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Use of Waste egg shells as novel chitosan-based adsorbent for dye removal

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Abstract: Chitosan is amino polysaccharide and it is considered to be one of the most promising and applicable materials in adsorption applications. Chitosan is commercially obtained by deacetylation of chitin produced from crustaceans and waste food stuffs like egg shells as the most abundant natural sources. The existence of amino and hydroxyl groups in its molecules contributes many adsorption interactions between chitosan and pollutants. In this experimental study, Chitosan was first prepared by proper treatment of eggshells and adsorption is compared of commercially available chitosan with the prepared chitosan from eggshell. Adsorption capacity of chitosan was also checked by use of Langmuir and Freundlich adsorption isotherms. This research work reveals that information that chitosan prepared by waste food stuff can be used as potential adsorbent for waste water treatment.

Keywords*: Chitosan, Marine creature, Eggshell, Waste water treatment.*

1. Introduction

Chitin is an amino polysaccharide existing in fungi, plants, animals and has been expected as a promising material because of a variety of biomasses. It is a natural polysaccharide corresponding to the second most naturally abundant polysaccharide, after cellulose. Chitosan is also an important polysaccharide easily obtained through deacetylation treatment of chitin [1]. Nowadays, chitin and chitosan are produced from bio wastes obtained from aquatic organisms and waste stuff. Approximately 70% of marine capture fisheries are used for processing and a considerable quantity of the processed catch remains as wastes. Chitosan is commercially obtained by deacetylation of chitin produced from crustaceans [2].

The advantages of using chitosan are the polymer can be obtained from waste products of the seafood industry and the polymer is renewable, non-toxic and biodegradable. Thus, as a natural renewable resource, chitosan exhibits unique properties such as biocompatibility, biodegradability, non-toxicity and antibacterial properties. Chitosan has a number of commercial and possible biomedical uses. In industry, it can be used in self-healing polyurethane paint coating. Chitosan can be used in water processing as a part of a filtration process. Chitosan has been used to precipitate caseins from bovine milk and cheese making. Chitosan is a biopolymer obtained by deacetylation of naturally occurring biopolymer chitin. Chitosan is a cationic copolymer of 2-glucosamine and N-acetyl-2-glucosamine. Chitosan is able to coordinate with metals because of high concentrations of amino ($-NH_2$) functional groups. Thus, the amino and hydroxyl groups in chitosan act as the active sites for adsorption. Chitosan has a tendency to form a gel in acetic acid and this chitosan gel can be precipitated in alkali to obtain spherical beads [7]. Chitosan has used as adsorbent for decontamination of wastewaters from various pollutants, either organic or inorganic species [4]. A variety of procedures have been developed over the years for the preparation of chitin and chitosan. Some of these forms the basis of chemical processes for industrial production of chitosan from crustacean shell waste. Isolation of chitin from waste stuff consists of two basic steps: (1) protein separation-deproteinization, and (2) calcium carbonate (and calcium phosphate) separation demineralization. These steps also can be conducted in a reverse order, i.e. demineralization, followed by deproteinization. However, if protein recovery is an objective, its extraction before demineralization is preferred so as to maximize protein yield and quality. Manufacture of chitosan from waste egg shells which include decalcification, deproteinization, decolorization and deacetylation.

2. Material and Methods

Waste eggshells were collected from the market. An eggshell is with outer covering of a hard-shelled egg and of some forms of eggs with soft outer coats. About 100 gm waste eggshells were taken in a beaker, washed thoroughly with water to remove adherent impurities and dried in sun light for 1 day.

Dried eggshells were crushed using mortar & pestle and converted into powder form. Required uniform sizes of crushed eggshells were obtained by using sieve shaker. Removing moisture from the eggshells powder is mandatory and thus, again drying was performed in a oven for nearly 10 h at 100º C.

Figure 1: Waste Egg shells

2.1 Decalcification Process

Decalcification process is the removal of calcium ions from powder of eggshells. 100 ml of 5 % HCl solution (Prepared in the laboratory) and 30 g eggshells waste were taken in a conical flask. Eggshells were treated with dilute aqueous HCl solution in magnetic stirrer at room temp. It was stirred for 3 hours and after stirring, this solution filtered by using filter washed by water.

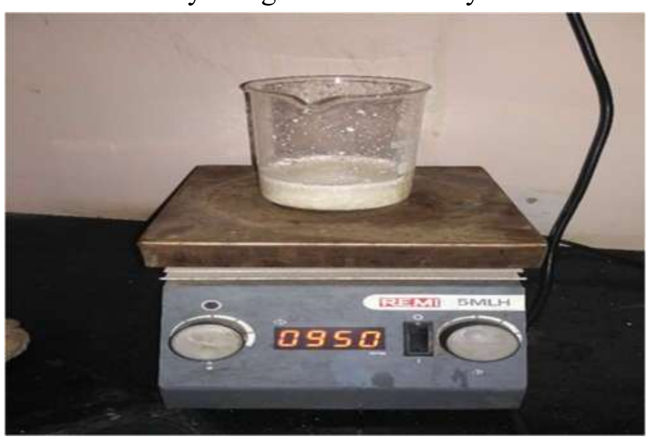

Figure 2: Decalcification process

2.2 Deproteinization Process

Deproteinization process includes treatment of 0.5% NaOH with filtrate solution after completing deproteinization for 24 hours at room temperature and 0.5% Oxalic Acid treated with filtrate for 4 hour at room temperature. This solution was filtered by using Whatman filter paper and was washed by water. After completing Deproteinization, chitin is obtained as powder form. Chitin (being raw material) is most important chemical for preparation of chitosan.

2.3 Decolarization Process

Decolarization was the process of removing color from powder of waste egg shells. Initially, 0.5% $KMnO_4$ solution is treated with filtrate solution of after completing deproteinization for 24 hour at room temperature and 0.5% Oxalic Acid treated with filtrate for 6 h at room temperature. The solution was filtered by using Whatman filter paper and was washed by water.

Figure 3 : Decolarization process

2.4 Deacetylation

Deacetylation is the process of conversion of chitin to chitosan. Chitin was heated to nearly 100ºC for 5-7 hour with 50% caustic soda solution (NaOH) in vessel. Excess alkali was drained off and the mixture was washed with water several times until it is free from alkali. The product was dried under sunlight for 12 hours till the moisture content almost becomes nil and thus chitosan obtained is in the form of flakes.

2.5 Adsorption Isotherms

2.5.1 Freundlich Adsorption Isotherm

An adsorption isotherm is a curve relating the concentration of a solute on the surface of an adsorbent to the concentration of the solute in the liquid with which it is in contact. Freundlich Isotherm Equation

$$\log (x/m) = \log k + (1/n) \log C \qquad (1)$$

Where x is the amount of the adsorbate and m is the amount of the adsorbent, C is the equilibrium concentration of the adsorbate at a particular temperature and k & n are constants. If a graph is plotted between log (x/m) as ordinate versus (log C) as abscissa, a straight line would result. The slope of this line will be equal to 1/n and log k will be given by the intercept which the straight line makes on the ordinate.

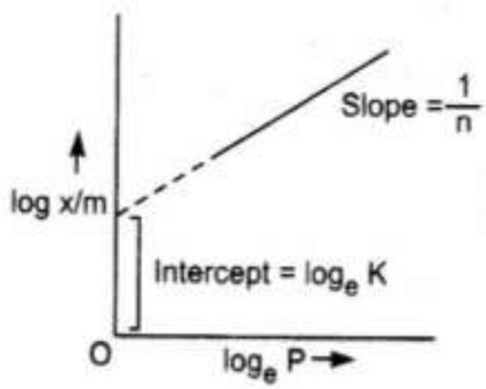

Fig.4 log x/m Vs log_eP

2.5.2 Langmuir Adsorption Isotherm

Langmuir also derived another relationship between the amount adsorbed and the equilibrium concentration of the solute as

$$C/(x/m) = 1/a + Q\,(C/a) \qquad (2)$$

Where α and β are constants.

This equation can be verified by plotting (C/x/m) against C, which would result in straight line. The slope will give β / α and the intercept will give 1 / α.

2.5.3 Adsorption study of Reactive Blue dye on chitosan by Freundlich and Langmuir Adsorption Isotherm

Experiment conducted by taking well cleaned and dried five stoppered reagent flask labelling them from 1 to 5 in a sequence.

Table 1 : Experimental Data

Flask No.	1	2	3	4	5
Reactive Blue Dye (ml)	25	20	15	10	5
Water (ml)	5	10	15	20	25

Fig.5 Reactive Blue dye fill in beaker

Concentration of decided solutions was taken using colorimeter as described in above table. Weighted 1 gm chitosan taken in each of the bottle. Concentration values of these solutions were taken with the help of colorimeter.

3. Results and Discussion

3.1 Adsorption Analysis

0.024 gm reactive red dye solution is being used to observe the adsorption capacity of the prepared chitosan in the laboratory. Initially, the commercial chitosan which is
already available in the market is being taken and checked the adsorption capacity using colorimeter. It turned to be 83.33%. Then, the chitosan which was prepared from eggshells is also being analyzed for their adsorption capacity by colorimeter. It exhibited to 68.33%.

Table 2 : Experimental Data of Adsorption (%)

Sr. no.	Samples	Colorimeter Reading (gm/ml)	Adsorption (%)
1	Chitosan (Eggshells)	0.0164	68.33
2	Chitosan (Commercial)	0.02	83.33

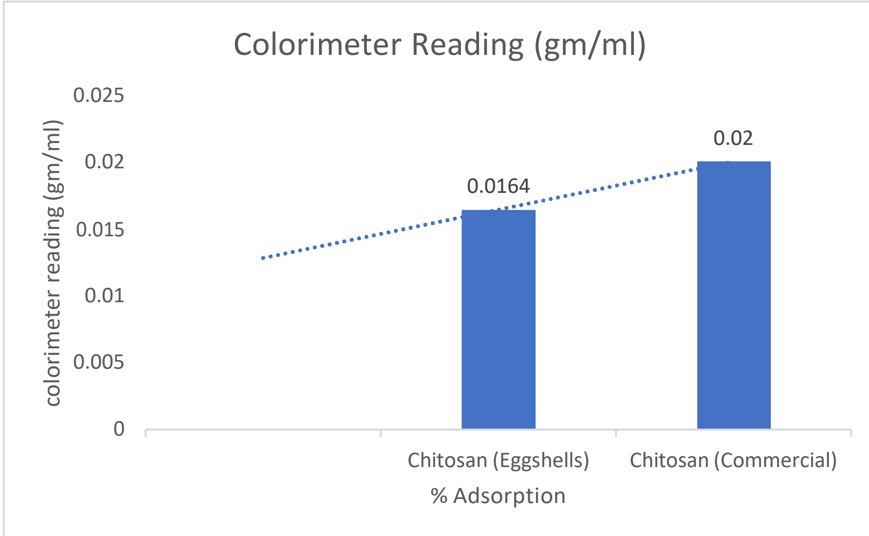

Figure 6 : Comparison between commercial chitosan and Egg shell (chitosan)

a) Blue dye solution

(b) Prepared chitosan in dye solution

(c) commercially available chitosan in dye solution

Fig.7 Different Solution (a) Blue dye solution (b) prepared chitosan in dye solution (c) commercially available chitosan in dye solution

3.2 Freundlich adsorption and Langmuir adsorption isotherm:

Concentration of Reactive red dye before and after adsorption is mandatory to be calculated. 0.0024 gm quantity dye in 400 ml water was taken and get initial concentration of solution which turned out to be 6 x 10^{-5}. During the experimental work, constant volume of 60 ml solution in five different flasks were taken and different amount of adsorbent in each of them.

Table 3 : Experimental Data of Freundlich adsorption isotherm

C(Initial) (gm/ml)	X (gm)	M (gm)	X/M	Log (X/M)	(-log) (X/M)	C (Final)	Log C	-Log C
0.00006	0.0036	0.25	0.0144	-1.84164	1.841638	0.000050	-4.30103	4.30103
0.00006	0.0036	0.50	0.0072	-2.14267	2.142668	0.000038	-4.42021	4.42021
0.00006	0.0036	0.75	0.0048	-2.31876	2.318759	0.000042	-4.37675	4.37675
0.00006	0.0036	1.00	0.0036	-2.44370	2.443697	0.000040	-4.39794	4.39794
0.00006	0.0036	1.25	0.0028	-2.54061	2.540608	0.000033	-4.48148	4.48148

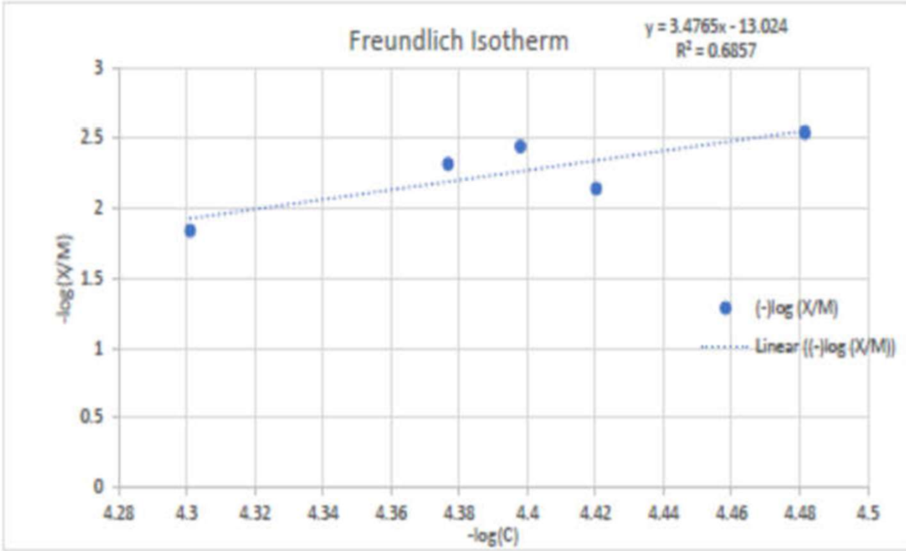

Figure 8 : (-log)(X/M) vs – log (C)

Table 4 : Experimental Data of Langmuir adsorption isotherm

C (Initial) (gm/ml)	X (gm)	M (gm)	X/M	C/(X/M)	C (Final) (gm/ml)
0.00006	0.0036	0.25	0.0144	0.003472	0.000050
0.00006	0.0036	0.50	0.0072	0.005278	0.000038
0.00006	0.0036	0.75	0.0048	0.008750	0.000042
0.00006	0.0036	1.00	0.0036	0.011111	0.000040
0.00006	0.0036	1.25	0.0028	0.011458	0.000033

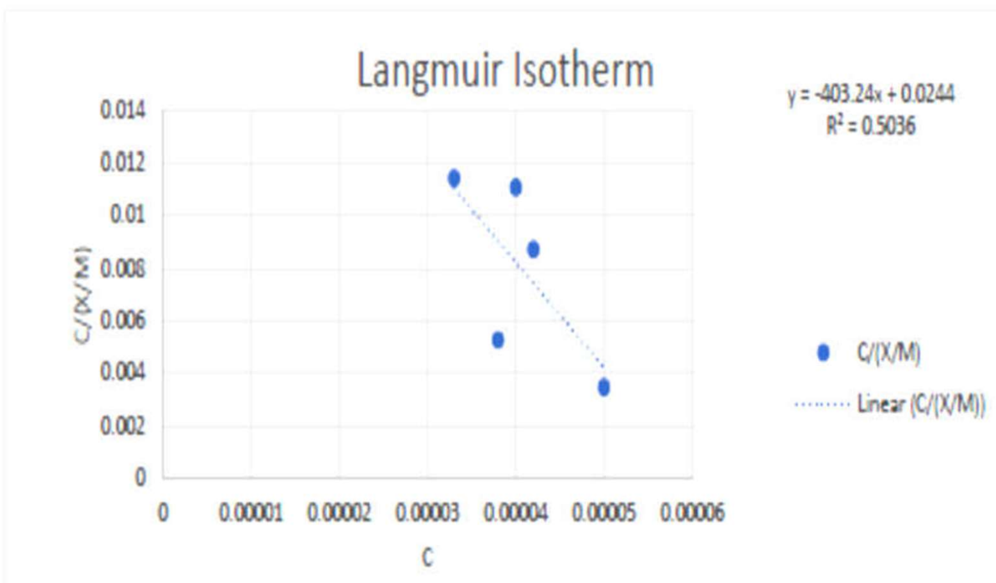

Figure 9 : C/(X/M) vs C

The graph of log (x/m) v/s log (C) (Figure (5)) is almost straight line which reveals the following nature of adsorption pattern as per Freundlich Isotherm and their R^2 value of graph is 0.6857 but while comparing the graph of C/(x/m) v/s C is not perfectly straight line for all concentration, as it does not

perfectly follow Langmuir Isotherm and their R^2 value of graph is 0.5036. From the experimental work it is observed that while comparing the two quantities (0.5 gm and 1.25 gm) chitosan, it is reported to be low final concentration i.e adsorption is high in case of 0.5 gm chitosan usage.
The results were also studied taken time as basis. After 24 hours and 48 hours observation were noted here and reported the final view based on adsorption isotherm plot.

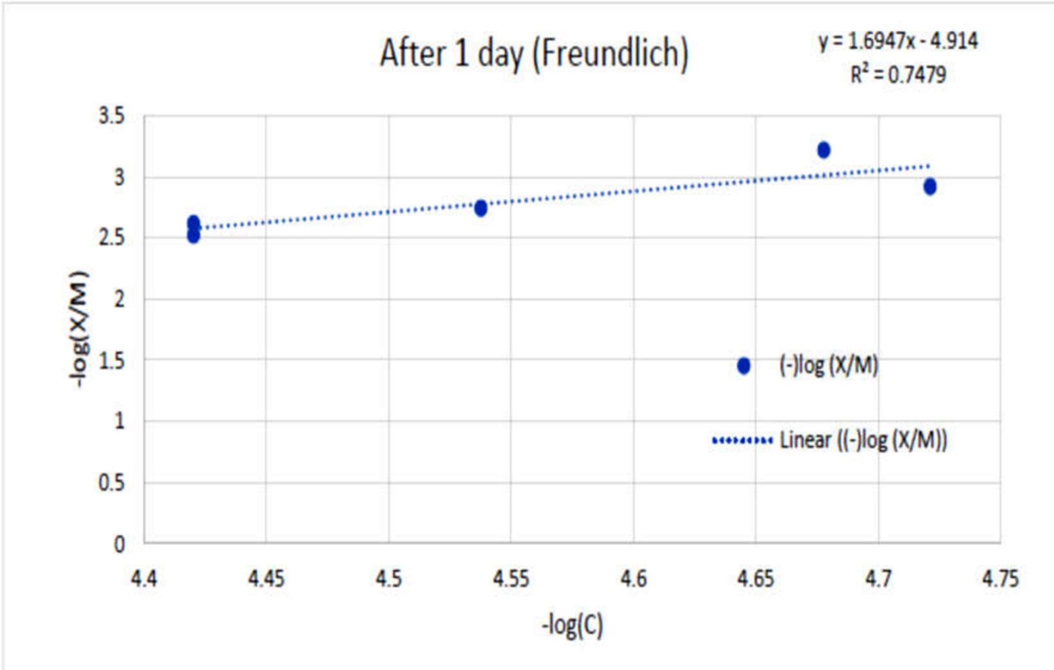

Figure 10 : (-log)(X/M) vs – log (C) (Freundlich Isotherm) after 24 hours observation

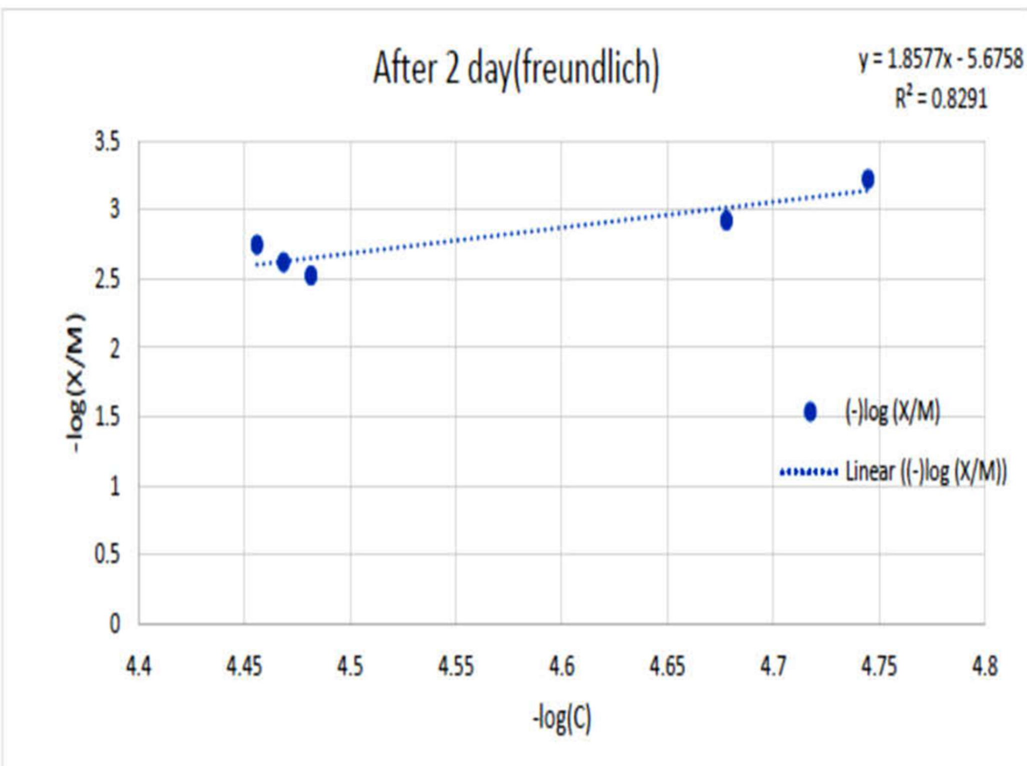

Figure 11 : (-log)(X/M) vs – log (C) (Freundlich Isotherm) after 48 hours observation

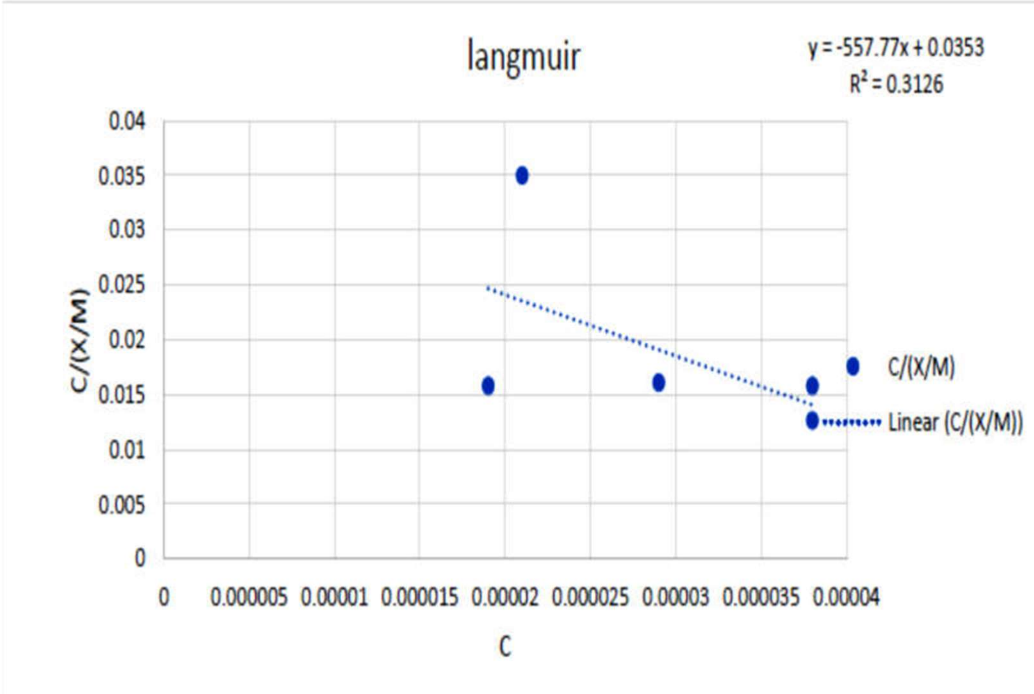

Figure 12 : C/(X/M) vs C (Langmuir isotherm) after 24 hours observation

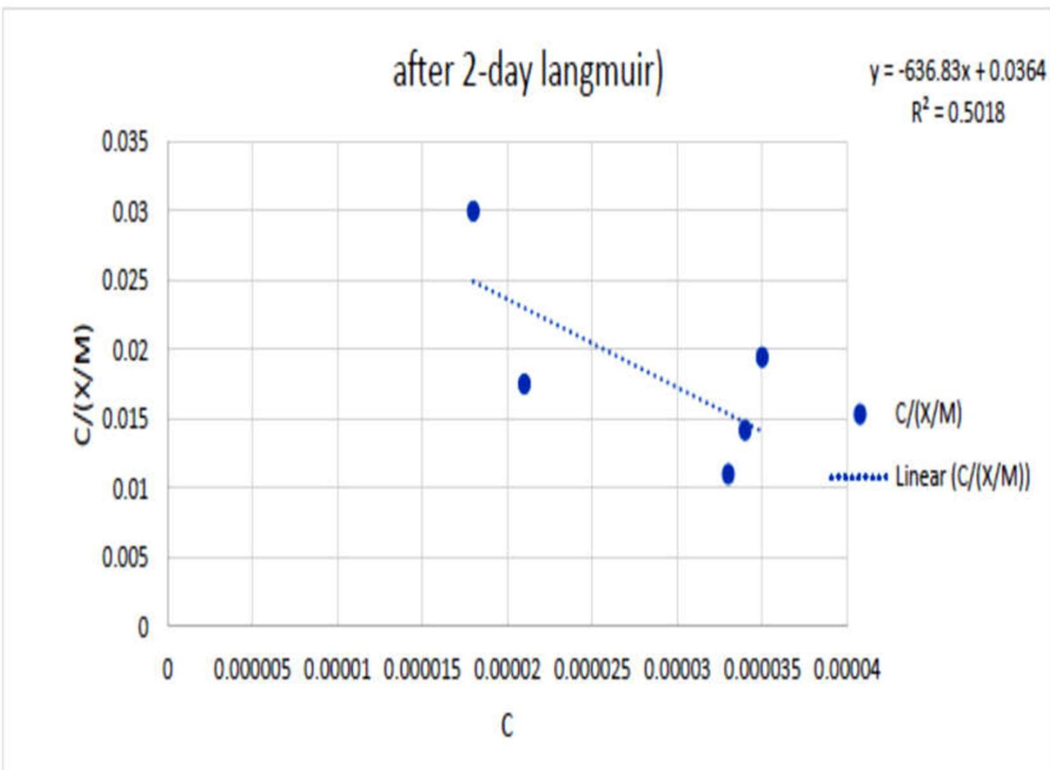

Figure 13: C/(X/M) vs C (Langmuir isotherm) after 48 hours observation

4. Conclusion

After the successful experimental work, author have given observation on adsorption activity as

- After 24 hours, the adsorption capacity of prepared chitosan is less as compare to commercial chitosan which is around 68.33 % (While commercially available is 83.33 %). The reason behind the less adsorption capacity of around 15% is improper surface creation which actually the key factor in enhancing the capacity of adsorption phenomenon.

- There is no much change found in the adsorption curve or adsorption phenomenon while depicting the longer time (24 and 48 hours). The maximum adsorption takes place at the initial phase of time only.
- While applying both the isotherms, Freundlich and Langmuir, Freundlich is best fitted in the plot and suitable for the prescribed results.

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