

# Polymeric Cellulose Derivative: Carboxymethyl-Cellulose as useful Organic Flocculant against Industrial Waste Waters

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## ABSTRACT

The present study focuses on polymeric cellulose derivative; Carboxymethyl-Cellulose (CMC) prepared from locally available cheap cotton stalks considered as waste. The successful synthesis and extraction of polymer CMC was done with etherification process involving use of Monochloroacetic acid and Sodium Hydroxide. The polymeric flocculant was examined and optimized for its dosage level (70mg/L), optimum pH level (7pH) and reduction efficiency in percentage (average 60%) of Total Dissolved Solids (TDS), Conductivity, Turbidity, and Chemical Oxygen Demand (COD), when applied against industrial wastewater samples. The statistical study of natural polymeric flocculant was conducted using Langmuir and freundlich adsorption isotherm models. Langmuir model was found suitable for adsorption of effluent pollutants and revealed mono layer formation on a homogeneous surface of CMC.

**Keywords :** Carboxymethyl Cellulose; Cotton Stalks; Organic Flocculant; Wastewater

## 1 INTRODUCTION

CHEMICAL process industries are producing number of valuable products and generating highly toxic and hazardous wastewater. The direct disposal of toxic wastewater into river or sea causes harmful effects on marine and human life. These wastewaters increase the concentration of pollutants at high rate and damage living tissues of marine bodies. Due to this reason, the available water resources in world are going to be polluted and shortage of pure water is increasing day by day. Overall the world is trying to preserve the water resources by implementing cost effective wastewater treatment policies. During 1951 to 2008 per capita water consumption in Pakistan reduced continuously by 5000 cm<sup>3</sup> to 1100 cm<sup>3</sup> due to improper implementation of wastewater treatment policies [9], [12]. In terms of population, around 55% found short of safe water reach, uncovered by economic survey [6]. Pakistan is also rich in industrial sector and generating high rate of wastewater per day that is disposed off directly into rivers and seas without any proper and effective treatment. It is confirmed now that industrial wastewater possesses thousands of organic and inorganic impurities which create disinfectant byproducts and cause of many water borne diseases. The removal of suspended solids, pathogens, organic matter from effluents requires mandatory disposal or treatment before human consumption in order to prevent water borne plagues and diseases [8]. Many technologies and processes are utilized for the treatment of industrial wastewater, still efforts have to be taken for cost effective treatment of industrial wastewater by utilizing natural or waste materials.

Natural plants rich in carbohydrates and polysaccharides. About 90% wastewater could be converted into chemical and biochemical forms. Cellulose is available in abundant quantities as renewable biopolymer in nature [15]. Cotton stalks as an agricultural waste produced from cotton crops in Pakistan have no proper utilization in industrial applications. Currently cotton stalks are used as fuel for domestic cooking. Pakistan cultivated cotton crops up to 13.3 million bales and Sindh province cultivated close to 3.5 million bales in year of 2012. The waste generation rate of cotton stalks in Sanghar district was 1.42 millions tons (14,23,129 tons) in year 2009 [7]. Cotton stalk consists of cellulosic and polysaccharide materials. The properties of agricultural waste cellulosic materials have been modified chemically using carboxymethylation reactions [11]. The Cellulose derivative i.e. Carboxymethyl Cellulose (CMC) have carboxymethyl groups  $-CH_2 - COOH$  attached to hydroxyl groups on the backbone of cellulose molecule of glucopyranose units. Synthetic cellulose materials have applications in agricultural, wastewater treatment, drug release and hygienic products because of their abundant resources, low production-cost and bio-degradability [1]. In contemporary world, research is focused on wastewater treatment using organic polymers with applications on contaminants [15].

Present research emphasized on the effective reduction of toxic pollutants from industrial wastewaters by utilizing cost effective agricultural waste cotton stalks as advanced natural polymeric flocculant. In other words, CMC is the point of focus.

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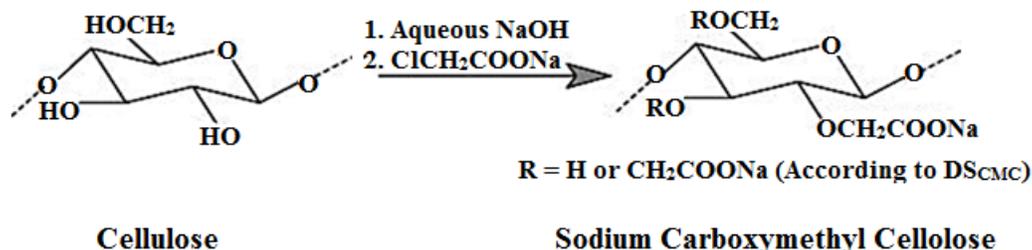


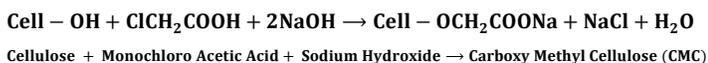
FIG-1: REACTION SCHEME OF THE CARBOXYMETHYLATION OF CELLULOSE [11]

## 2 MATERIALS AND METHODS

### 2.1 Synthesis of Sodium Carboxymethyl Cellulose from Cotton Stalks

The samples of fresh cotton stalk linters were collected from agricultural fields of district Sanghar (Sindh). The cotton stalks were shredded from linters and cut into small pieces with the help of cutter. The pieces were dried into oven for 48 hours at 105°C. These dried samples were crushed into cooled disc pulverizer, (type 1025 W, Tokyo, Japan) at 250 rpm. The ground samples were sieved by Sieve Shaker type (A-871205, Japan) with speed of 300 rpm. The quantity of fine powder sample (25 grams) was measured through analytical balance (CHYO Electronic balance JP2-160) and was poured into beaker (100mL).

Synthesis of carboxymethyl cellulose involved two chemical reaction steps. In first step, cotton stalks cellulosic material was treated with NaOH (30% concentrated solution) for 5 hours which acted as swelling and dilutant agent. The alkaline cellulosic material was further treated with monochloro acetic acid (50 mL) at temperature of 75°C for 4 hours with constant stirring on magnetic hot plate stirrer [3]. After its cooling, volume of 25 mL of iso-propanol solvent (Merck Co. Germany) was added into sample, then solution was left for 10 minutes and filtered. The chemical reaction scheme of the carboxymethylation of cellulose is shown in Fig-1. The alkali medium reaction of cellulose and the etherification process in aqueous system was carried out at 75°C, according to the chemical reaction formula:



Then sample was washed through distilled water for some moment, and then discarded through vacuum filtration system. The pH of prepared carboxymethyl cellulose was maintained up to 7.0 by use of 1M HCl. Again 30 mL of methanol was added into sample and filtered. The filtered sample was kept in oven (Arm field Co. Ltd) for drying and activation for 24 hours at 70°C then cooled. Prepared carboxymethyl cellulose polymeric flocculant was used for treatment of industrial wastewater samples [3].

### 2.2 Collection and Analysis of Industrial Wastewater Samples

Industrial wastewater samples of Hyderabad industrial area were collected from five different industries locations for consecutive three months. The samples were collected and stored according to standard sampling protocol [4]. Effluent pollutants such as conductivity, total dissolved solids (TDS), turbidity and chemical oxygen demand (COD) were considered for analysis and its treatment purpose. Turbidity of wastewater samples was measured through Spectrophotometer (DR-2000, Hach, USA) by using absorptometric method at wavelength 450 nm. TDS, Conductivity were observed via conductivity meter (Model# 5475017, Hach, USA). The COD of the samples were examined by common method involving potassium dichromate.

### 2.3 Treatment of Industrial Wastewater through CMC Flocculant

The industrial wastewater samples were treated through prepared polymeric flocculant CMC. The reduction in pollutants such as COD, turbidity, TDS and conductivity were measured against wastewater samples (100mL) when poured into conical flask and different quantities of polymeric flocculant (20 – 80mg) were added in it. The parameters were kept constant i.e. 5 minutes time for agitation at temperature 30°C with 1 hour settling time. After flocculation process, flocs were settled and filtered. Each sample was analyzed for effects of flocculant dosage on reduction of pollutants. Effects of pH variations (5–8) on the pollutants reduction were analyzed [13].

### 2.4 Determination of Adsorptive Capacity of Cellulose Derivative CMC

The quantity of sorption  $q$  in batch experiments, ( $mg.g^{-1}$ ), was ascertained by Eq.(1):

$$q = \frac{(C_0 - C_e)V}{m} \quad \dots (1)$$

Where  $C_0$  and  $C_e$  ( $mg/dm^3$ ) confirms solute concentration at initial and final levels in solution,  $V$  ( $dm^3$ ) presents solution volume and  $m$  the polymeric flocculant CMC mass ( $mg$ ) [10].

In same way, adsorption percent of pollutants by use of flocculant was determined by Eq.(2):

$$\% \text{ Sorption} = \frac{(C_i - C_f)}{C_f} \times 100 \quad \dots (2)$$

The parameters  $C_i$ ,  $C_f$  (mg/L) represents initial and final concentrations of effluent pollutants [14].

### 3 RESULTS AND DISCUSSIONS

#### 3.1 Treatment of Wastewater at Optimum Polymeric Flocculant (CMC) Dose

The industrial wastewater samples, collected from industrial area of Hyderabad city were analyzed for their pollution contents. The effluent parameters were analyzed according to the laboratory protocols. The effluent samples were treated by use of carboxymethyl cellulose at dosage variations (20–80 mg/L). From Lab results and research study, it was observed that removal rate of turbidity increased along with increase in CMC dose. Better results were observed at dose of 70 and 80mg/L. High dosing of CMC, maximum contact time and rate of mixing at specific temperature, improved the turbidity removal rate. In sample-1, removal efficiency (63.5%) was achieved at optimum dose (70mg/L). In same way, sample-3, the turbidity removal efficiency (63.7%) was achieved at optimum dosing of CMC (70mg/L). Fig-2 expresses the effects of CMC dose in the reduction of turbidity of wastewater samples. Steady state condition was observed in further increasing dose. No further turbidity reduction rate was observed. Thus prepared flocculant has good capability in flocculation process to remove high rate of turbidity at optimum dosing of flocculant due to their chemical behavior, composition and structure. The adsorptive capacity of CMC was measured for turbidity reduction at dosage variations. In case of initial dosing of 20 and 30mg, adsorptive capacity was higher, further on increasing dose, their adsorptive capacity declined. Again on 70mg dose, adsorptive capacity was improved. Higher adsorptive capacity was observed at optimum dose of CMC (70mg/L). Fig-3 shows the effects of CMC dose in the reduction of turbidity of wastewater samples.

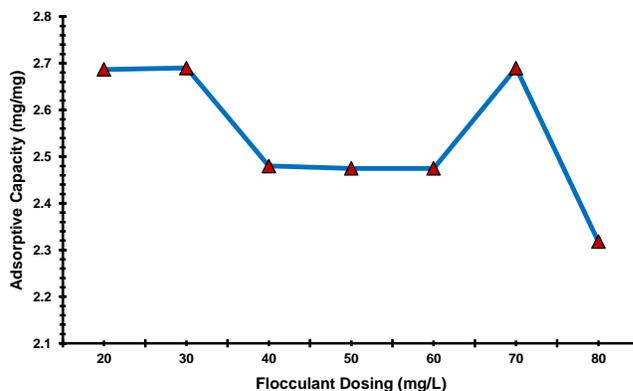


Fig-3: Adsorptive capacity ( $q_e$ ) of CMC for turbidity reduction

#### 3.2 Effects of pH Variations on Turbidity Removal

Industrial wastewater samples were treated through prepared natural polymeric flocculant carboxymethyl cellulose at optimum dose (70mg/L) at pH variations (5–8). In acidic nature, CMC showed low removal rates. Industrial wastewater samples of different industries locations were treated at pH range of 5–8. From the laboratory experimental work, it was concluded that flocculation in acidic nature was ineffective for reduction of turbidity of waste water sample in use of CMC. When pH (7) of sample was maintained and wastewater was treated at 70mg/L dose, higher removal rate of turbidity was recorded. Further increasing alkalinity of sample, less reduction rates were observed. In case of higher pH in alkaline form, uniform reduction rate of turbidity was observed. In water sample-3, maximum turbidity reduction rate was observed. Fig-4 shows the effect of pH variations on the turbidity reduction of wastewater samples. Treatment efficiency of flocculation changed according to composition of wastewater.

#### 3.3 Removal Efficiency of CMC on Turbidity Reduction

Industrial wastewater samples were collected from five locations of industrial SITE area of Hyderabad for three months consecutively. From preliminary analysis of water samples, the turbidity initial of samples observed at 572 FTU, 594 FTU and 141 FTU. It was higher than NEQS limit.

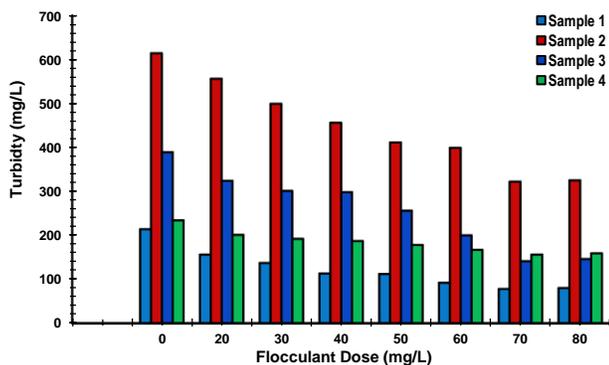


Fig-2: Effects of CMC dose in the turbidity reduction of wastewater samples

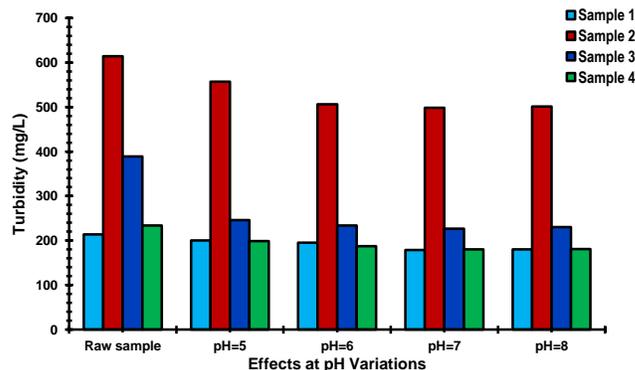


Fig-4: Effects of pH variations on the turbidity reduction of wastewater samples

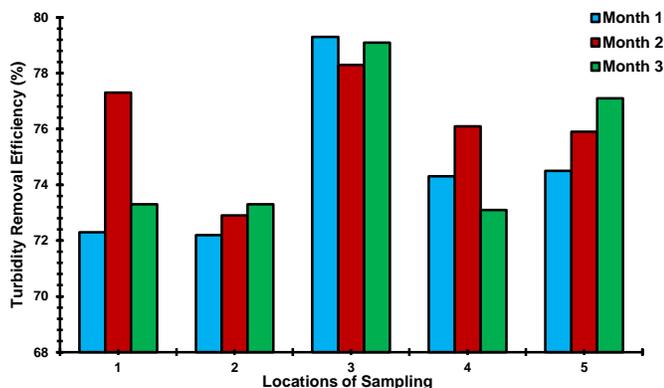


Fig-5: Removal efficiency of turbidity in use of CMC

Therefore samples were treated with carboxymethyl cellulose at dosing of 70mg/L. In location-1, removal rates of turbidity were achieved as 72.3%, 77.3% and 73.3% for consecutive three months respectively. In same way, at location-2, turbidity removal rates were achieved as 72.2%, 72.9% and 73.3% for consecutive three months respectively. At location-3, turbidity removal rates were achieved by 79.3%, 78.3% and 79.1% for consecutive three months samples. Turbidity removal rates of location-4 and 5 were similar to locations-1 and 2. It was concluded that carboxymethyl cellulose flocculant was effective for turbidity reduction and reduced turbidity up to 75% at optimum dosing of 70 mg/L of CMC. Efficiency of CMC varied according to the nature, composition and rate of pollution load. Fig-5 shows the removal efficiency of turbidity in use of CMC.

### 3.4 Effects of Carboxymethyl Cellulose on COD Reduction

From the preliminary laboratory wastewater analysis, it was found that COD of samples was between the ranges of 3.0 – 6.5 mg/L. This limit is not objectionable from EPA authority. This range has less effect on marine animals and consumed less oxygen. Samples were collected from five different locations and were treated with flocculant CMC at dose of 70mg/L. The removal efficiency of CMC for COD was remained between 50-65%. In this study, samples were collected on monthly basis for 3 months. At location-1, initial COD of samples of three

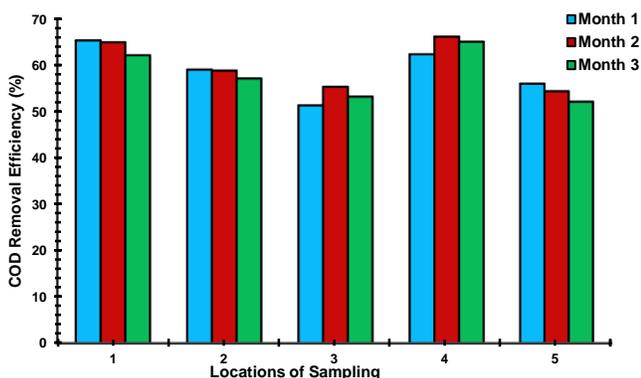


Fig-6: COD Removal efficiency in use of CMC

consecutive months was recorded at 3.96, 3.80 and 3.21 mg/L respectively. After flocculation treatment with CMC the COD removal rates were achieved upto 65.3%, 64.9% and 62.1%. At location-2, initial COD of samples was 8.97, 7.98 and 9.01 mg/L respectively. After treatment with CMC, removal rates were achieved as 59.01%, 58.80% and 57.10%. At location-3, the initial COD of samples was 5.81, 5.79 and 5.60 mg/L respectively. After treatment, it was recorded as 1.87, 1.61 and 2.21 mg/L respectively. At location-4, initial turbidity was between ranges of 5.00, 5.80 and 5.62 mg/L. The removal efficiency was recorded as 62.30%, 66.10% and 65.01% for consecutive three months. At location-5, initial COD of samples was 6.12, 6.38 and 6.01 mg/L. After treatment the removal efficiency was recorded as 55.98%, 54.36% and 52.10%. From above observations, it was concluded that carboxymethyl cellulose flocculant could reduce COD of samples between ranges of 50 – 73%. Fig-6 shows COD removal efficiency in use of CMC. This removal efficiency of CMC varied due to complex nature, properties and high pollutants load in wastewater.

### 3.5 Effects of CMC on Removal of Total Dissolved Solids (TDS)

In same previous practice, industrial wastewater samples were treated with cellulose derivative flocculant CMC at dosing of 70 mg/L. In this study, effect of flocculant on TDS removal was analyzed in industrial wastewater samples. At location-1, the initial TDS of samples was 4320, 4121 and 3310 mg/L respectively. After treatment through flocculation technique, removal efficiency for TDS was recorded as 54.02%, 54.10% and 47.64%. At location-2, initial TDS were 2907, 3001 and 3105 mg/L respectively. After flocculation treatment the removal efficiency for TDS was calculated as 52.10%, 52.20% and 54.87%. In location-3, higher reduction rate for TDS was recorded. The removal efficiency was calculated as 54.45%, 55.11% and 52.50% for consecutive three months. At location-4, removal efficiency for TDS was analyzed as 50.20%, 46.67% and 48.40%. In same way for location-5, the removal efficiency was recorded as 50%, 51.67% and 49.60%. Fig-7 shows the TDS removal efficiency in use of CMC flocculant.

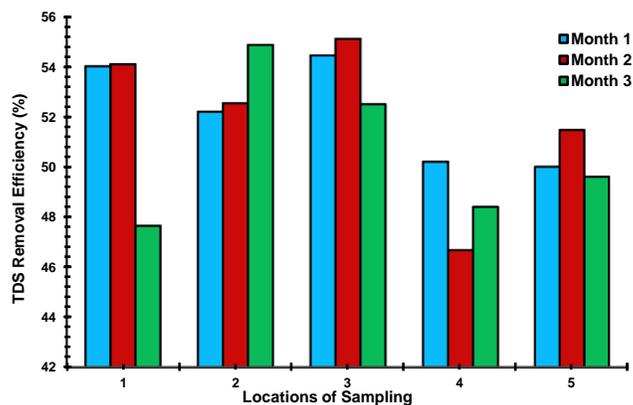


Fig-7: TDS Removal efficiency in use of CMC flocculant

TABLE-1: COMPARATIVE REMOVAL EFFICIENCY OF CARBOXYMETHYL CELLULOSE

	SAMPLING POINTS	TURBIDITY REMOVAL (%)	COD REMOVAL (%)	TDS REMOVAL (%)	ELECTRICAL CONDUCTIVITY REMOVAL (%)	OVERALL REMOVAL EFFICIENCY (%)
Month-1	1	72.3	65.30	54.02	51.30	60.73
	2	72.2	59.01	52.20	53.10	59.10
	3	79.3	51.30	54.45	53.00	59.51
	4	74.3	62.30	50.20	50.80	59.40
	5	74.5	55.98	50.00	50.50	57.74
Month-2	1	77.3	64.90	54.10	50.50	61.70
	2	72.9	58.80	52.54	53.00	59.31
	3	78.3	55.30	55.11	52.80	60.37
	4	76.1	66.10	46.67	50.40	59.81
	5	75.9	54.36	51.47	49.80	57.88
Month-3	1	73.3	62.10	47.64	50.46	58.38
	2	73.3	57.10	54.87	53.10	59.59
	3	79.1	53.20	52.50	53.06	59.47
	4	73.1	65.01	48.70	50.96	59.44
	5	77.1	52.10	49.60	49.82	57.16

### 3.6 Effect of CMC on the Reduction of Conductivity

Conductivity of wastewater samples increased along with increase in pollution load. In this study, the reduction of conductivity work was performed against use of carboxymethyl cellulose as flocculant. In preliminary analysis of location-1 samples, conductivity was recorded as 945, 989 and 965  $\mu\text{S}/\text{cm}$  respectively. After treatment it was reduced as 51.30%, 50.50% and 50.46%. In location-2, conductivity removal rates were 53.1%, 53.0% and 53.1%. In location-3, conductivity removal rates were observed as 53.00%, 52.80% and 53.06%. In location-4, conductivity removal rates were observed as 50.80%, 50.40% and 50.96%. Finally in location-5, removal rates of conductivity were observed as 50.50%, 49.80% and 49.82%. Carboxymethyl cellulose as organic flocculant was effective in reducing conductivities of industrial wastewater samples. The average conductivity removal efficiency rates were up to 53%. Fig-8 shows the electrical conductivity removal efficiency in use of CMC.

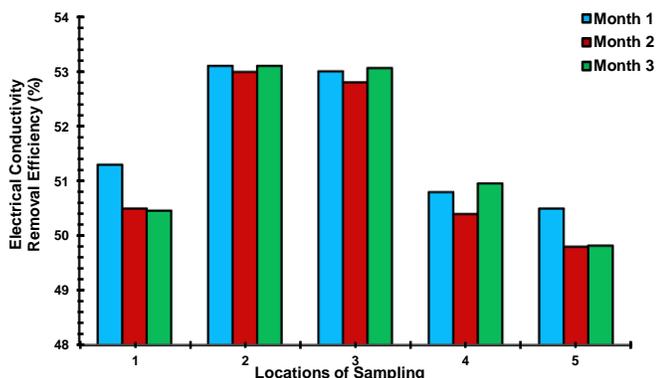


Fig-8: Electrical conductivity removal efficiency in use of CMC

### 3.7 Comparative Study for Pollutants Reduction in use of CMC

From the experimental work, it was found that prepared organic flocculant was effective in the reduction of wastewater pollution load. In preliminary stage, optimum dose of CMC was observed. On that basis, all samples were treated according to optimum dosing. The removal efficiency of CMC was obtained approximately 60% for concerned effluent pollutants. CMC could reduce turbidity, COD, TDS and conductivity effectively. Table-1 summarizes the comparative removal efficiency of carboxymethyl cellulose.

### 3.8 Study of Adsorption Isotherms

The study of adsorption isotherms in a medium are meant to measure quantitatively the concentration of the sorbate and sorption at temperature of 27°C. Research on isotherm suggests liquid/solid sorption with regard to its sorption capacity, behaviour and interaction of sorbate and sorbent [2]. As Langmuir isotherm is considered to cover monolayer of the adsorbate with homogeneous surface of adsorbent. Isotherms of Freundlich suggested presence of heterogeneity of the adsorbent surface [2]. When isotherms models were tested for the determination of adsorption behavior and mechanism, both models showed higher values (0.969) of  $R^2$ .

TABLE 2

LANGMUIR AND FREUNDLICH CONSTANTS FOR EFFLUENT POLLUTANTS ADSORPTION USING CMC

Langmuir Isotherm Model		Freundlich Isotherm Model		
b (L/mg)	$R^2$	K (mg/g)	n	$R^2$
4.487	0.969	$3.67 \times 10^7$	0.4925	0.919

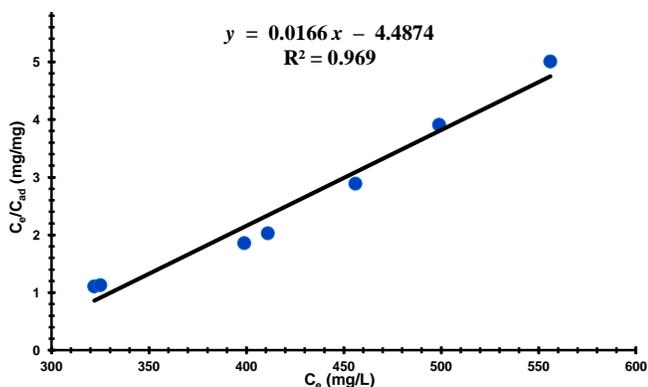


Fig-9: Langmuir Adsorption Isotherm for Effluent Pollutants Reduction using CMC

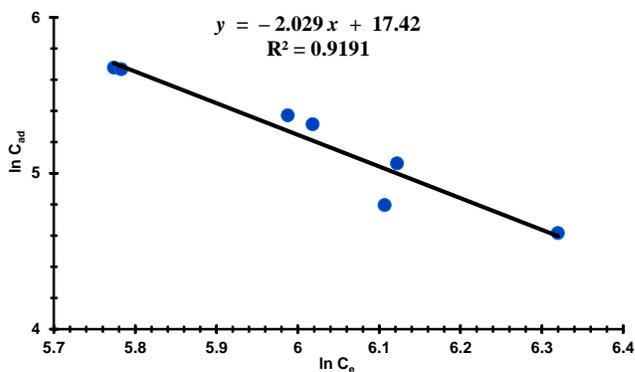


Fig-10: Freundlich Adsorption Isotherm for Effluent Pollutants Reduction using CMC

Fig-9 graphically shows Langmuir adsorption isotherm for effluent pollutants reduction using CMC. Similarly Fig-10 shows Freundlich adsorption isotherm for effluent pollutants reduction using CMC. From statistical analysis, Langmuir isotherms are good for adsorption of industrial wastewater contaminants. There is monolayer coverage of the pollutants on a homogenous surface of CMC. The Langmuir and Freundlich model's constants were found out from their model's equation calculations and were shown in Table-2.

## 4. CONCLUSIONS AND RECOMMENDATIONS

### 4.1 Conclusions

From experimental work and industrial wastewater analysis, it was concluded that wastewater of Hyderabad industrial SITE area is toxic and polluted. Industries are not treating wastewater before its disposal. It has high toxic effect on the marine life. Effective treatment is needed and wastewater should be treated according to Pakistan Environmental Protection Agency (EPA-Pakistan) regulations. EPA-Pakistan is an attached department of the Ministry of Climate Change and responsible to implement the Pakistan Environmental Protection Act, 1997 in the country [5].

Low cost and effective natural organic flocculant carboxymethyl cellulose prepared from agricultural waste of cotton stalk using physico-chemical treatment. This flocculant was used for the reduction of wastewater pollutants such as turbidity, COD, TDS and conductivity. In this study, flocculant optimum dose (70 mg/L and pH-7) was found most effective for wastewater treatment. On increasing flocculant dose, removal efficiency was observed. At some extent, steady state condition was observed in pollutants reduction. The overall efficiency of CMC was recorded up to 60%. It was finally concluded that prepared flocculant was effective for the removal of pollutants from industrial wastewater.

### 4.2 Recommendations

1. Low cost flocculant CMC shall be prepared from agricultural waste of cotton stalk and used for low contaminated wastewater treatment.
2. Industrial wastewater must be treated properly using effective treatment technique according to EPA regulations

before its disposal to river.

3. Monitoring on regular basis by Government functionaries should be established for improvement of the environment.
4. Force the industrialists to educate their workers to adhere to protect industry environment.
5. To make CMC flocculant mandatory for pre and post stage effluent treatment to curtail sludge volumes.

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