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Synthesis and Characterization of Chitosan based Catalyst for Catalysis Applications

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ABSTRACT

In this research, chitosan based catalyst synthesis and characterization was investigated for different application in catalysis as chitosan based catalyst was combined with metal oxides in order to be used in dye removal of methyl orange (MO). Diluted acetic acid was used to dissolve chitosan then different percentages for metallic ions were mixed with diluted chitosan and then, 500 µL were added to the sample to reduce the metal salts then, microwave was used to heat the mixture for 5 minutes and then dried at 80-100 °C. Finally, X- ray diffraction was used to confirm the catalyst prepared. The aim of this research is to investigate and explore the feasibility of chitosan/metallic ions composite for removing MO from aqueous solutions. The influence of several operating parameters for adsorption of MO, such as contact time, temperature and pH.

Key words: Dye removal, Chitosan, Metal oxides, catalyst, Wastewater treatment.

1. INTRODUCTION

This research main aim is to investigate the preparation and characterization of chitosan based catalyst for different catalytic applications. [1-7] It is also oriented to explain the performance of chitosan catalyst in several catalytic applications like dye removal in order to remove dyes from textile and wastewater treatment. [8-14]
Metal oxides are used in both types of heterogeneous and homogenous catalysis. Heterogeneous catalyst is better than homogeneous catalyst as it could be separated from reaction mixture more easily. [15-22] Chitosan is a natural polysaccharide with appealing intrinsic properties, such as, non-toxicity, biocompatibility and biodegradability. [23-31] Moreover, chitosan displays a powerful adsorption performance toward dyes from aqueous solutions. [32-40] It is massively applied in the removal of organic dyes and metal ions However, pure chitosan as an adsorbent has several disadvantages, including high cost and low chemical stability, which limits its application in adsorption processes. [41-45]
The research focused recently on investigating new methods of eliminating polluted dyes from industrial wastewaters was highly interested. Azo dyes appear to be the most effective among different types of the dyes used, because these dyes are widely used in many textile, food and color paper industries. Azo dyes usually have a complex aromatic structure and nearly complex structural azo(-N= N-) groups. [46-50] These colors are deeply colored because the azo-groups in these colours, and therefore the color can go away if these groups are broken. [51-54]Because these compounds have a rigid structure, it is not easy to break these materials into smaller fragments in normal condition.
This type of dyes can cause many types of environmental pollution including air, water and soil pollution by the presence of these dyes in wastewater. [55-58] Some of these teeth can also produce carcinogenic materials and/or release certain toxic substances into the environment. In general, certain physical, biological and chemical methods involve traditional methods of removing dyes from textile industrial wastewaters. Recently, methods for photodegradation seem to be an interesting alternative approach which can efficiently be used to remove these dyes from textile effluents. [59-62]
Because of its clear and distinct colour change, methyl orange is a pH indicator commonly used in titrations. Since the color changes at the pH of a mid-force acid, it is typically used in acid titrations. In contrast to a universal indicator, methyl orange does not change color in its entirety, but it has a stronger final point. In a less acidic solution, methyl orange moves from red to orange and finally to yellow with the reverse resulting in an increased acidity solution. In acidic conditions the entire color change occurs. It is reddish in acid and yellow in alkaline. [63-64]
Methyl orange moves from red to orange in a solution which is less acidic and finally becomes yellow in order to increase its acidity. In acidic conditions the entire color change takes place. It's reddish in an acid and yellow in alkaline. [64-66]

2. EXPERIMENTAL

Solubility of chitosan was investigated using three different solution which are (water, acidic acid and diluted acidic acid) to find out that the optimum solution that have the ability to dilute the chitosan is diluted acidic acid mixture within 1-2% in distilled water and with the presence of stirring and heating using hot stirrer.

Different percentage of chitosan were prepared with metal ions such as (Cu, Ni, Fe,Co). Then 500 µL of Hydrazine hydrate were added as a strong reducing agent to the mixture of chitosan and metal ions then heated using microwave for 5 minutes. Then, sample was dried using oven and catalysts was collected for being used in the process. Then, 50 ppm concentration of methyl orange day is prepared in a diluted distilled water. The wavelength of 50 ppm of methyl orange was checked to be 465 nm. After preparing day samples a standard curve have to be drawn to study the relation between absorbance with the concentration of dye by the catalysts prepared. To assure accurate standard curve different concentrations were used such as [35, 30, 25, 20, 15, and 10] as a trial with constant volumes. After getting the mass and volume required using the following equation mv = mv standard curve is done. Using the extracted catalysts as every portion is divided by five to be used for all dye samples. Catalysts absorbance is calculated over different time intervals as follow [10, 20, 30, 40, and 50] minutes using 50 ppm concentration of dye. Prepared samples after that are poured into cylindrical flasks to be diluted with distilled water to rich total volume of 50 ml. Finally, portions for each sample are inserted into the spectrophotometer to know the rate of absorbance of the catalyst over dye concentration for each sample.

3. RESULTS AND DISCUSSION

Figure 1 shows the effect of reduction after adding 500 µL Hydrazine hydrate on solution of chitosan and copper and on the sample 0.2 chitosan the sample has changed the color because it has a little amount of chitosan so the reduction come fast before entering the microwave.

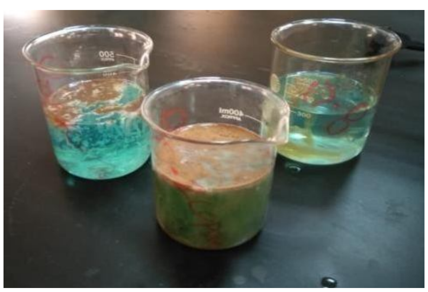

Figure 1: Effect of adding Hydrazine hydrate on copper and chitosan solution

Figure 2: Effect of adding Hydrazine hydrate on Nickel and chitosan solution

Figure 2 shows the effect of reduction after adding 500 µL Hydrazine hydrate on solution of chitosan and nickel and we also see here that the color changed fast on the sample 0.6 ,0.4 and 0.2 g from chitosan and this indicator that the reduction is done.

Figure 3: Effect of adding Hydrazine hydrate on Iron and chitosan solution

Figure 3 shows the effect of reduction after adding 500 µL Hydrazine hydrate on solution of chitosan and ferric on the sample 0.4 and 0.6 gram from chitosan and the color changed.

Table 1: Concentration Calculations for Iron based catalyst supported on different wt % Chitosan

Chitosan	Ferric	Resulted
0.9	0.2945	**1.2**
0.8	0.589	**0.82**
0.6	1.178	**1.67**
0.4	1.767	**2.2**
0.2	**2.356**	**2.02**

Figure 4: Chitosan Based Iron Oxide Catalyst

Table 2: Concentration Calculations for Copper based catalyst supported on different wt % Chitosan

Chitosan	Copper	Resulted
0.9	0.2167	**0.8**
0.8	0.433	**0.8**
0.6	0.8667	**1.1**
0.4	1.3	**1.24**
0.2	**1.733**	**1.58**

Figure 5: Chitosan Based Copper Oxide Catalyst

Table 3: Concentration Calculations for Nickel based catalyst supported on different wt % Chitosan

Chitosan	Nickel	Resulted
0.9	0.2621	**0.91**
0.8	0.525	**1.08**
0.6	1.05	**2.1**
0.4	1.576	**1.8**
0.2	**2.101**	**1.7**

Figure 6: Chitosan Based Nickel Oxide Catalyst

Figure 7 display the XRD diffraction pattern of copper chloride nanoparticles which was prepared with the microwave method, and the characterization of (CuCl) was achieved by XRD pattern of catalyst sample as we see in figure 7. XRD of CuCl match that reference code is 01-081-1841 corresponding to cubic structure and the diffraction peaks are ascribed to the (111), (200), (220), (311), (222), and (400).

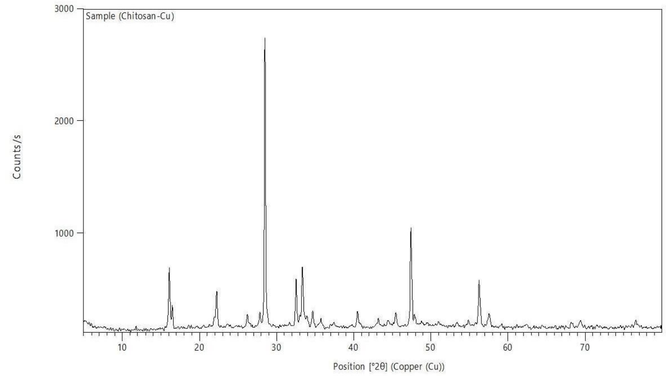

Figure 7: XRD pattern of Copper based catalyst

Figure 8 display the XRD diffraction pattern of iron chloride nanoparticles which we prepared with the microwave method. And the characterization of $[FeCl_2(H_2O)_4]$ was achieved by XRD pattern of catalyst sample as we see in figure 8. XRD of $[FeCl_2(H_2O)_4]$ match that reference code is 01-071-0917corresponding to Monoclinic structure and the diffraction peaks are ascribed to the (100), (001), (110), (012), (111), (120), (222), (231) and (024).

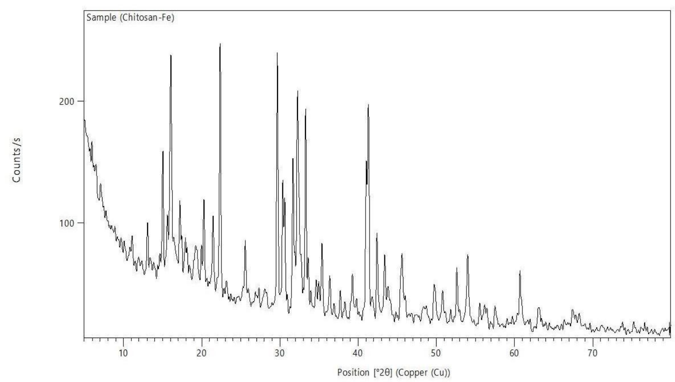

Figure 8: XRD pattern of Iron based catalyst

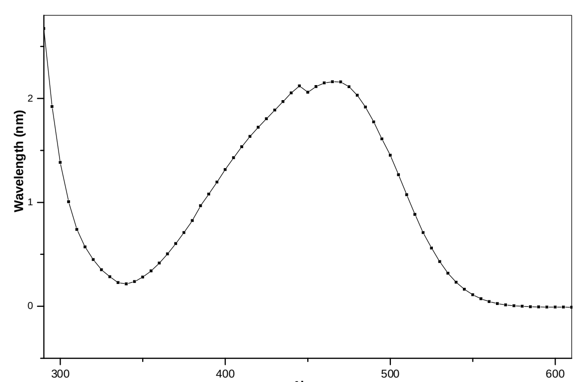

Figure 9: Wave Length Curve of Methyl Orange

In order to find the wave length of the methyl orange this curve was obtained by using the Spectrophotometer by applying the dye (methyl-orange) in the device and start to gain the reading of the wave length curve it showed that peak of the curve was 465 nm which fits with the known wave length of methyl orange in the books done by scientist.The methyl orange has been prepared with concentration 50 PPM by applying the equation MV=MV to obtain the needed concentration

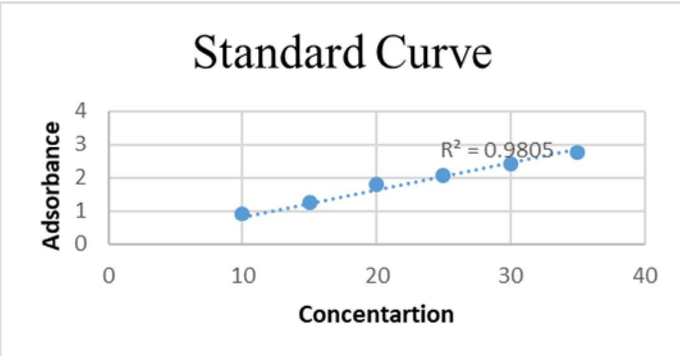

Figure 10 : Standard curve for Methyl orange

From the curve, it is evident that λ_{max} was determined at a value of = 465 nm, which is in accordance with values in literature. For practical purpose, the wavelength will be fixed at 465 nm whilst carrying out all absorbance measurements of both the standard curve and the subsequent sample determinations.

The standard curve shows the relation between concentrations and absorbance. The curve was obtained by applying different concentrations of methyl orange and obtains its absorbance. This curve makes the reading of concentrations more easily when applying the samples as the absorbance hit the curve and read the concentration. The curve gave R^2 =0.9805 which is acceptable.

4. CONCLUSION

This research main aim is to study chitosan based catalyst and its application and characterizations. Chitosan based catalyst has many applications including dye removal. Spectrophotometer is the device that was used in our experiment to measure the dye removal and make sure that the paper reached its goal by applying the application of dye removal. The results showed that chitosan based catalyst with metal ions has a great effect in dye removal and the best metal ions gave good results is nickel. In order to study the characterizations of the chitosan based catalyst wide x-ray diffraction (WXRD) test was applied and it showed a great result.

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