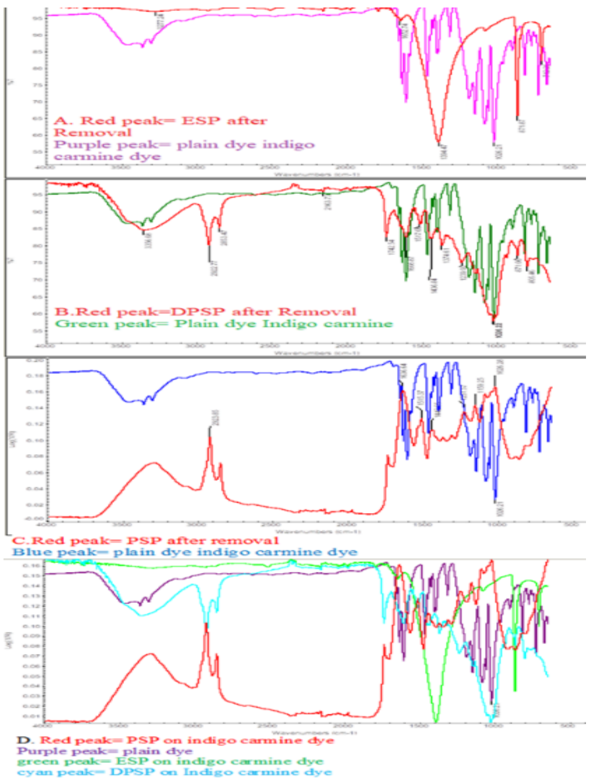


Figure 4: FTIR images of Indigo carmine dye Before and after ESP, DPSP and PSP

FTIR spectra before and after generated for ESP, DPSP and PSP shown in figure 4. (a) purple peak show Indigo carmine dye define range from 712.26cm disubstituted C-H bending, 1026.21 cm S=O stretching sulfoxide, Red peak shows indigo carmine dye by Eggshell from 871.87cm C-H bending,1394.47 sulfate S=O strong stretching, range 3277.24cm strong broad O-H stretching carboxylic acid usually centered on 3000cm (b) Green peak shows indigo carmine dye 805.46cm C-Cl stretching of strong halo compounds, ranges 1036.22cm CO-O-CO stretching strong broad, peak at 1436.84cm due to O-H medium bending, 1608.87cm C=C a, b-unsaturated strong ketone, Red peak shows after removal by date seeds range at 1036.22cm CO-O-CO stretching strong broad, 1239.92cm N=C=S isothiocyanate strong stretching, peak at 1374.61cm medium O-H bending of phenol,17.42.54cm due to C=O stretching strong Y:1770 o-lactone, range 2163.73cm S-C≡N stretching strong thiocyanate, 2853.47cm to 2922.77cm and 3356.08cm due to O-H stretching weak broad intramolecular bonded. (C) Red peak after removal by papaya seeds ranges 1026.28cm due to S=O sulfoxide strong stretching, 11.59.25cm due to strong C-O stretching tertiary alcohol, 1231.57cm owing to C-O stretching strong vinyl ether,1515.37cm C-Br strong stretching,1636.64cm strong C=c mono substituted, range at 2923.85cm peak weak broad intramolecular bonded O-H stretching.

3.4 Energy dispersive X-ray analysis

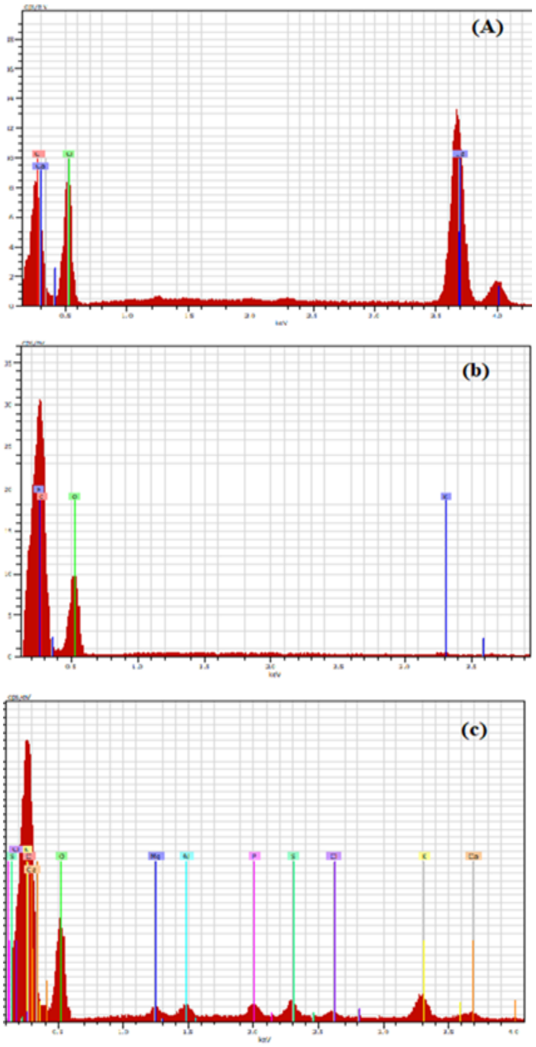


Figure 5: EDS analysis of adsorbents A-ESP, B-DPSP and C- PSP

Power dispersive analysis X-ray Spectroscopy (EDS) fig.5The EDS analysis of eggshell powder. As in illustration eggshell composed of elements percentage volume carbon 18.51%, oxygen 47.84%, calcium 33.65%. EDS show the calcium carbonate was main element of eggshell powder and SEM was helpful to analysis surface morphology images show the spongy structure of material due to presence of these elements. While Date seeds presence chemical elements before Carbon 18.98%, oxygen 80.44%, potassium 0.59%.SEM images show porous structure of dates and absorbance dye on surface of material. Papaya seeds show composed of elements carbon 29.51% Oxygen 63.67% Magnesium 0.50% Aluminum 0.58% phosphorus 0.76% potassium 2.66% calcium 0.70% Sulfur 1.07% chlorine 0.54% after carbon 55% oxygen 43% calcium 0.99% Magnesium 0.31% Aluminum 0.7% SEM images of PSP show the surface mesoporous structure.

3.5 Effect of dose of Adsorbents

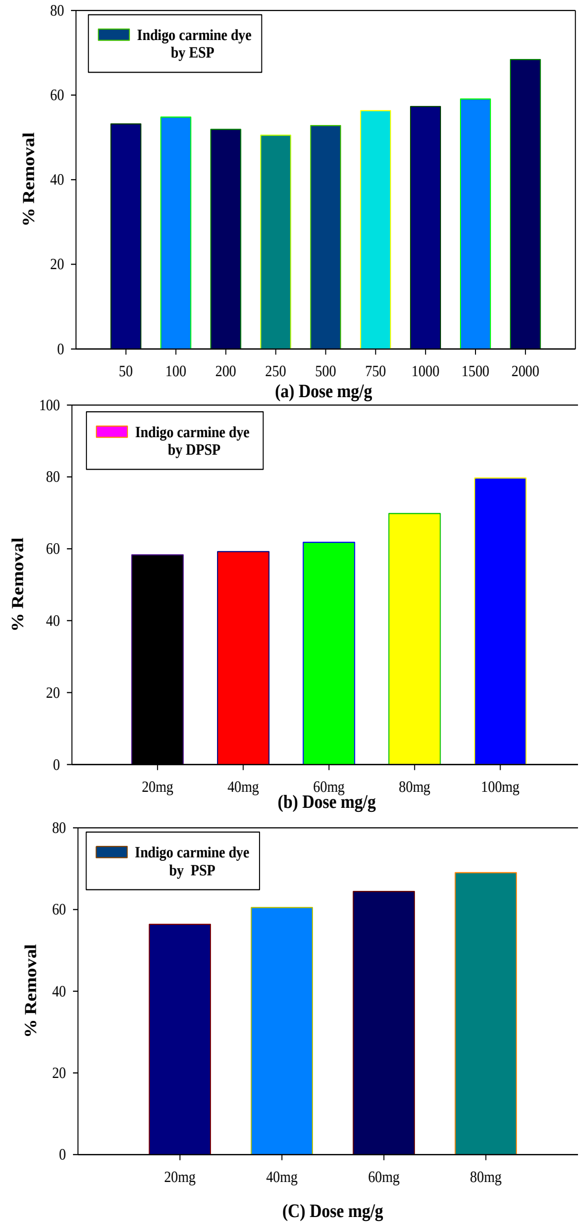


Figure 6: Effect of dose amount indigo carmine dye by (a) ESP, (b) DPSP, (c) PSP

Effect of dose dye against ESP prepared 10th flask in 500ml volume adjust pH 3 using 0.1M HCL with the help of pipet adding drop by drop up to pH3 this procedure applied for all experiments against three adsorbents namely ESP, DPSP and PSP and remining 50ml was placed for initial absorbance. Adding different dose in mg/g 50mg, 100mg, 200mg, 400mg, 500mg, 750mg, 1000mg, 1500mg, 2000mg of ESP for DPSP dose range is 20mg,40mg, 60mg,80mg, and 100mg. While for PSP dose range is 20mg, 40mg. 60mg and 80mg respectively. After preparation of samples flask placed on orbital shaker for different shaking time Using constant contact time for ESP and PSP is 45mints and for DPSP duration is 30mints using time speed at100rpm was constant for all experiments. Time was record using stopwatch. Then samples were filtered with Whatman filter paper 12.5cm. At the end absorbance was checked with the help of Uv-vis spectrophotometer. Fig.6 Results shows when increase dose range dye removal also approved maximum dose for ESP is 100mg and for DPSP and PSP is maximum dose is 80mg was effective range. Indigo carmine dye removal was increase with increase dose mg/l. The availability of active surface site approved with increase in dose and accumulation of adsorbent (Othman et al., 2006; Can et al., 2006).

3.6 Effect of pH of adsorbents

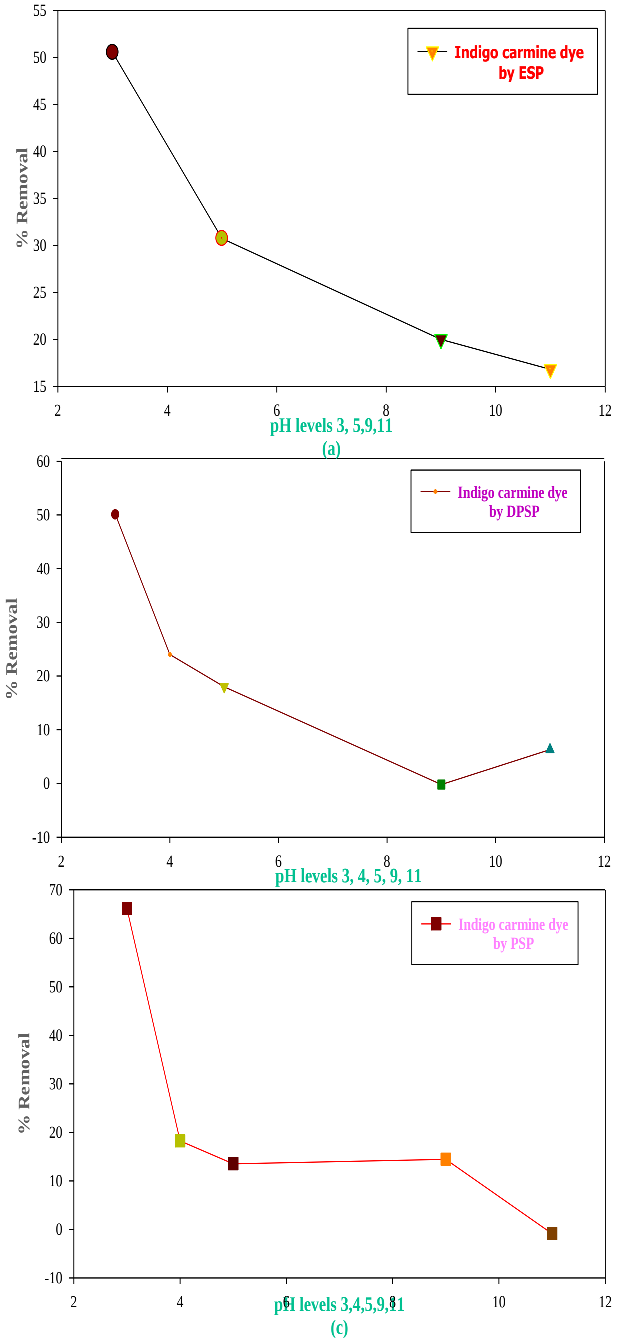


Figure 7: Effect of pH of indigo carmine dye on adsorbents (a) ESP, (b) DPSP, (C)PSP

The pH value of sample was important factor in determination of impurities in substance. The main factor in adsorption process. The role of initial pH is more efficient in process compared to final pH. Adsorption study pH of indigo carmine dye by ESP, DPSP and PSP for all adsorbents effective pH range after experiments were considered is pH3. For uptake pH using 0.1% Hcl and 0.1% NaoH after adjust pH flask was separated individually in 50mg/l. For ESP range is 3,5,9,11.

DPSP and PSP 3,4,5,9,11. After adjusting pH adding above mentioned effective dose amount 100mg of ESP, DPSP and PSP in each flask. Then sample were placed in orbital shaker using effective time for ESP and PSP contact time 45mints for DPSP is 30min. while speed rpm 100 was constant for all experiments. After agitating samples were filtered then absorbance was analyzed with spectrophotometer.Fig.7 shows Results were shown that best optimum condition of pH is 3 for all three adsorbents when increase in pH dye removal was decrease. When decrease pH from 5 then suddenly dye removal was improved. Results proven that initial pH was favorable in removal of dye as compared with final pH (Wagh and Shrivastava, 2015; Basin et al., 2012).

3.7 Effect of initial Concentration of adsorbents

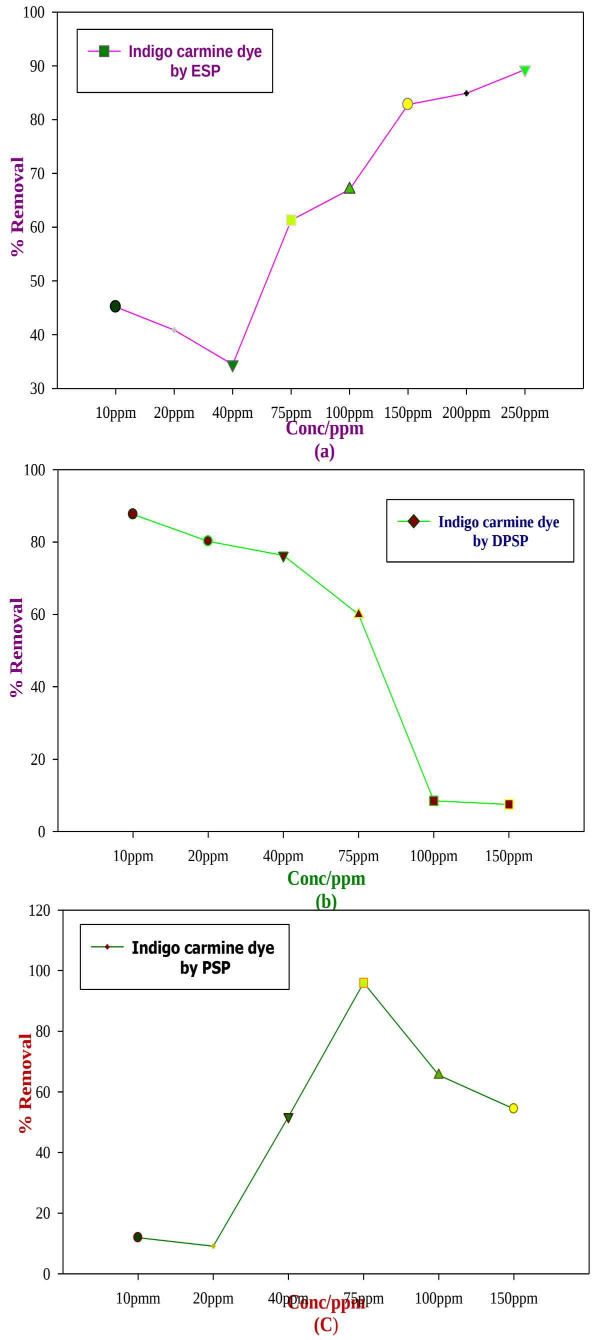


Figure 8: Effect of concentration of Indigo carmine dye by (a) ESP, (b) DPSP, (c) PSP

Concentration study of Indigo carmine dye prepared nine standard solutions range 10ppm, 20ppm, 40ppm,75ppm, 100ppm, 150ppm, 200ppm, 250ppm for ESP, 10ppm, 20ppm, 40ppm, 75ppm, 100ppm for DPSP and 10ppm, 20ppm, 40ppm, 75ppm, 100ppm, 150ppm for PSP. using 0.1% Hcl adjusting pH in each standard separately for required concentration. Then sample separated adding different adsorbents effective dose of each adsorbent. Adding dose amount of ESP is 200mg/g, and DPSP and PSP is 100mg/g respectively. While contact time for ESP for concentration range is 45mints and for DPSP and PSP was 30mints respectively using Time speed at 100rpm for all adsorbents respectively. Figure 8 (a) shows when increase dye concentration adsorption site was also improved due decreased in concentration of dye (b and c) graph shows concentration of dye was slightly decreased from 75ppm due close the adsorption site and increase concentration of dye on the surface. Results were proven that best ideal concentration of sample between 50 to 75ppm in removal percentage was recorded 60% to 80% in each sample. When initial concentration of dye was increase from 10ppm to 50ppm. This proven that the indigo carmine removal by adsorbent is dependent on the concentration (Mane and Bhusari, 2012).

3.8 Effect of contact time of adsorbent's

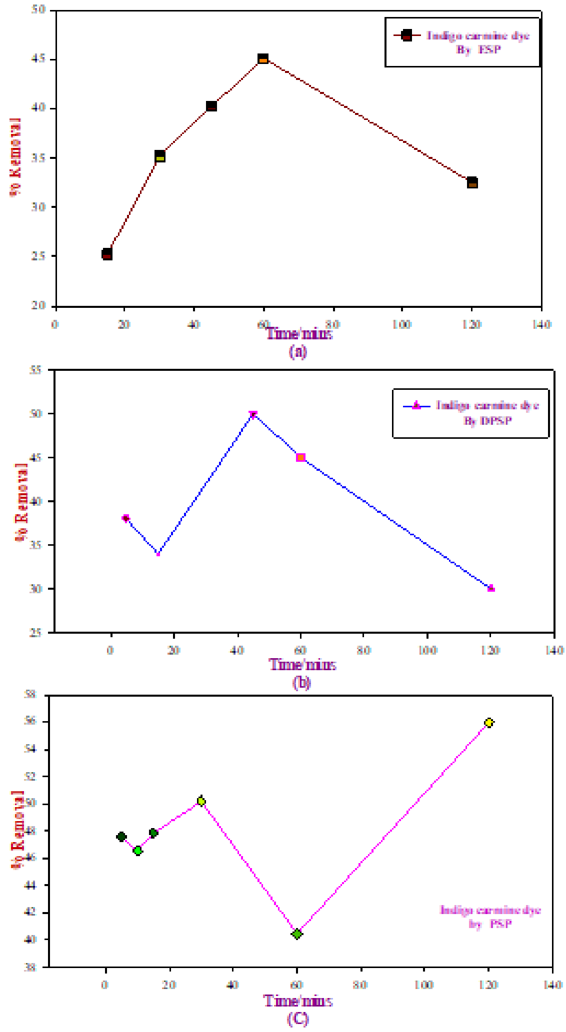
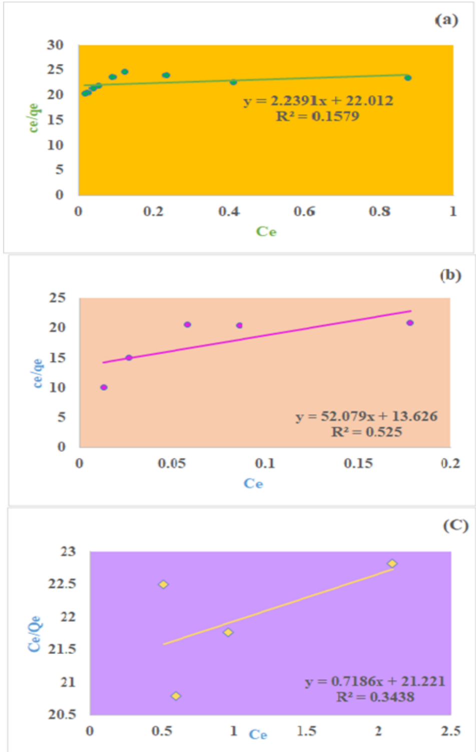


Figure 9: Effect of Contact time indigo carmine dye by (a) ESP (b) DPSP (c) PSP

Effect of Shaking time of sample on adsorbent's ESP, DPSP and PSP prepared fifth flask 15,30, 45, 60,120 of ESP 5, 15, 45,60, 120 for DPSP and 5,10,15,30,60,120 of PSP in volume 50mg/l. using 0.1% Hcl at speed 100rpm each sample was placed on orbital shaker for different time. using dose amount 200mg/g of ESP and 100mg of DPSP and PSP. At the end of time sample filtered with Whatman filter paper 12.5cm. Then absorbances of sample was checked with Uv-Vis spectrophotometer. Results were proved that best contact time for IC dye removal by ESP and DPSP is between 40 to 45 mints and 30mints on PSP respectively. While contact time of ESP and DPSP graphs shown when increase time dye removal was slightly decrease. But PSP in fig.9 shows when increase time dye removal was also increase (Nakamura et al., 2005; Ramesh and Sreenivasa, 2015).

3.9 Adsorption isotherms

Measurement between adsorbent and liquid phase distribution of dye position at the equilibrium state can be generally expressed in equation Freundlich and langmuir isotherms plots. Model were Applied for the explanation towards adsorption of Indigo carmine dye from aqueous solution by Eggshell, date palm seeds and papaya seeds. Langmuir adsorption isotherms is the highly application of adsorption isotherms that have been applied for adsorption to solution of aqueous solution. This isotherm built on a hypothesis for individual layer adsorption on an adsorbent with equivalent structure. The linear equation of Langmuir isotherm is as follows Where Ce is the concentration of dye in solution at equilibrium (ml/l), Qe adsorption capacity of per gram (mg/g), q_m is the theoretical saturated adsorption capacity(mg/g), langmuir plotted against Ce/Qe vs Ce Concentration and adsorption capacity at equilibrium. While Freundlich isotherms is an experimental calculation and relation the surface adsorbent the linear equation of Freundlich isotherms calculated from adsorption capacity and concentration at equilibrium simplify calculated Log Ce from Ce values and Log Qe from Qe values Freundlich get by drawing plot against logQe with logC_e values.



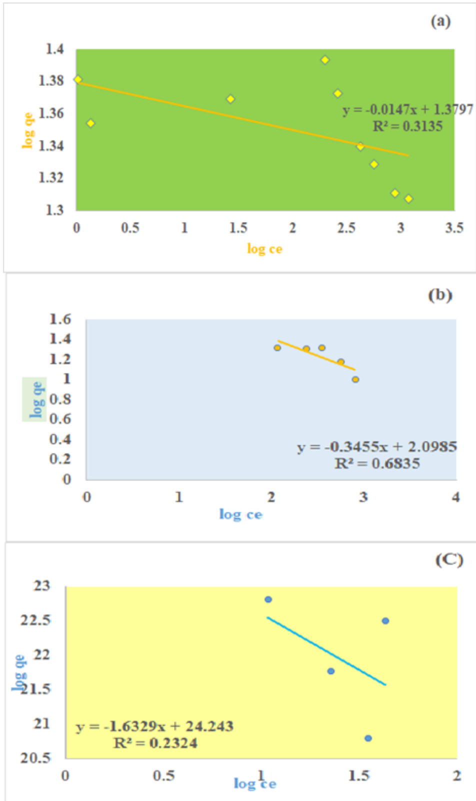


Figure 11: Freundlich Adsorption isotherm of Indigo carmine dye a) ESP b)DPSP c) PSP

Adsorption is proportional to the fraction of the adsorbent surface that is covered. The langmuir equation can be written in the following linear form:

Equation.2. $\frac{C_e}{Q_e} = \frac{1}{Q_m K_e} + \frac{C_e}{Q_m}$

Where Ce is concentration of adsorbate at equilibrium (mg/g). Kl is langmuir constant related to adsorption capacity (mg/g), which can be correlated with the variation of the suitable area and porosity of the adsorbent which implies that large surface area and pore volume will result in higher adsorption capacity. This isotherm built on a hypothesis for individual layer adsorption on an adsorbent with equivalent structure. The lined calculation of Langmuir line is as follows Wherever Ce is the absorption of dye in solution at equilibrium (ml/l), Qe adsorption capacity of per gram (mg/g), qm is the theoretical saturated adsorption capacity(mg/g), langmuir plotted against Ce/Qe vs Ce Concentration and adsorption capacity at equilibrium. Freundlich isotherms is applicable to adsorption process that occur on heterogenous surfaces. This isotherm gives an expression which defines the surface heterogeneity and the exponential distribution of active sites and their energies.

The linear form of Freundlich isotherm is as follow equation:3

$$\text{Log } Q_e = \text{Log } K_f + \frac{1}{n} 1 \text{ Log } C_e$$

Where Kf is adsorption capacity (L/mg) and 1/n is adsorption intensity, it is also indicating that relative distribution of the energy and the heterogeneity of the adsorbate sites.Fig.10 shows isotherms plotted against Ce/qe vs Ce while Figure 11 shows good relationship between adsorbent and adsorbate of Freundlich isotherms Graph is plotted against =log qe/log Ce Respectively.

3.9 Adsorption Kinetics at equilibrium

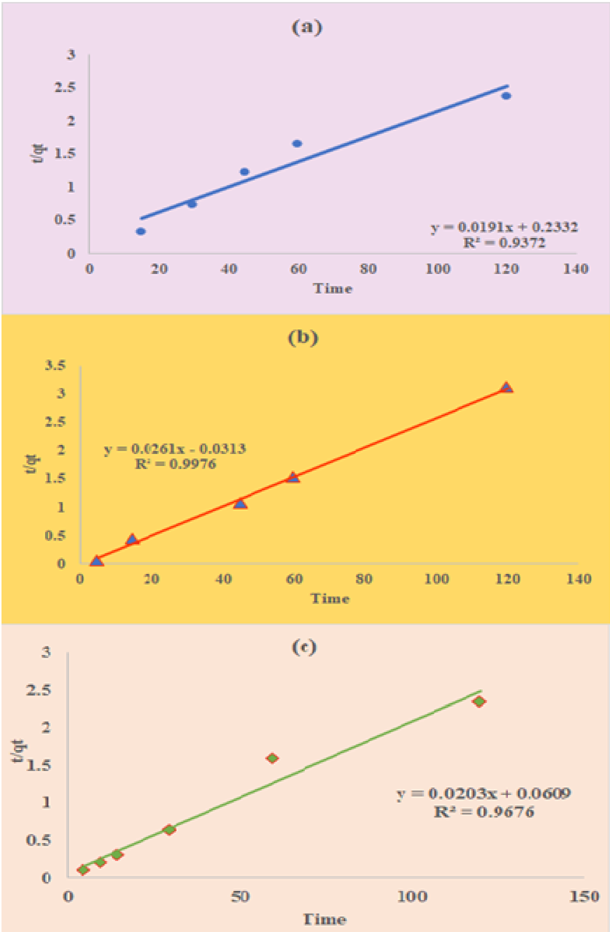


Figure 12: Pseudo second order Models a) show IC on ESP; b) Shows IC on DPSP; c) Shows IC on PSP

Pseudo First order kinetic equation was calculated to get insight adsorption process of indigo carmine onto adsorbents ESP, DPSP, PSP. Linearly data does not fit for pseudo-first order kinetics rates. Method of Adsorption process was tested at pseudo-second order kinetics equation of indigo carmine dye onto ESP, DPSP, PSP through transfer of the charge then reaction must be in second order plots. Unidentified t/qt plotted versus t, it establishes straight line, that may define the adsorption follows pseudo- second order kinetics. This plot is established upon the idea the rate controlling step may be chemisorption which include valance force due to electron splitting or exchange of electron among the adsorbent and adsorbate.

pseudo-second order model equation following formula. Equation:4

$$\frac{1}{(q_{e-}q_t)} = 1\frac{1}{q_e} + kt$$

This plot is established upon the idea the rate controlling step may be chemisorption which include valance force due to electron splitting or exchange of electron among the adsorbent and adsorbate. Figure 12 shows between calculated ce and qe adsorption might be follow pseudo second order kinetics in nature at pH3 and calculated R value is R^2=0.9372, 〖R〗^2=0.9976 〖,R〗^2=0.9676 reasonably indicate good relationship and calculated value closer to the experimental values that might be proven that removal of indigo carmine largely fitted in pseudo second order plots respectively.

3.11 Desorption studies of dye

Table 2: Desorption study of indigo carmine-loaded Eggshell (ES), Date palm seeds (DPS) and papaya seeds (PS)

Adsorbents	Initial Abs	Before Abs	Wavelength nm	After Abs	Removal %
ESP	3.355	1.373	613.00	0.038	28
DPS	2.380	1.071	613.00	0.148	88
PSP	2.380	1.071	613.00	0.148	72

This processor may involve appropriate selection of eluent. The way of adsorption which usefully diverges on the nature of bio-waste material. Eluent to the adsorbent preferred is nonpolluting and commercial effective or harmless should be preferred carefully. 0.1M Hcl was treated for this this purpose. Measure the quantity of adsorb material ESP, DPSP and PSP powder against removal of Indigo carmine dye maximum removal on adsorb dose following as 500mg/g of ESP, 100mg/g of DPSP and 80mg/g of PSP placed in oven at105°C for 24 hours. After complete drying then sample placed individually for desorption study of dye. Prepared three flasks with 50mg/l concentration with using 0.1M HCL solution. Get amount of dried material mentioned in above added in each flask separately. The sample placed on orbital shaker for using effective time at speed 100rpm. study conduct at room temperature. At the end of duration samples were filtered and change in colour observed solution recovery and absorbance was checked using Uv-vis spectrophotometer. Solution percentage calcuted using following equation.5

$$\text{Desorption efficiency} = \frac{\text{Amount of dye desorb}}{\text{Amount of dye adsorb}} \times 100$$

See Table 2. Shows where initial absorbance is dye actual absorbance at 50mg/l and before absorbance is calcuted from adsorption amount show amount of adsorb mg/g powder removal absorbance and after absorbance show amount of desorb material absorbance at the last % removal calculate using above equation. It shows desorption efficiency of indigo carmine dye onto eggshell powder, date palm seeds and papaya seeds powder 0.1M HCL eluent can effectively desorb (28% 88% and 72%) of IC from sorbent material.

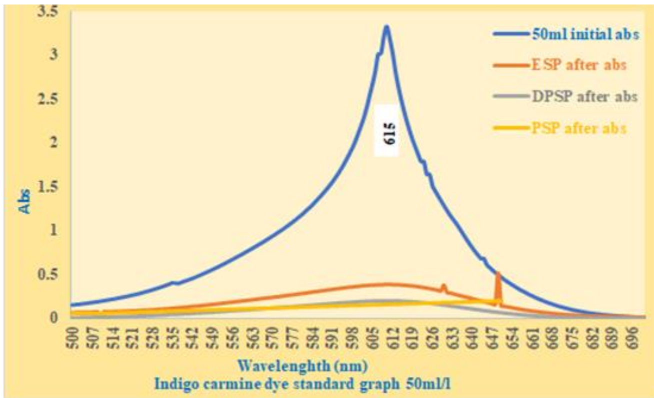


Figure 13: Standard graph of indigo carmine dye Before and after removal

4. Conclusion

Adsorption isotherms of were evaluated on Indigo carmine dye on ESP DPSP and PSP in aqueous solution. To evaluate and understand the nature of adsorbent and adsorbate. The bio-waste material which easily and abundantly available was using as a good adsorbent for dyes removal. study carried out following batch techniques. Isotherm's equation langmuir follows monolayer adsorption all the adsorbed molecules are in contact with the surface layer of the adsorbent. Freundlich follow multilayer adsorption the adsorption space accommodates more than one layer of molecules and not all adsorbed molecules are in contact with the surface layer of the adsorbent. Kinetic study second order shows adsorption happen in two sites, concentration of Dyes and adsorbents. kinetic model good relationship between theoretical and experimental values. Lastly results show that adsorbent material which is effective Against Indigo carmine dye in Acidic pH3 and environmentally friendly against the removal of dyes from wastewater in aqueous solution.

SUGGESTIONS

Future Recommendation

Langmuir and Freundlich isotherms help to suggest limited adsorption only monolayer and multilayer adsorptions.

Whereas D-R isotherm expresses adsorption mechanism with energy distribution onto heterogeneous surfaces. Therefore, it is recommended that D-R isotherms must be studied in future so that heat energy distribution of adsorbent and adsorbate should be studied.

Temkin isotherm express surface porosity of adsorbent if increase porosity the removal was also increase.

The adsorbent should characterize by using particle size analyzer (PSA) and other factors such as temperature should be studied.

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