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# Carboxymethyl Cellulose Based Zinc Oxide Nanocomposites: Synthesis, Characterization and Dye Removal Applications

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**Abstract:** Carboxymethyl cellulose/ZnO has been used for the design of composite hydrogel in order to obtain effective 3D hydrogel network structures for industrial waste water treatment applications. In this present work, dye removal CMC/ZnO composites were prepared by sol gel method. The prepared composites were characterized by FT-IR, XRD, SEM and TEM. The swelling studies have also been evaluated at different pH solution. The obtained results have shown composite hydrogel possesses excellent dye removal compared to raw material. The maximum dye adsorption equality has been found in the case of CMC/ZnO hydrogel. Therefore CMC based CMC/ZnO composites hydrogels could be applied effectively for removing cationic dyes from waste water. This kind of materials has been used for various industry and factory wastewater treatment.

**Keyword:** Carboxymethyl cellulose, dye removal, swelling

## I. INTRODUCTION

One of the serious issues concerning textile wastewater is colored effluent [1]. The natural colors from textile industries, paper, pharmaceutical, printing, tannery, and so forth are viewed as a wide assortment of natural poisons to the wastewater treatment systems [2]. Numerous azo dyes and their intermediates affect condition and human well being because of their cancer-causing nature and visibility. Along these lines, it is fundamental that wastewater is polluted with Sapphire Blue dye color to be treated before discharge [3].

The released wastewater from the textile industries contains unavoidable and higher contamination of dyes. The intensity of contamination depends on the texture, just as colors and chemicals utilized in textile industries, their obsession rate on the texture. Close to that the coloring grouping, coloring hardware and the alcohol proportion utilized in the material ventures can likewise influence the contamination force [4]. As it may, discharge this sort of color such as Sapphire blue (SB) into the amphibian condition results in different tricky issues, for example, hindrance the daylight entrance into the oceanic media, just as danger and cancer-causing nature of these dyes [5].

Various conventional strategies are available for textile wastewater treatment it can be classified as biological, chemical and physical treatments. The most productivity in the removal of colors toxins is adsorption system because of the methodology effortlessness, simplicity of system, task, and its non-affectability to lethal materials [6].

Metal oxide nanoparticles are a flexible material with numerous logical and mechanical applications [7]. In the most recent decade, nanocomposite improves the nanosize materials of metal and metal oxide particles are seriously sought after on account of their noticeable quality in various fields of utilizations. There are number of metal oxides are accessible in nature yet a portion of the metal oxides are most valuable as per their applications in everyday life in science and technology [8]. Hydrogels are characterized as hydrophilic polymers which, due to their auxiliary system, can retain water without being solvent under physiological states of temperature, pH, and ionic quality. Crosslinks can be framed by covalent, electrostatic, or on the other hand hydrophobic bonds, or dipole-dipole co-operations, hydrogels are a class of polymers that can ingest water or natural liquids and swell a few times their dry volume. Reliance of the swelling conduct of the hydrogel on changes in the outside condition was accounted. The cost of free-space between the systems in the swollen organize that serve for nucleation and development of nanoparticles and act as nanoreactors or nanopots [9].

Zinc oxide (ZnO) is a n-type semiconductor, is an extremely intriguing multifunctional material for its promising applications such as, semi conductor diodes, ultraviolet protection films, impetuses, sensors, earthenware production and sun oriented vitality transformation. Zinc oxide is generally utilized in different applications, such as, gas sensors, sunlight based cells, antireflection

coatings, varistors, surface acoustic wave gadgets, light transmitting diodes and arbitrary lasers [10]. Among various handling strategies, sol-gel is considered as the most adaptable techniques, due to low sintering temperature and great homogeneity at low cost [11].

Carboxymethyl cellulose (CMC) or cellulose gum or tylose powder is a cellulose subordinate with carboxymethyl bunches (-CH<sub>2</sub>-COOH) bound to a portion of the hydroxyl gatherings of the glucopyranose monomers that make up the cellulose spine. Usually used as sodium salt, sodium carboxymethyl cellulose. It is used as a mass diuretic, emulsifier, thickener in beautifying agent, pharmaceuticals and as a stabilizer for reagents. CMC-based hydrogels experience the ill effects of poor mechanical quality. A standout amongst the most encouraging approaches to enhance the mechanical properties of CMC-based hydrogels is join polymerization of acrylic acid, acrylamide and their copolymers onto CMC chains [12]. Acrylamide (or acrylic amide) is also included as a natural compound. Polyvinylpyrrolidone, additionally named Povidone or PVP, is a nonionic amphiphilic polymer. Due to its excellent mixing of properties like non toxic quality, biocompatibility, film framing and glue qualities, complex capacity to proton benefactors, and low osmotic weight, it finds differing applications in different fields of life like pharmaceutical and restorative items, sustenance, cements, and material assistants as contrasted with other water-solvent polymers like polyethylene oxide and polyvinyl alcohol [13].

In this investigation the removal of sapphire blue dye with ZnO nanoparticles by using sol gel method by batch adsorption process. CMC hydrogel nanocomposite was prepared by using Sodium Carboxymethyl cellulose (Na-CMC), Acrylamide (AAm), Polyvinylpyrrolidone (PVP) and ZnO nanoparticles was used to remove Sapphire blue dye from aqueous solutions, for the first time. Swelling behavior and adsorption properties of the hydrogel nanocomposite in water and dye solutions were also investigated. The synthesized hydrogel nanocomposite sample was further characterized by FT-IR, XRD and morphology of nanostructure was investigated by SEM & TEM. The removal of sapphire blue dye was observed a wide range of dye concentrations and dosage of sample [14].

## II. EXPERIMENTAL METHODS

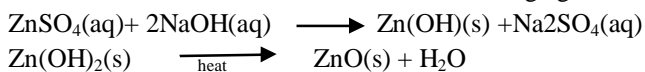
### A. Materials Used

All chemicals including Carboxymethyl cellulose sodium salt (Na-CMC), Acrylamide (AAm), Polyvinylpyrrolidone (PVP) were purchased in market. Zinc sulphate powder was used for further synthesis of zinc oxide nanoparticles. The distilled water is used throughout the reaction.

### B. Methods

1) *Synthesis OF ZnO Nanoparticles:* (ZnONPs) were synthesized by precipitation technique. In this technique, (ZnONPs) were set up in two different ways. In the set, 100 ml of 1M ZnSO<sub>4</sub> has been added to 100 ml of 2M NaOH arrangement in drops. At the point when the expansion was finished, the mixture was kept at room temperature under constant mixing by using stirrer for a time of 2-4 hours. The steady mixing utilizing magnetic stirrer made the precipitation homogeneous. The negligible particles which diminish the particular surface free vitality of precious stone core. The hindrance leads to agglomeration and development of the gem core so the molecule size of the item is decreased.

The collected precipitate acquired was separated and washed with distilled water. The formed white precipitate of Zn(OH)<sub>2</sub> was permitted to settle, filtered using filter paper of pore measure 0.4 μm in a suction siphon and washed with distilled water a few times and dried in hot oven at 150°C for 45 minutes. The incorporated (ZnONPs) were additionally lighted at 180 W with microwave radiation in a microwave for 30 minutes. The above methodology was pursued to mix (ZnONPs) in various test conditions. ZnSO<sub>4</sub>, NaOH and oxalic acid were utilized as stabilizing agents. The precipitation response was obtained as



The resultant (ZnONPs) particles after irradiated were gathered and put away in dark colored jugs. The synthesized (ZnONPs) were exposed to powdered XRD and FTIR so as to confirm the nanostructure [15].

2) *Preparation Of Nanocomposite:* New superabsorbent nanocomposite hydrogel was synthesized by free-radical polymerization by using Carboxymethyl cellulose (CMC), Acrylamide (AAm), Polyvinylpyrrolidone (PVP) and ZnO nanoparticles. A series of three different monomers CMC, AAm and PVP were mixed in 20 ml of distilled water in different concentration in a beaker. Then different concentration was mentioned in the below table. The above mixture are continuously stirred for 1 hr at 100°C until a homogeneous viscous mixture was obtained. Then ZnO nanopowder is incorporated in it and stir it for 1 hr at 100°C in

hot plate. Keep the specimen in oven at 90°C for 1 hr. The formed paste, was transferred to Petri dish, dried in an oven at 90°C, then the formation of white color gel is obtained. Solgel is dried for 24 hrs and further it is washed in distilled water several times. The obtained sample has been kept it in oven at 50°C for 1 hr. Adsorbent is formed and preserved for further testing.

Table 1. Feed composition of CMC based adsorbent

S.NO	Sample code	Volume (ml)	CMC (gm)	AAM (gm)	PVP (gm)	ZnO (gm)
1	CAP1	20	0.50	0.50	0.50	0.25
2	CAP2	20	0.50	0.20	0.80	0.25
3	CAP3	20	0.50	0.40	0.60	0.25
4	CAP4	20	0.50	0.60	0.40	0.25
5	CAP5	20	0.50	0.80	0.20	0.25

3) *Preparation Of DYE Solution:* Sapphire blue color (SB) is considered as an ecological pollutant was utilized in this research .Stock arrangements at (0.05-0.3g/L) of color arrangement were set up by dissolving the required amount in distilled water [3].

### III. RESULT AND DISCUSSION

#### A. Swelling Behaviour

The swelling properties of prepared nanocomposite hydrogel 0.1 g of dried hydrogel test was submerged in 100 ml distilled water for 24 h at room temperature. The swollen hydrogel was removed from the distilled water at certain time interval and weighed after removing from distilled water. The equilibrium swelling proportion  $S_{eq}$  % was determined using the accompanying condition.

$$S_{eq}(g/g) = \frac{W_s - W_d}{W_d} \longrightarrow 1$$

Where  $S_{eq}(g/g)$  is equilibrium swelling ratio,  $W_s(g)$  is the weight of the swollen adsorbent, and  $W_d(g)$  is the weight of the dried adsorbent [12].

1) *Effect of Time ON Swelling Behavior:* To investigate the swelling effect of time on various composition of hydrogel was carried out at different ranges from 30-150 minutes. So the maximum time is observed in 150 minutes for best swelling capacity. The adsorbent have fast swelling rate and high swelling ratio due to the high porosity and interconnection among some of the pores within the polymeric network [12].

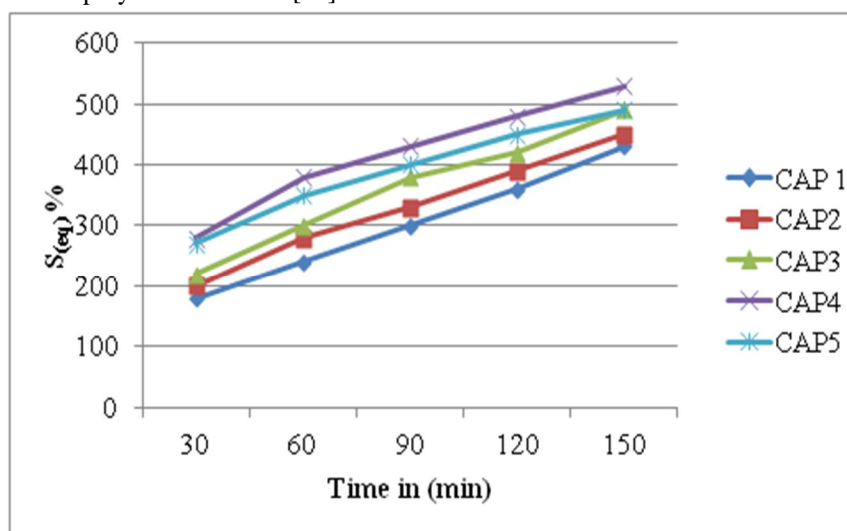


Fig.1 Swelling equilibrium of adsorbent at different time

The above series (CAP1-CAP5), in which CAP4 posses better swelling in nature based on the composition (CAP4) nanocomposite hydrogel .This result indicates when the time increases , Swelling behaviour also increases due to hydrophilic nature adsorbent attain maximum swelling when the time increases. It is concluded when the time increases from 30-150 min  $S_{eq}$ % has also been increased in fig.1.

2) *Effect of pH on Swelling Behaviour:* The pH sensitivity of nanocomposite hydrogels in various acidic and basic solutions with different pH values ranging from 2 to 9 were prepared by diluting 0.1M aqueous solutions of NaOH and HCl, 0.1 g of dried nanocomposite has been immersed in solutions with different pH values. The pH 9 indicates the best for swelling ratio results. In general swelling is greater in higher pH than lower pH due to hydrophilic nature of adsorbent expanded at higher pH when the pH is increases ,swelling also increases as shown in fig.2. Thereafter, the equilibrium swelling ratio is directly proportional to the pH. [12].

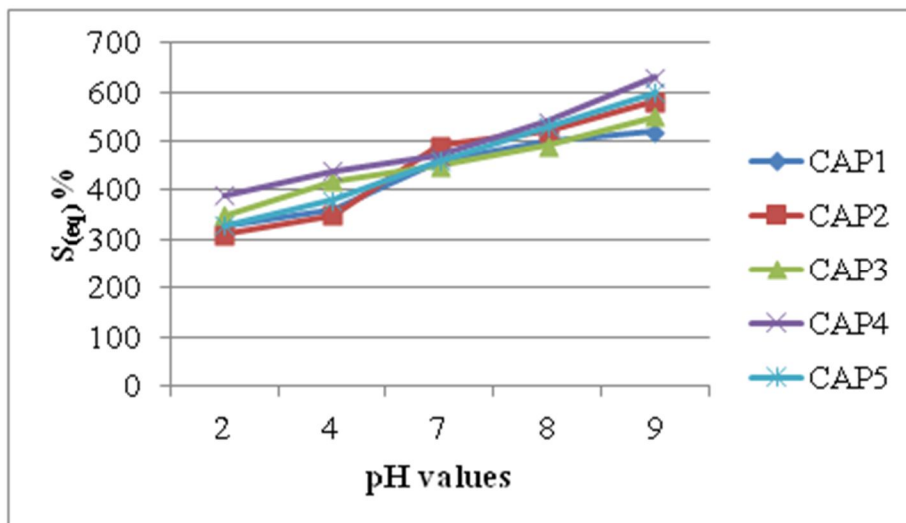


Fig.2 Swelling equilibrium of adsorbent at different pH

### B. Adsorption Studies

Sapphire blue dye (SB) adsorption experiments was conducted in order to determine the productivity of hydrogel adsorbent and the impact of controlling parameters like pH, contact time, dosage and introductory focus. The stock solution of 100 mg/L of color was set up by dissolving its 0.1 g in 1000 mL distilled water. All adsorption tests were completed in a 250 mL conical flask with 10 mL of dye solution at 32°C at 120 rpm using an orbital shaker at pH 7. Batch adsorption were completed by different experimental parameters such as, pH, contact time, initial concentration, and adsorbent dosage. Batch experiments was performed in triplicate for exactness. The concentration of dye after adsorption was observed with the help of calibration curve. The calibration curve was constructed by running standards of the dye on UV-Visible spectrophotometer at  $\lambda = 600 \text{ nm}$ .

$$\% \text{ of Removal} = \frac{(C_0 - C_t)}{C_0} \times 100 \quad \longrightarrow \quad 2$$

where  $C_0$  and  $C_e$  ( $\text{mg L}^{-1}$ ) are the concentrations of dye before and after adsorption. The amount of Sapphire blue adsorbed at equilibrium,  $q_e$  ( $\text{mg/g}$ ), was calculated using the following equation.

$$q_e = \frac{(C_0 - C_e)V}{M} \quad \longrightarrow \quad 3$$

where  $C_0$  and  $C_e$  ( $\text{mg/l}$ ) are the initial and equilibrium concentrations of Sapphire blue, respectively,  $V$  (l) is the volume of the Sapphire blue solution and  $M$  (g) is the adsorbent mass [5].

$C_0$ -Initial concentration

$C_e$ -Final concentration

### C. DYE Adsorption Analysis

Sapphire blue dye was adsorbed by CAP 4 nanocomposite hydrogel sample. The CAP 4 has been chosen as a better adsorbent due to excellent swelling behavior at different batch experiment [14].

### D. Batch Experimental Conditions

1) *EFFECT OF Ph:* The pH of dye solution plays an important role in the whole adsorption process, particularly on adsorption studies. Due to the significant influence of pH of adsorption media on the efficiency of adsorption process, we have investigated the influence of the initial pH of the solution and decreases removal efficiency than higher pH. The maximum dye adsorption has been observed at pH 7, the maximum decolorization to be 89.43% as shown in the fig.3 [5].

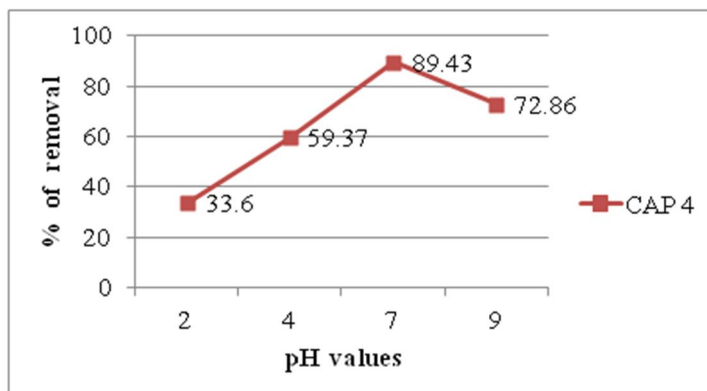


Fig.3 Effect of pH on adsorption of dye

2) *Effect Of Contact Time:* The effect of contact time on dye adsorption as shown in figure 4, As the contact time increased, the adsorption capacity ( $q_e$  mg/g) of adsorbent also increased at a time interval for 15-90 (min). Effective decolorization was found to be achieved at 90 min. The maximum decolorization percentage was found to be 77.04% at a time period for 90 minutes. Therefore, 90 min is considered as the optimum contact time for the adsorption. Contact between the adsorbent and adsorbate increases the probability of the adsorption and it increases with longer contact time. So as the time increases adsorption also increases due to adsorption attains maximum and saturated at equilibrium.[5].

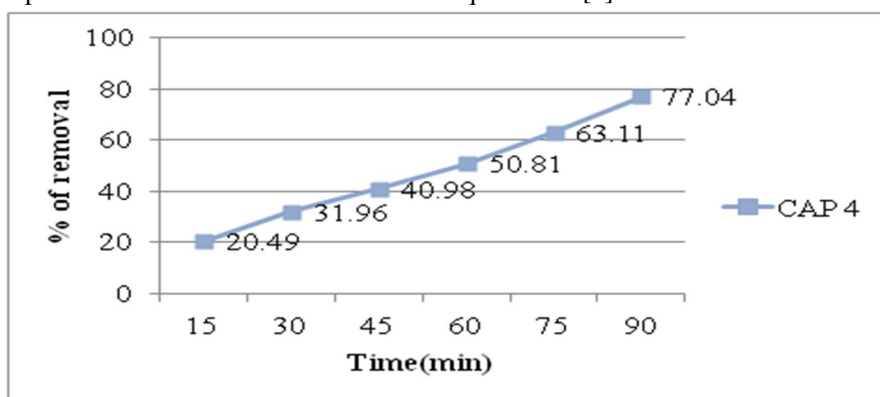


Fig.4 Effect of time on dye removal percentage

3) *Effect of Initial Dye Concentration:* The effect of increasing dye concentration on adsorption capacity is shown in fig.5. The inhibitory concentration of dye varies from 0.05 to 0.2 g/L. Initially the concentration of dye value is increased then the percentage of removal is decreased because of high concentration of dye solution contains higher dye molecules compared to low concentration. So adsorption is very high at low concentration dye solution. So the minimum dye concentration was found to be 0.05 g of dye is the best for removal efficiency and the removal of percentage is 87.5% [5].

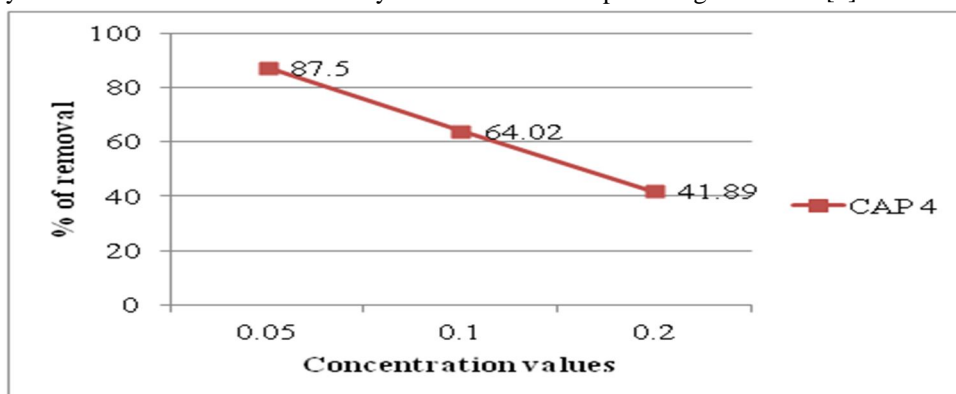


Fig.5 Effect of dye concentration on removal percentage

4) *Effect OF Dosage*: The effect of dosage of superadsorbent nanocomposite hydrogel was varied from 0.05-0.3gm/L. It was observed that the nanocomposite dose increases, the percentage of removal of dye also increased as shown in fig.6. Hence 0.3g/L of nanocomposite hydrogel has been considered as an optimum dosage and the removal percentage is 74.21% .Due to large amount of dosage posses better efficiency. And also the maximum removal percentage has been obtained at higher dosage vacancy site available at higher [5].

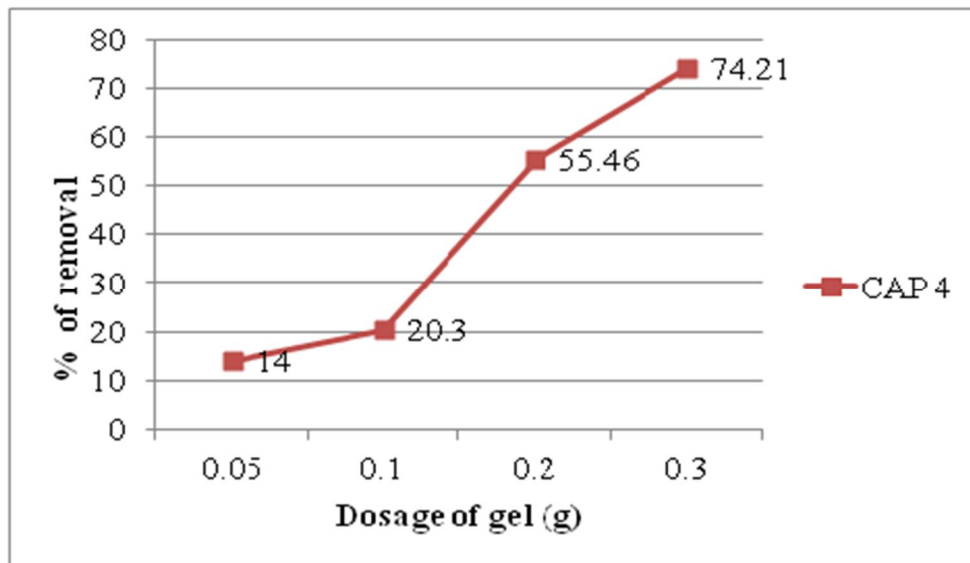


Fig.6.Effect of dosage of gel on dye removal percentage

*E. Characterization*

1) *FT-IR Spectral Studies*: Characterizations of FTIR spectra of CMC-AAm-PVP hydrogel nanocomposite were acquired in the wave number range of 4000– 500 cm<sup>-1</sup>,The FT-IR spectra of CMC barred hydrogel or adsorbent as shown in the figure7.The peaks of CMC has been observed at 3275.13 cm<sup>-1</sup> (O-H string) 2918.30 cm<sup>-1</sup> (C-H ) methylene bend, 1585.49 (C=O) amide,1018.41 (C-OH)stretch frequency.

In the CMC/AAm/PVP nanocomposite a peak at 3350.35 cm<sup>-1</sup> (OH string) due to the vibration of CMC is observed which indicates that CMC introduced into the nanocomposite. To identify the possible functional compound on adsorbent involved in the dye FT-IR spectrum observed before and after dye adsorption.

There is a slight change or shifting of adsorption of dye on the surface of peak at 3350.35, 2954.95, 1423.47, 563.21 cm<sup>-1</sup> adsorbent due to 3269.34, 2897.08, 1419.61, 543.93 cm<sup>-1</sup> for dye adsorption [16]

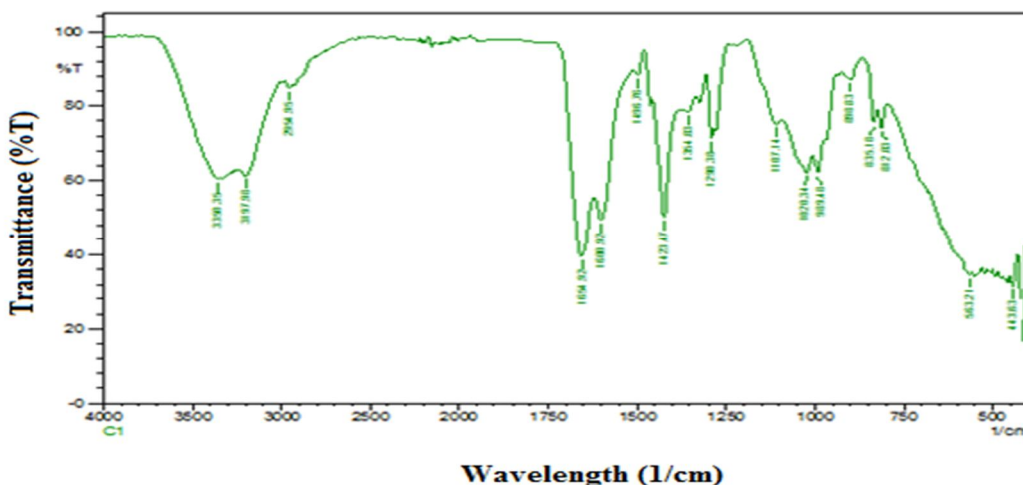


Fig. 7.a. FT-IR spectrum of CMC adsorbent before adsorption of dye

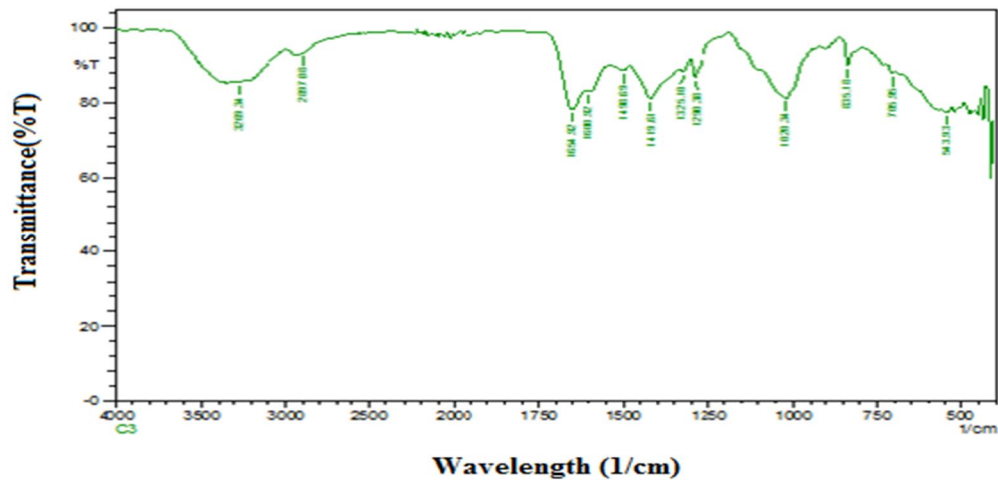


Fig.7.b. FT-IR spectrum of CMC adsorbent after adsorption of dye

- 2) *SEM Analysis:* The SEM images of adsorbent has shown the rough and cracked surface morphology provides an excellent adsorption sites as shown in the figure 8a. It has been observed that surface of the adsorbent after adsorption changed and the small cavities has been disappeared on incorporated nanoparticles in figure 8b.

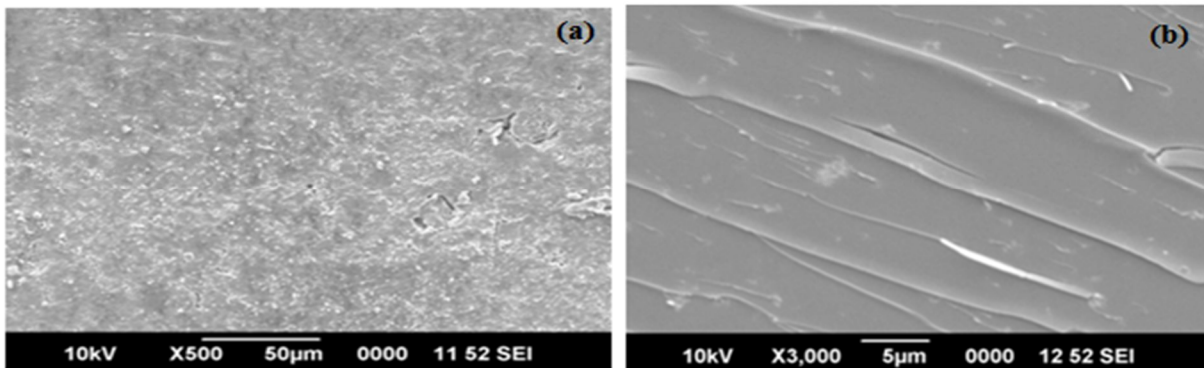


Fig.8 SEM images of adsorbent (a)Before adsorption of dye (b) after adsorption of dye

- 3) *TEM Analysis:* The dispersion of ZnO nanoparticles has been studied by TEM and the images of CAP 4 in the figure 9. As per these images the ZnO nanoparticles in hydrogel have spherical or thread like structure and the size of nanoparticles obtained lower than 1nm.

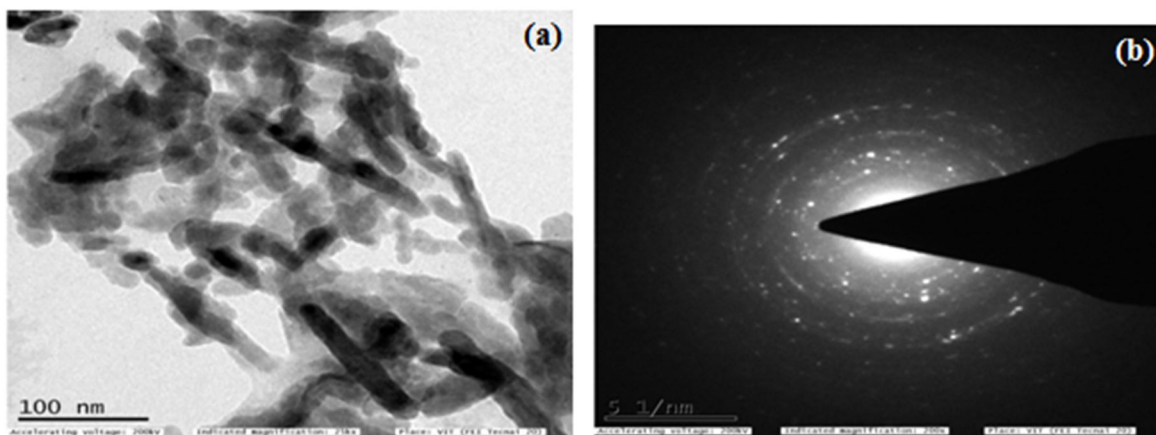


Fig.9 TEM images of CMC nanocomposite

- 4) **XRD Analysis:** In fig 10, shows that two peaks appeared in the CMC /ZnO near to the  $2\theta$  angles of  $14.8^\circ$ ,  $16.2^\circ$ ,  $24.2^\circ$ ,  $34.2^\circ$  which are characteristics of the (101), (101), (002) and (040) reflections of CMC. The remaining peaks were cellulose crystalline type on compared with other composite type. On compared with other composites peak of ZnO the diffraction peak of CMC are very weak, which could be attributed to ZnO growing on the surface of CMC in the CMC /ZnO which makes it harder to collect the diffraction data of CMC during the XRD analysis.

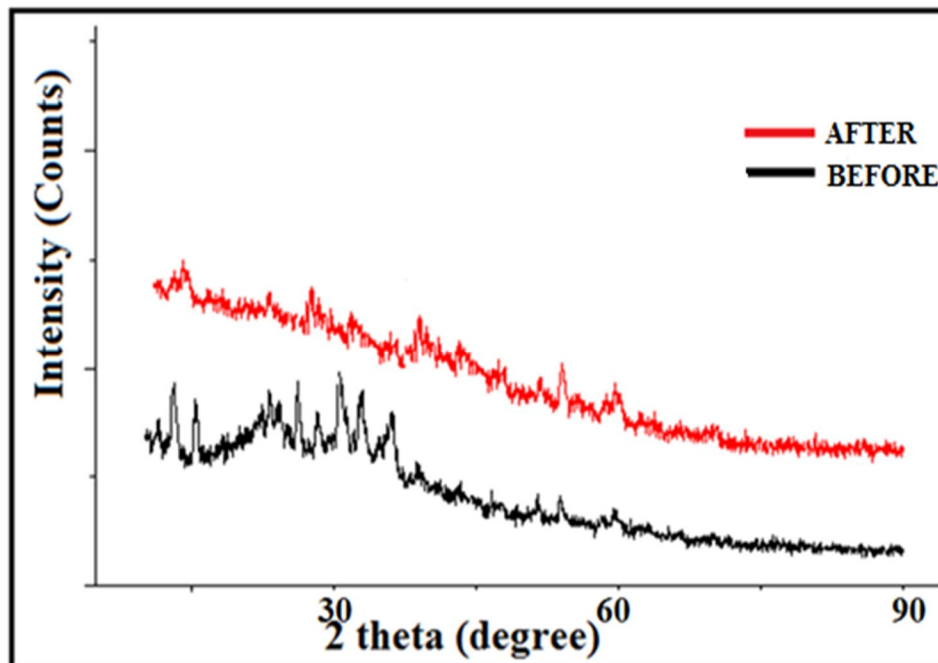


Fig.10 XRD pattern of CMC nanocomposite

#### IV. CONCLUSION

In this investigation CMC/ZnO based composites were synthesized and optimized for removing of Sapphire blue dye (SB). The synthesized were characterized using various characterization such as FT-IR, SEM, TEM and XRD. The ability of SB removal was evaluated under various parameters. The results revealed the maximum SB removal is 89.43% is obtained, it could be concluded as CMC/ZnO adsorbent for SB dye from waste water. This kind of composites act as an excellent adsorbent for waste water treatment.

#### V. ACKNOWLEDGEMENT

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