THE MILLET HUSK USED AS EFFECTIVE NATURAL ADSORBENT FOR REMOVAL OF COD AND WATER POLLUTANTS.

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ABSTRACT: *Rapid industrialization has led to the discharge of waste effluent in environment and this has been great concern because of its health effect on human beings .The conventional methods of treatment of this waste water includes chemical precipitations, chemical oxidation, ion exchange, membrane filtration, electro dialysis, etc. These methods are costly, energy intensive and often associated with generation of toxic byproducts. The purpose of this study is to investigate the utilization of millet husk (which is totally unused after crop product is removed) as low cost adsorbent for treatment of waste water from dye intermediate product. Acid treated carbonized millet husk and powder particles were use in this experiments at different pH value(4 pH, 5pH, 6pH) and different adsorbent dose (1 gm , 2 gm , 3 gm) and different contact time (20 min , 40 min , 60 min) . Effluent parameters were analyzed in the laboratory according to standard methods of American Public Health Association (APHA). According to analysis, optimum results were obtained at 5.5 pH, 3 gm dosage and 60 minutes contact time. That removes COD (93.93%) which is most affected problem of recent scenario. It also remove sulphate (90.74%) , nitrate (84.74%) , phenol (83.33%) , nitrite (76.08%) , turbidity (67.30%) , ammonia (59.73%) , TS (59.18%) , TSS (60.00%) , TDS (58.62%) .The main advantage of this method is that usable and treated adsorbent is reused after washing with distilled water as 50 percent capacity. So it is discovered that Millet husk can be used as cost affective adsorbent and rapid process for treatment of Dye Intermediate waste water effluent successfully. According to that Adsorption Isotherms studies that shown organic pollutants follow Langmuir and BET model adsorption pattern and inorganic pollutants follow Freundlich and Temkin adsorption patterns.*

Key Words: *Adsorption, Low Cost natural adsorbent (millet husk), COD, % Removal, Adsorption Isotherms*

1.0 Introduction

1.1 GENERAL INTRODUCTION OF WATER:

Water is a chemical substance that is essential to all known forms of life. The (UNEP) United Nations Environment Program estimate there is 1.4 billion cubic kilometres of water available on earth and the water existing in many forms. Water is very important substance. It exists in organisms in large quantity. Importance of water is that the water is used everywhere for different purposes. Water is important for drinking,cooking, etc. It is also important for industries for manufacturing process. Water is also important for aquatic life because the aquatic organisms lie in water. The plankton are formed their food chain.[1]

1.2 GENERAL INTRODUCTION:

Besides the rapid growth of the world population, industrialization, unplanned urbanization, agricultural activities, as well as the excessive use of chemicals have contributed to environmental pollution. Inorganic and organic wastes produced by human activities have resulted in high volumes of contaminated water which threatens human health and other living organisms. Discharge of pollution into water bodies not only can aesthetically cause issues, but also it is harmful to biological organisms and ecology.[2] Considering the fact that removal of these toxic pollutions from wastewater is highly essential for the well-being of humans, various technologies have been used for removal of such metal ions and dyes such as reduction, precipitation, adsorption, oxidation, and ion exchange. However, adsorption process is known to be the most suitable method because of its high efficiency and economic consideration [3][4]. Recently, numerous approaches have been studied for the development of cheaper and effective adsorbent.

1.3 PRESENT SENARIO OF WATER POLLUTION IN INDIA:

The major issues concerning 'Water Resources' in India can be broadly classified into issues of water quantity (availability) and quality, for use in the domestic, industrial and service sectors. A look at the present scenario, with respect to these two aspects, gives an overview of the existing problems and provides a platform for improvement in terms of action at the policy and consumer level.

The per capita availability of freshwater in India is a little over 2000 cubic meters. However, there is a large spatial and temporal variation in the availability of freshwater. While some areas in Rajasthan get just around 100 millimetres of rainfall annually, some parts of Meghalaya get over 11,000 millimetres. This reflects on the per capita availability. For example, it is around 650 cubic metres in the western region, supplied by the rivers of Kutch and Saurastra and 18,500 cubic metres in the East, supplied by the Brahmaputra. Temporal variations are with respect to the number of rainy days in a year. In India, most of the rainfall is received during the two major monsoons, South-West and North-East. In fact, the other seasons are relatively dry.[5][6]

It is estimated that 85 % of urban population has access to drinking water. However, only a small percentage of the people have access to safe drinking water. The main source of drinking water is the reservoirs that are located far away from the urban centres. To cite an example, Bangalore draws water from River Cauvery, which is around 100 Kms away from the city. Over the last couple of decades, there has been large exploitation of ground water for domestic purposes. Roads and pavements are made of concrete in most of the cities and thus, have prevented the possible recharging of groundwater aquifers during rains, leading to high run-offs and drastic decrease in the groundwater table.

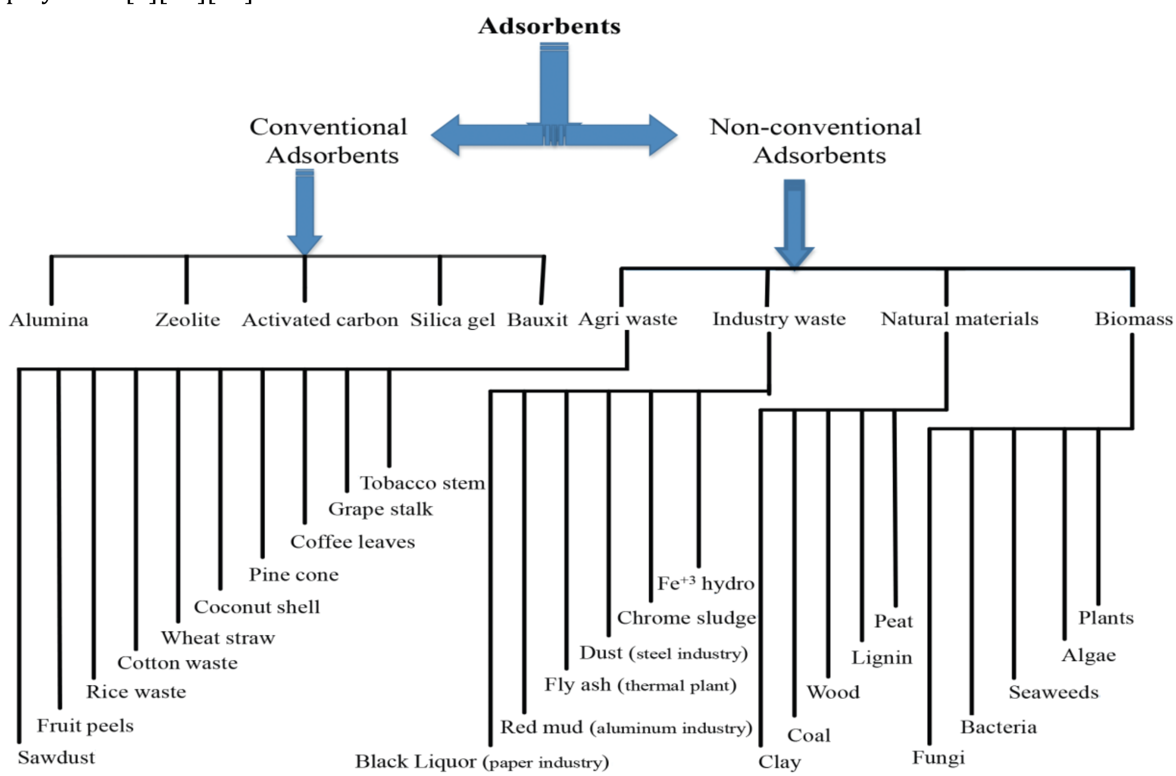
1.4 Low Cost Adsorbents:

The removal of Water Pollutants by using low cost adsorbent is found to be more encouraging in extended terms as there are several materials existing locally and profusely such as natural materials, agricultural wastes or industrial by-products which can be utilized as low-cost adsorbents. To be commercially viable, an adsorbent should have high selectivity to facilitate quick separations, favourable transport and kinetic characteristics, thermal and chemical stability, mechanical strength, resistance to fouling, regeneration capacity and low solubility in the liquid in contact.[7][8]

Adsorption process has several advantages over the conventional methods of heavy metal removal.

Some of the gains of adsorption process are: Economical,Metal selectivity, Regenerative, Absence of toxic sludge generation, Metal recovery, Effective.

The adsorbents mostly used are agricultural waste, industrial byproducts, natural materials or modified biopolymers. [9][10][11]



2.0 SAMPLING:

- Waste water sample was collected from '***DYEINTERMEDIATE INDUSTRY'*** at Ankleshwar.
- That having components as follow:
- 4- Nitro-2-Amino phenol

- ParanitroAzobenzen
- 4-Aminodiphenayl amino-2-sulphonic acid.

3.0 RESEARCH METHODOLOGY*:*

3.1 Analysed Parameters Before Treatment:

In given table: 1 all parameters which are present in waste water sample are analysed before adsorbent treatment according to *American Public Health Association (APHA*) (23rd addition) with standard methods to check Initial concentration of pollutants. These data is helpful to calculate their removal percentages and concluded which isotherms models follow in the particular parameters.[12][13]

TABLE : 1Methods of Analysis of water quality parameters.

SR.NO	PARAMETERS	METHODES	INSTUMENTS
1	pH	-	pH meter
2	Turbidity	-	Turbidity meter
3	Thermometer	-	Thermometer
4	TS	Gravimetric	Oven
5	TSS	Gravimetric	Oven
6	TDS	Gravimetric	Oven
7	Alkalinity	Titrimetric	-
8	COD	Open Reflux	COD Digester
9	Ammonia	Spectrophotometric	Spectrophotometer
10	Chloride	Argentometric	-
11	Nitrate	Spectrophotometric	Spectrophotometer
12	Nitrate	Spectrophotometric	Spectrophotometer
13	Sulphate	Spectrophotometric	Spectrophotometer
14	Phenol	Spectrophotometric	Spectrophotometer

3.2 Adsorbent Preparation:

The millet husk used for the experiment was collected from a local agricultural land. The samples were collected in a polyethylene bag and transported to the laboratory for further treatment. The millet husk was first handpicked to remove dirty particles and washed with tap water several times to remove adhering particles. It was grinded using pestle and mortar and sieved to obtain 300 and 500 μm sizes using a sieve mesh. It was later washed twice with deionised water, drained and oven dried for 12 hours at 90°C. The dried millet husk was cleaned according to the method used by Gilbert (2012). The millet husk was soaked in 1M HNO_3 for 24 hours at room temperature. This was done to remove soluble organic compounds in the husk. The acid soaked millet husk was filtered and washed several times with tap water then with deionised water until a fairly constant pH was obtained .The acid cleaned husk was oven dried at 90°C for 24 hours.

3.3 Various Experimental Conditions:-

A.)Effect of Adsorbent Dosage:

The effect of adsorbents on the removal of pollution from aqueous solutions was studied by varying the amount of adsorbents from ***1gm ,2gm, 3gm.***This was done by adding following amount in 100 ml of industrial sample .The mixtures were corked and agitated at 200rpm for 60mins and filtered. The filtrates were analysed for various parameters and treatment. [14][15]

B.) Effect of pH:

The effect of pH on the removal of pollutants from aqueous solutions was studied by varying the pH value ***4 pH , 5 pH , 6 pH*** .This was done by adjusting following value in 100 ml of industrial sample . The mixtures were corked and agitated at 200rpm for 60min and filtered. The filtrates were analysed for various parameters and treatment.[16][17]

C.)Effect of Contact Time:

The effect of different contact time on the removal of pollutants from aqueous solutions was studied by varying the contact period of times ***20 minutes , 40 minutes , 60 minutes*** .This was done by giving following contact time in 100 ml of sample . The mixtures were corked and agitated at 200rpm for different pH and filtered. The filtrates were analysed for various parameters and treatment.[18][19]

4.0 RESULT

4.1 Removal Percentage of waste water pollutants:-

According to study optimum results occurrence on 5.5 pH, 3 gm dosage and 60 minutes.

TABLE: 2**Analyzed Parameters and their Removal %that Fulfill 'IS' Discharge Standers intoSurface Water.**

Sr. No	***Parameters***	***Initial concentration (ppm) (before treatment)***	***Final concentration (ppm) (after treatment)***	***IS effluent discharge permissible limits (ppm)(IS:10500,1992)***	***% Removal***
1	Colour	Brownish grey	Pale yellow	-	-
2	Odour	highly objectionable	less objectionable	Unobjectionable	-
3	Turbidity	520 NTU	17 NTU	-	67.30%
4	Alkalinity	560	400	200	28.57%
5	COD	3300	200	250	93.93%
6	Nitrate	295	45	45	84.74%
7	Sulphate	810	75	200	90.74%
8	Ammonia	745	30	50	59.73%
9	Nitrite	172.5	41.25	45	76.08%
10	Phenol	270	45	1	83.33%
11	TS	490	200	-	59.18%
12	TSS	200	80	100	60.0%
13	TDS	290	120	500	58.62%

- After treatment according to standardmethod all parameters are now ready to release in surface water bodies except Alkalinity , Phenol, parameters need further treatment.

4.2 REMOVAL OF COLOUR

FIGURE 1: Removal of colour at 5 pH; 40 min ; 3 gm dosage FIGURE 2: Removal of colour at 5 pH; 60 min ; 3 gm dosage

4.3 Removal % of parameters:

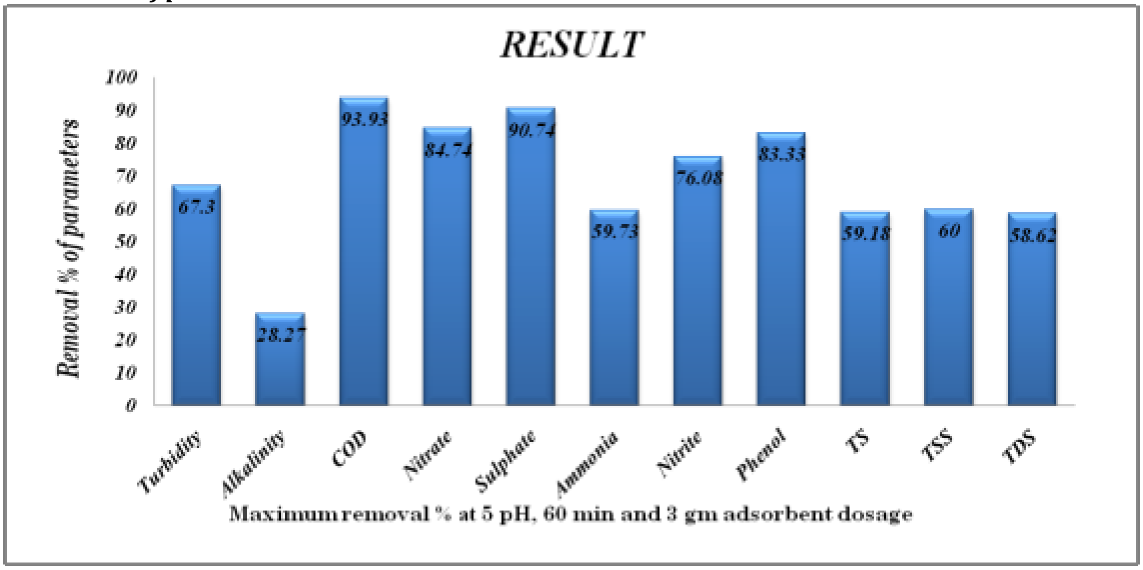


FIGURE 3:Maximum removal % at 5 pH, 60 min and 3 gm adsorbent dosage

5.0 Batch isotherms studies

Isotherm experiments were conducted to investigate the relationship between the solid phase concentration of an adsorbate and the solution phase concentration of the adsorbate at an equilibrium condition. The removal percentage (R %) of parameters was calculated for each run by following equation:

R (%) = [(Ci - Ce) / Ci] x 100………………………………..(1)

Where, Ci and Ce are the initial and final concentration of parameters (mg/l) in the solution. The adsorption capacity of the adsorbent for each concentration of parameters at equilibrium were calculated using the equation (2),

qe (mg/g) =[(Ci - Ce)/ M] * V…………………………(2)

Where, Ci and Ceare the initial and final concentration of chromium (mg/l) in the test solution respectively , Vis the volume of solution and M is the mass of Adsorbent (gm),(Bansal et.al, 2008).[20][21]

6.1*Adsorption Isotherms*

In the present study, various adsorption isotherm models have been used to study the adsorption capacity and equilibrium coefficients for adsorption. Four commonly used isotherms (viz. Langmuir, BET, and Freundlich and Temkin isotherm) were studied.

- ***The Langmuir adsorption isotherm*** has the simplest form and shows reasonable agreements with a large number of experimental isotherms. Therefore, the Langmuir adsorption model is probably the most useful one among all isotherms describing adsorption, and often serves as a basis for more detailed developments. (Noll K. E et al, 1992).

  Langmuir's equation is a commonly used for isotherm modeling, which involves two parameter expressed as follows. Langmuir isotherm equation is derived from simple mass kinetics, assuming chemisorptions. This model is based on two assumptions that the forces of interaction between sorbet molecules are negligible and once a molecule occupies a site no further sorption takes place. Also these equations can reduce to Henry's law at lower initial concentrations.[22] Alternatively at higher concentrations, it predicts a monolayer sorption capacity (Janos et al, 2003).[23] It assumes that the uptake of various impurities occurs on a homogenous surface by monolayer adsorption without any interaction between adsorbed ions The linear form of the Langmuir adsorption isotherm is represented as ;

  Ce/qe = [1/Q^0b+1/Q^0*Ce]………………….(3)

Where;

Ce is the equilibrium concentration of adsorbate(mg/l) ,

Andqe is the amount of every parameters adsorbed per gram at equilibrium (mg/g),

Q^0 (mg/g) and b (L/mg) are Langmuir constants related to adsorption capacity and rate of adsorption, respectively. The values of Q^0, and b were calculated from the slop and intercept of the Langmuir plot of Ce versus Ce/qe.

- ***Freundlich equation*** assumes that the uptake of impurities occurs on a heterogeneous surface by multilayer adsorption. The linearized Freundlich model isotherm was applied for the adsorption of impurities and is expressed as:

Log 10 q = log 10 (K_f) + (l/n) log10 (Ce)…………(4)

Where;

qe, is the amount of impurities adsorbed at equilibrium (mg/g),

andCe is the equilibrium concentration of different parameters in solution (mg/l).

Kf and n are the constants incorporating all factors affecting the adsorption process (adsorption capacity and intensity). The values of Kf and n were calculated from the intercept and slope of the Freundlichplot: log C, versus log q,

- ***Temkin isotherm*** model is given by the following equation:

X=a+bIn C………………. (5)

Where;

C = Concentration of adsorbate in solution at equilibrium (mg/L).

X-Amount of metal adsorbed per unit weight of adsorbent (mg/gm)

aand b are constants related to adsorption capacity and intensity of adsorption and related to the intercept and slope of the plots of In C against X. (Abdel-Ghani N. T. al., 2008)

- ***BET isotherm*** was developed by Brunauer, Emmett and Teller as an extension of Langmuir isotherm, which assumes that first layer of molecules adhere to the surface with energy comparable to heat of adsorption for monolayer sorption and subsequent layers have equal energies. Equation in its linearized form expressed as:

Cf/(Cs- Cf)q = 1/Bq_{max} - (B-1/Bq_{max})(Cf/Cs)……………….(6)

Where;

Cs is the saturation concentration (solubility limit) (mg/L) of the solute,

Cf is solute equilibrium concentration.

B and qmax are two constants and can be evaluated from the slope and intercept. (Sharma M. et al, 2009).[24][25][26]

6.2 comparisons of isotherms:-

- In this Adsorbent Isotherms studies that shown different parameters follow different adsorption model Pattern according to its adsorption capacity .here in table (3) that shown different parameters which follow different models.

TABLE 3: Different Parameters Follow Different AdsorptionModel Pattern

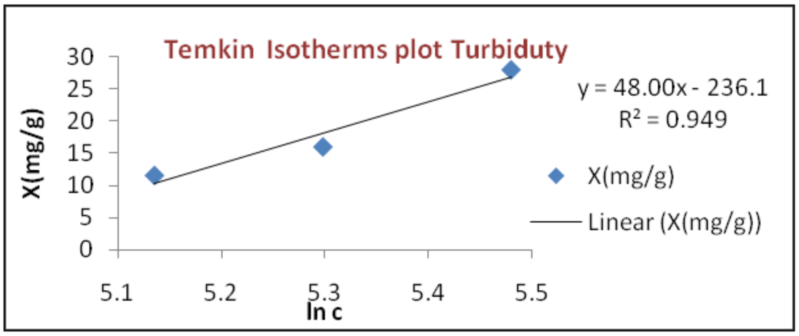
Sr.No	*Parameters*	*Followed Isotherms*
1	Turbidity	Temkin
2	Nitrate	Temkin and Freundlich
3	Sulphate	Temkin and Freundlich
4	Ammonia	Temkin and Freundlich
5	Nitrite	Temkin and Freundlich
6	Phenol	Temkin
7	Alkalinity	Langmuir
8	COD	Langmuir and BET
9	TDS	Langmuir and BET
10	TSS	Langmuir and BET

- Where;
- Langmuir isotherms, homogenous surface by monolayer adsorption.
- Temkin and Freundlich isotherms, heterogeneous surface by multilayer adsorption.
- BET isotherms, monolayer sorption and subsequent layers.[27][28][29][30]
- Therefore Turbidity, Nitrate, Sulphate, Ammonia, Nitrite, Phenol adsorption is followed in a manner of multilayer at heterogeneous surface.

- Alkalinity adsorption is followed in a monolayer adsorption.
- In case of COD, TDS and TSS first monolayer adsorption takes place and then, subsequent multilayer adsorption is followed.

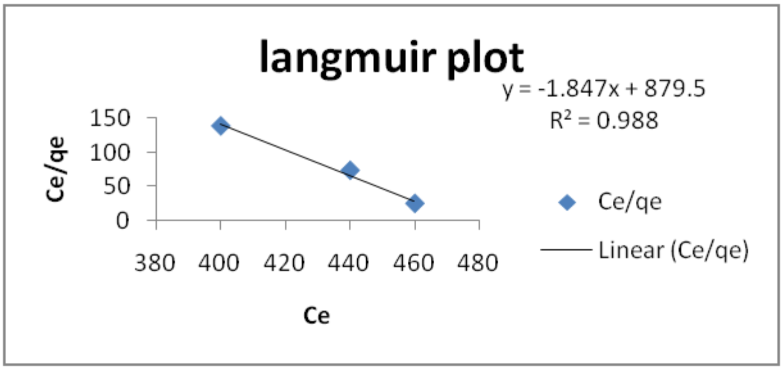
6.3 Followed Graphs of Different Parameters:-

1) Turbidity: Temkin



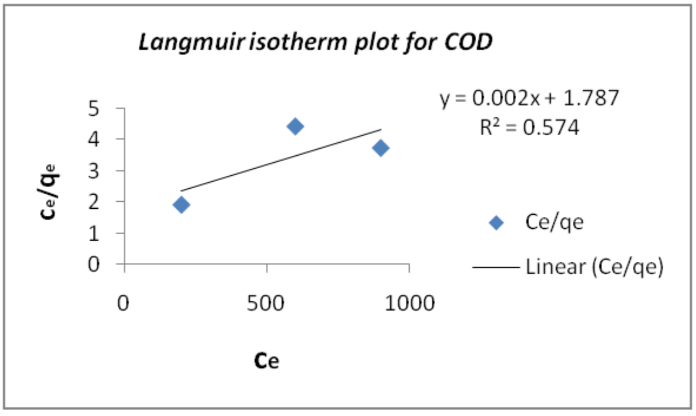
Sr.No	*Parameters*	*a(mg/g)*	*b(L/mg)*	*R^2*
1	Turbidity	48	-236.1	0.0949

2) Alkalinity: Langmuir

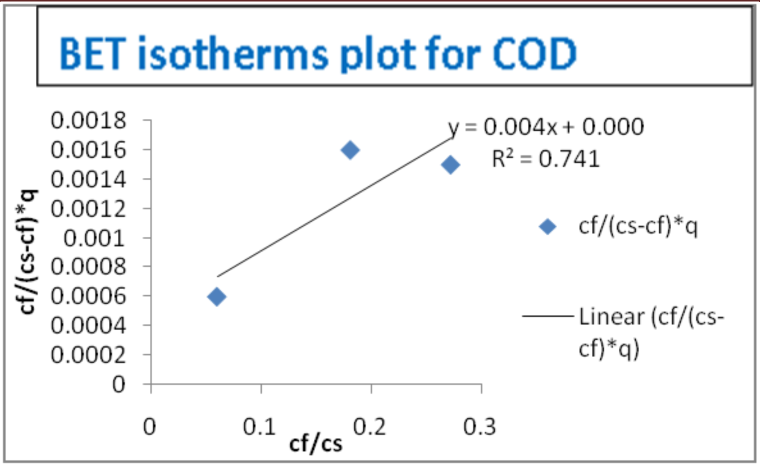


Sr.No	*Parameters*	*Q^0(mg/g)*	*b(L/mg)*	*R^2*
1	*Alkalinity*	*-0.541*	*-0.002*	*0.988*

3) COD : Langmuir and BET

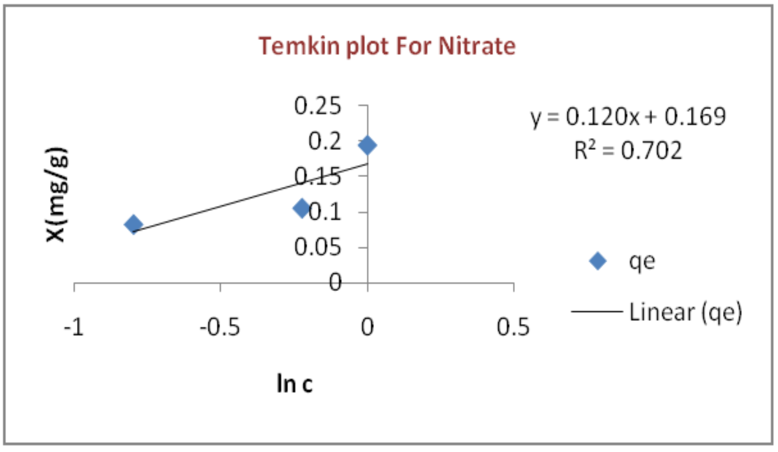


Sr.No	*Parameters*	*Q^0(mg/g)*	*b(L/mg)*	*R^2*
1	COD	500	0.001	0.574



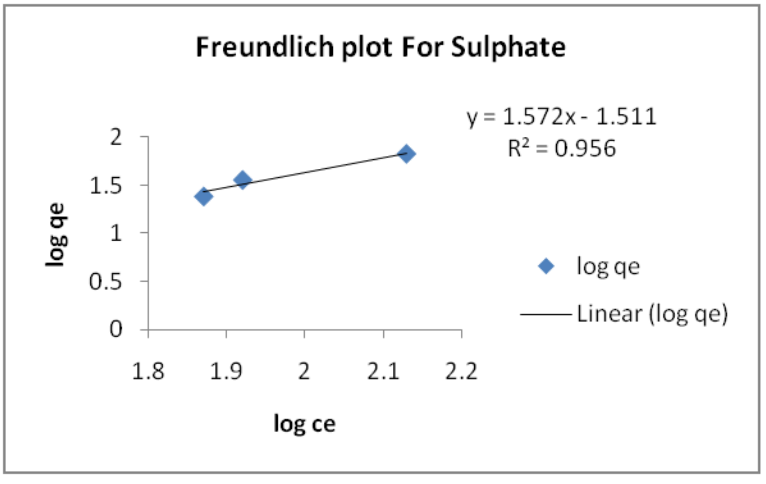
Sr.No	*Parameters*	*q_{max} (mg/g)*	*b(L/mg)*	*R^2*
1	*COD*	*250*	*1*	*0.741*

4) Nitrate:Temkin

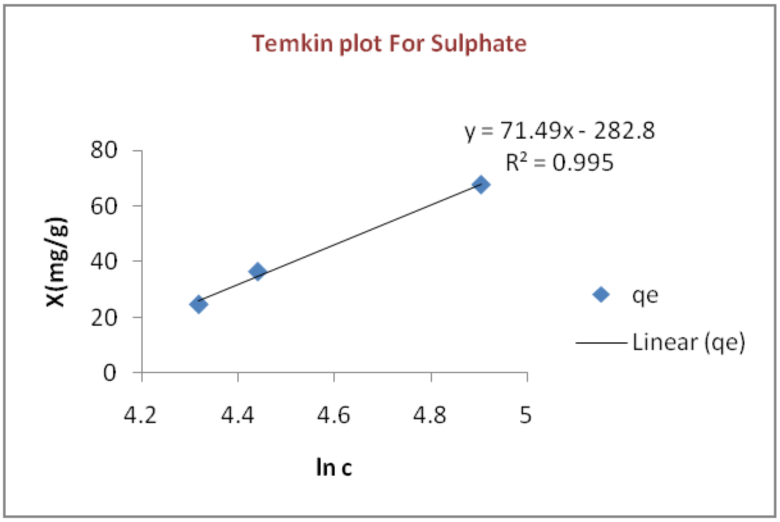


Sr.No	*parameters*	*a(mg/g)*	*b(L/mg)*	*R^2*
1	*Nitrate*	*0.12*	*0.169*	*0.702*

5) Sulphate: Freundlich and Temkin

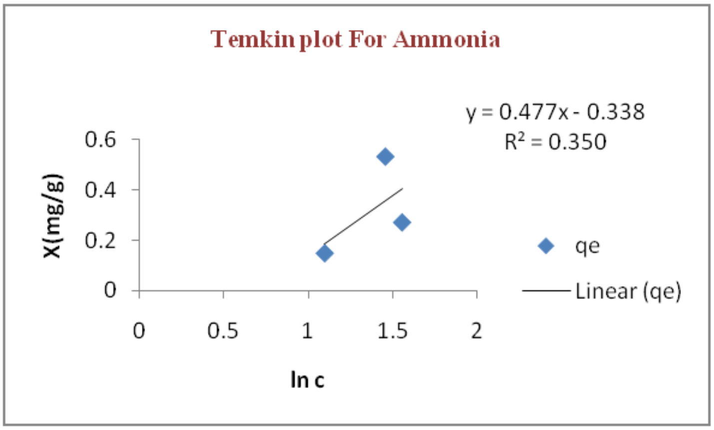


Sr.No	Parameters	K_f (mg/g)	n(L/mg)	R^2
1	Sulphate	0.63	-	0.956



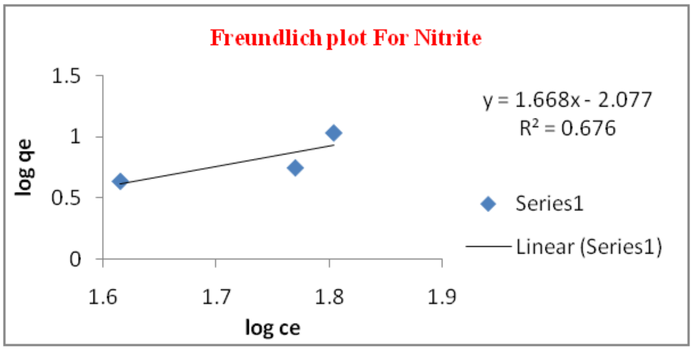
Sr.No	Parameters	a(mg/g)	b(L/mg)	R^2
1	Sulphate	71.49	282.8	0.995

6) Ammonia: Temkin



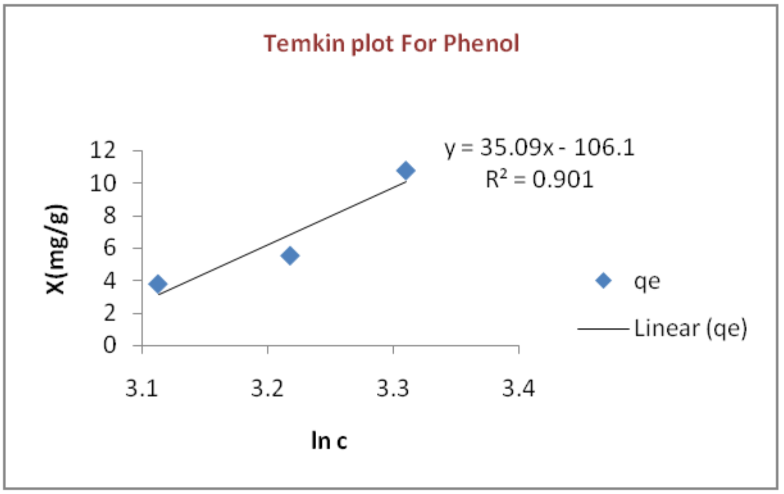
Sr.No	Parameters	a(mg/g)	b(L/mg)	R^2
1	Ammonia	0.477	-0.338	0.35

7) Nitrite:Freundlich



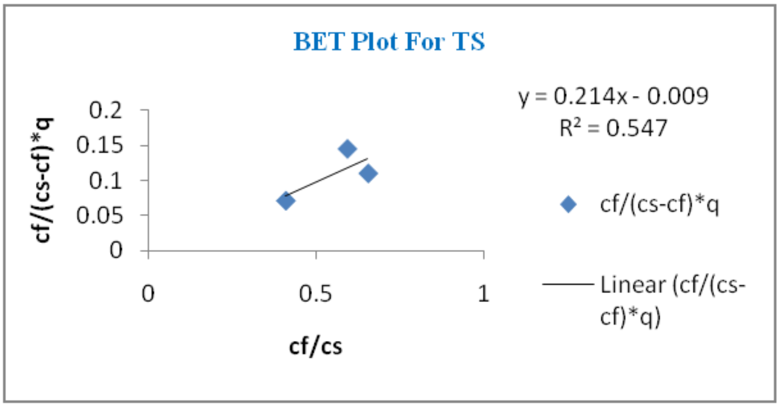
Sr.No	*Parameters*	*K_f (mg/g)*	*n(L/mg)*	*R^2*
1	*Nitrite*	*0.599*	*-*	*0.676*

8) Phenol :Temkin



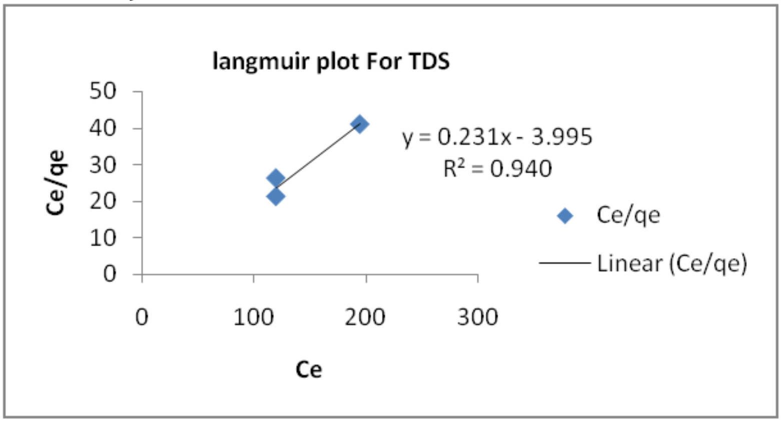
Sr.No	*parameters*	*a(mg/g)*	*b(L/mg)*	*R^2*
1	Phenol	35.09	106.1	0.901

9) TS (Total Solids): BET

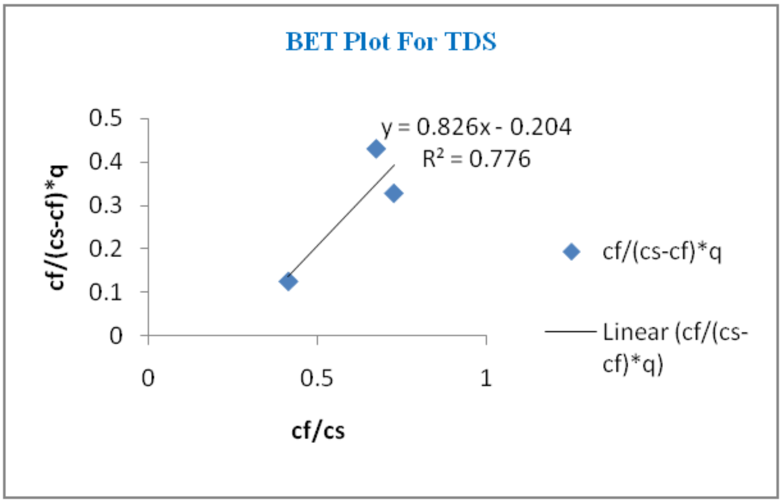


sr.no	*parameters*	*qmax(mg/g)*	*B(L/mg)*	*R2*
1	TS	4.87	0.95	0.547

10) TDS (Total Dissolved Solid):

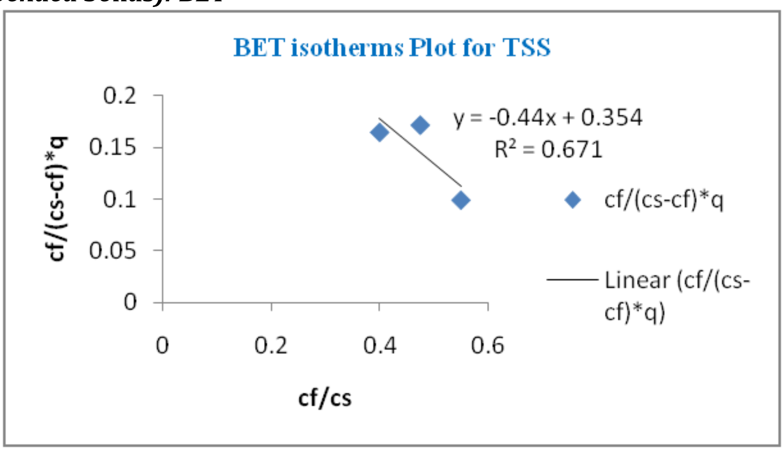


sr.no	Parameters	Q^0(mg/g)	b(L/mg)	R^2
1	TDS	4..32	-0.57	0.94



Sr.No	Parameters	q_{max}(mg/g)	B(L/mg)	R^2
1	TDS	1.60	0.751	0.776

11) TSS (Total Suspended Solids): BET



Sr.No	Parameters	q_{max} (mg/g)	B(L/mg)	R^2
11	TSS	11.62	-0.195	0.671

- ***Temkin adsorption isotherm followed in very well manner for Sulphate and Phenol.***
- ***Langmuir and BET adsorption pattern followed for COD.***

6.0 CONCLUSION:

- To recent worldwide trend to achieve higher environmental standards favors the usage of low cost systems for treatment of effluents the application of Low Cost Natural adsorbent (Great millet husk: which is totally unused after agricultural crop is removed from their) in water treatment is boon for Environment Scientist and Government authorities. It is due to their inexpensiveness and free availability. Their applications are more important in developing and under developing countries. This results indicates that this procedure show a promising efficiency for removing COD and color and other parameters. The effectiveness of the millet husk is very good. After using one time; It also be further used as 50 % adsorbent capacity.

- According to isotherms studies that concluded that, Langmuir and BET isotherms are interrelated with each other. That says that the parameter which follow Langmuir adsorption isotherms that parameters also follow BET adsorption isotherms models. and Temkin and Freundlich are also interconnected with each other.[34,35]
- According to studies, In Langmuir and BET isotherms models the value of $R^2$is more than 0.7 is very good value and good adsorption process followed in adsorption. In this all parameters Alkalinity, COD, TS, TDS and TSS parameters follow Langmuir isotherms models. It indicates first layer of molecules adhere to the surface with energy comparable to heat of adsorption for monolayer sorption and subsequent layers have equal energies.[36-38]
- A negative value for the intercept (in essence the uptake of the monolayer is physical nonsense. By linearization of the Langmuir isotherm as you do it, you overemphasize data points at a low concentration (typically those with a high associated error). There are different ways of linearizing the Langmuir expression, e.g.

$Ce/Qe = 1/Q_m *Ce+ (1/bQ_m)$…………………… (6.3.1)
$Qe = Q_m- (1/b)*(Qe/Ce)$…………………… (6.3.2)

- The best way would be to perform a non-linear regression on this data (with as the initial guess one of the solutions to the linear regressed models - which is physically sound).
- Here we can say that BET isotherms as an extension of the Langmuir isotherms to account for multilayer adsorption and Langmuir isotherms applies to each layer. BET surface area is essentially comes from how much amount is adsorbed.
- The polar group on the surface is responsible for adsorption due to cation exchange capacity.
- This all data may be useful in designing and fabrication of an economic treatment plant for the removal of impurities of waste water.[39]

7.0 RECOVERY OF ADSORBENT:

- Used adsorbent is than further given treatment with H_2SO_4 and tap water, not 100% capacity, but at least 50% of adsorption capacity.

8.0 References

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