

Fabrication of Carboxymethyl Cellulose Supported Bi-Fe Nano Catalyst for the Removal of Dyes from Aqueous Medium under Direct Sunlight Radiation

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Abstract

Carboxymethyl cellulose (CMC) supported Bi-Fe nanocomposite is synthesized by very simple, cost effective and environment friendly co-precipitation method. The characterization of the synthesized Carboxymethyl cellulose (CMC) supported Bi-Fe nanocomposite is performed by the help of UV-visible spectrophotometer, X-ray diffraction (XRD), scanning electron microscopy (SEM) and Fourier transform infrared spectrophotometer (FTIR). The UV-visible and Fourier transform infrared spectrophotometer (FTIR) confirmed the presence of Bi-Fe nanocomposite. The X-ray diffraction (XRD) showed the structure and particle size of the prepared Carboxymethyl cellulose (CMC) supported Bi-Fe nanocomposite. The scanning electron microscope (SEM) confirmed the morphology of the structure of the nanocomposite from micrometer to nanometer. The degradation of methylene blue (MB) was done in 180 minutes. It was also very effective for the degradation of methyl orange and ortho-nitrophenol. 0.05mM solution of methyl orange was degraded up to 85% in 150 minutes and 1mM solution of ortho nitrophenol was degraded up to 70 % in 30 minute. The time effect also confirmed this factor. The concentration of MB is taken as 0.01mM. In every 10ml solution 1mg of catalyst was added. Same concentration of catalyst was taken for MO and ortho-nitrophenol. The degradation done by the carboxymethyl cellulose (CMC) supported Bi-Fe nanocomposite was up to 82% for MB, 85% for MO and 70% for ortho-nitrophenol as confirmed by UV-visible spectrophotometer.

Keywords: Photocatalytic degradation, Carboxymethyl cellulose, Bismuth nitrate, Iron sulphate, Methylene Blue, Methyl Orange, Ortho-Nitrophenol

Introduction

Nanotechnology gained very importance in the field of chemistry, physics and other field of biomedical sciences(Ojha, Jain, Mehra, & Jain, 2021).In the last few decades, the nanotechnology has gained a lot of importance in front of researcher and scientist(L. Y. Lu, Lin, Liu, & Yu, 2012). They investigated the synthesis, application and the characterization of different type of nanocomposites and material by using different methods. Metals have been used in chemistry since long time in different forms. It has also been used in the fields of biomedical sciences, chemical sciences, engineering, electronics and other branches of sciences along photonics too(Zhang & Applications, 2021). Metals have been used in these fields in macro form but they gained more importance when are in the finely grounded form. So, now, new ways of synthesizing nano material

have been introduced. Micro and nanometallic material have been introduced now a days such as bimetallic and trimetallic composite have been synthesized in the laboratory by different techniques such as sol-gel, hydrothermal, pyrolysis and other chemical and mechanical methods(Kgatle, Sikhwivhilu, Ndlovu, & Moloto, 2021)(Jayan, Sethulekshmi, & Venu, 2021). Bimetallic technology has been developed now a days in the fields of chemistry and medical sciences. The green synthesis technology also gained importance due to nonhazardous and simple method of synthesis(Cao et al., 2021). The bimetallic nanoparticles have attracted a lot of attraction due to the synergistic effects. The bimetallic catalyst has gained very importance for the degradation of dyes and purification of polluted water. Bimetallic have a lot of importance due to their vital role as catalyst and other fascinating properties(Haldar, Kundu, Patra, & interfaces, 2014). The composite of different methods has been introduced like the composite of palladium, gold, platinum, titanium, vanadium, tungsten, iron, copper, cobalt, nickel, tin and zinc(Al-Mamun, Kader, Islam, & Khan, 2019). For the last few decades, the water has been the major issue around the world and almost in every country. The protection of water from the polluted material can be done by using different type of methods and technology. The nanotechnology is one of the main techniques used for the purification and treatment of polluted water around the world now a days. So many scientists have highlighted the main point of contamination of water either by human activities or by industries which spread anthropogenic effects on the quality of water in whole world(Ali & Ahmad, 2020). The main type of pollutant is due to the presence of organic material in the water, Such as dyes from the fabrics industry or by any textile mill(Hashim, Shaw, AlKhaddar, Kot, & Al-Shamma'a, 2021). The nano catalyst has widely been used as catalyst for the treatment and purification of water around the different countries of the world(F. Lu & Astruc, 2020). Carboxymethyl cellulose is well known organic compound. When it is used as supporter for any bimetallic nanoparticles(Abd El-Lateef, Albokheet, & Gouda, 2020). Then it is also increasing the effectiveness have been used for the degradation of so many dyes. But when it is supported by the CMC its effectiveness and catalytic properties increase. The current study is designed to study the effective behavior of Bi-Fe nanocomposite which is supported by carboxyl methyl cellulose(Zaahari, Huat, Kaus, Kassim, & Othman, 2019). So, CMC supported Bi-Fe for degradation and wastewater treatment become very effective and low-cost synthesized by coprecipitation method. The wastewater treatment and degradation of Methylene blue, Methyl orange and ortho-nitrophenol have been done successfully. The research project has investigated the different application of synthesized Bi-Fe NCs supported by CMC. The application of this synthesized CMC supported Bi-Fe nanocomposite proved the degradation of methylene blue up to 82%, MO up to 85% and ortho-nitrophenol up to 70%.

Materials and Methods

Materials

FeSO₄.7H₂O was purchased by the Fisher chemical U.K. Potassium hydroxide, sodium hydroxide, carboxymethyl cellulose, FeCl₂.4H₂O was purchased from sigma Aldrich Germany. Bi(NO₃)₃.5H₂O was purchased from Uni-Chem. Chemical reagent Methyl orange, methylene blue and ortho-nitrophenol from sigma Aldrich Germany. All the chemical used were analytical grades and no purification was required (Zhu, Zhou et al. 2019).

Synthesis of Fe nanoparticle (Co-Precipitation Method)

The iron nanoparticles were prepared by the standard solution of FeSO₄.7H₂O by dissolving it with 300mL of distilled water and 0.1M solution of KOH was added to it to maintain pH between 11. It was stirred and heated on hot plate for 6-7 hours at 60-70°C^o. The solution was settled down for

several hour then decanting and after drying the precipitates were collected. The resulted powder of black color of nanoparticles of iron was obtained in powder form.

Synthesis of Bi nanoparticle (Co-Precipitation Method)

The Bi nanoparticle were synthesized by the standard solution of $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$. The KOH was added to it to maintained its pH above 11. Then also heated up to $60\text{-}70\text{C}^0$ for 6 hour and allow the solution to settle down for few hours then was dried in an oven to get the Bi nanoparticle in yellow color. The resulted powder of yellowish color of nanoparticles of bismuth was obtained in powder form.

Synthesis of Bi-Fe nanocomposite (Co-Precipitation Method)

15 mL of standard solution of $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ and 35 mL of standard solution of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ was mixed in a beaker and was put on the hot plate with stirring and pH was maintained at 11 by adding 0.1 M solution of KOH. The heating was continued for 6-7 hours at 65C^0 . Then decanting and drying in oven was done to obtained the final product of Bi-Fe nanocomposite. The resulted powder of blackish brown color of nanocomposite of bismuth-iron was obtained in powder form as shown in figure 3.

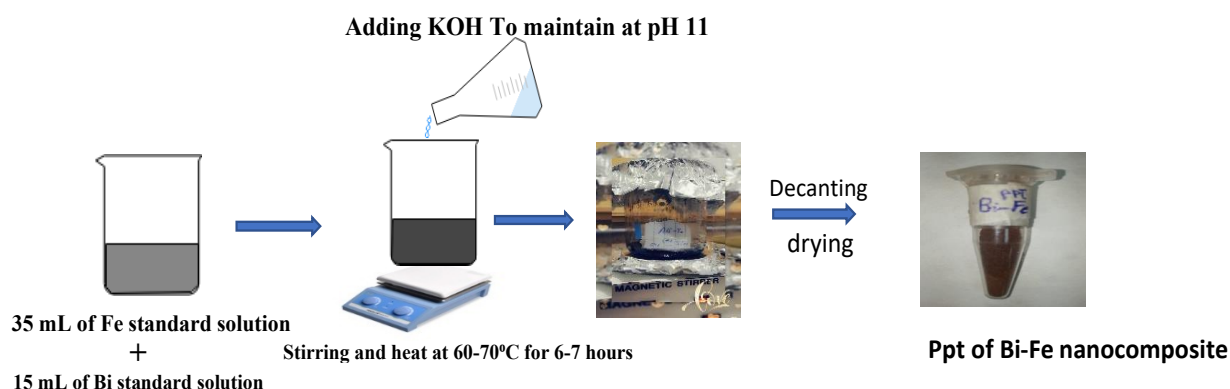


Figure 1. Schematic diagram for Bismuth-Iron NCs synthesis

Synthesis of 0.1 M CMC solution

The CMC solution was prepared by mixing 0.6g of CMC in 25mL of distilled water. It was stirrer for 120 minutes at 50C^0 on hot plate. A viscous solution was prepared.

Synthesis of Beads of CMC supported iron nanoparticles (Co-Precipitation Method)

The beads of CMC supported iron nanoparticle was prepared by putting 5mL of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ solution on Petri dish. Then 2.5 mL of solution of Fe nanoparticle and 2.5 mL of 0.1M CMC solution was mixed in Petri dish and filled in injection and was drop wise added to 5mL of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ solution to prepared beads of CMC supported Fe nanoparticles. The beads prepared have blackish color. The resulted powder of black color of CMC supported Iron NPs was obtained in powder form.

Synthesis of Beads of CMC supported Bismuth nanoparticles (Co-Precipitation Method)

The beads of CMC supported Bismuth nanoparticle was prepared by putting 5mL of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ solution on Petri dish. Then 2.5 mL of solution of Bi nanoparticle and 2.5 mL of 0.1M CMC solution was mixed in Petri dish and filled in injection and was drop wise added to 5mL of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ solution to prepared beads of CMC supported Bi nanoparticles. The golden color

beads of CMC supported Bi nanoparticles was formed. The resulted powder of brownish color of CMC supported bismuth NPs was obtained in powder form as shown in figure 6.

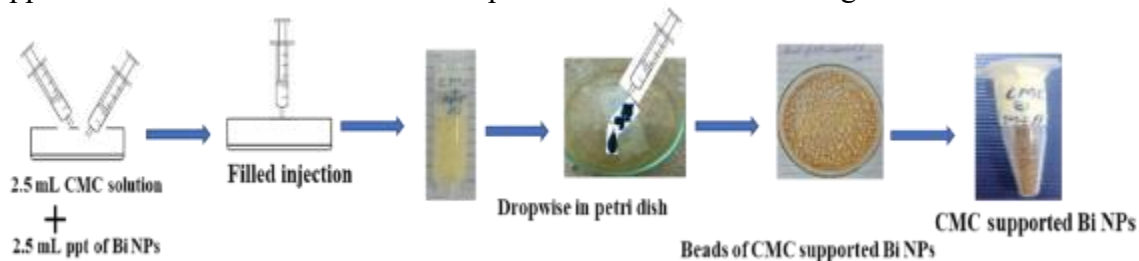


Figure 2. Schematic diagram for bead of CMC supported Bismuth NPs synthesis

Synthesis of Beads of CMC supported Bismuth-Iron nanocomposite(Co-Precipitation Method)

The beads of CMC supported Bismuth-Iron nanocomposite was prepared by putting 5mL of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ solution on Petri dish. Then 2.5 mL of solution of Bi-Fe nanocomposite and 2.5 mL of 0.1M CMC solution was mixed in Petri dish and filled in injection and was drop wise added to 5mL of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ solution to prepared beads of CMC supported Bi-Fe nanocomposite. The light blackish color beads of CMC supported Bi-Fe nanocomposite was synthesized

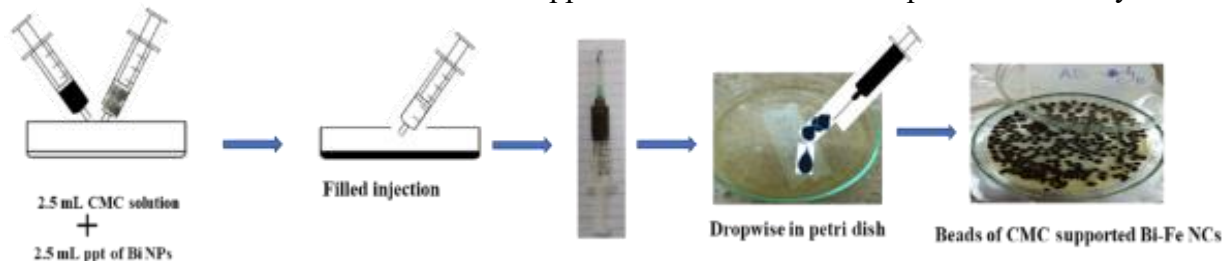


Figure 3. Schematic diagram for bead of CMC supported Bismuth-Iron NCs synthesis

Synthesis of CMC supported Bi-Fe nanocomposite

The synthesized beads were converted to powder form in a mortar. The finally powder were form and its surface area were increased to enhance catalytic properties. The resulted powder of blackish brownish color of CMC supported Bi-Fe NCs was obtained in powder form.

Instrumentation

The catalytic properties of synthesized NPS were investigated with the help of CEIL-7400ce UV-visible spectrophotometer by measuring the lambda max under sun light and also by the help of FTIR spectra on FTIR spectrophotometer. The X-ray diffraction study was also done at the scanning rate of 0.05min^{-1} with the help of Berker Dg advance diffractometer at the acceleration voltage was done with the help of NOVA SEM 450 to get the images of synthesized Bi NPs at different ranges.

Result and Discussion

UV spectra of iron

The UV spectra gained by the help of UV-Visible spectrophotometer appeared at different value between 300-400nm and lambda max appear at 352nm. It confirms the presence of iron nanoparticle (Klačanová et al., 2013).

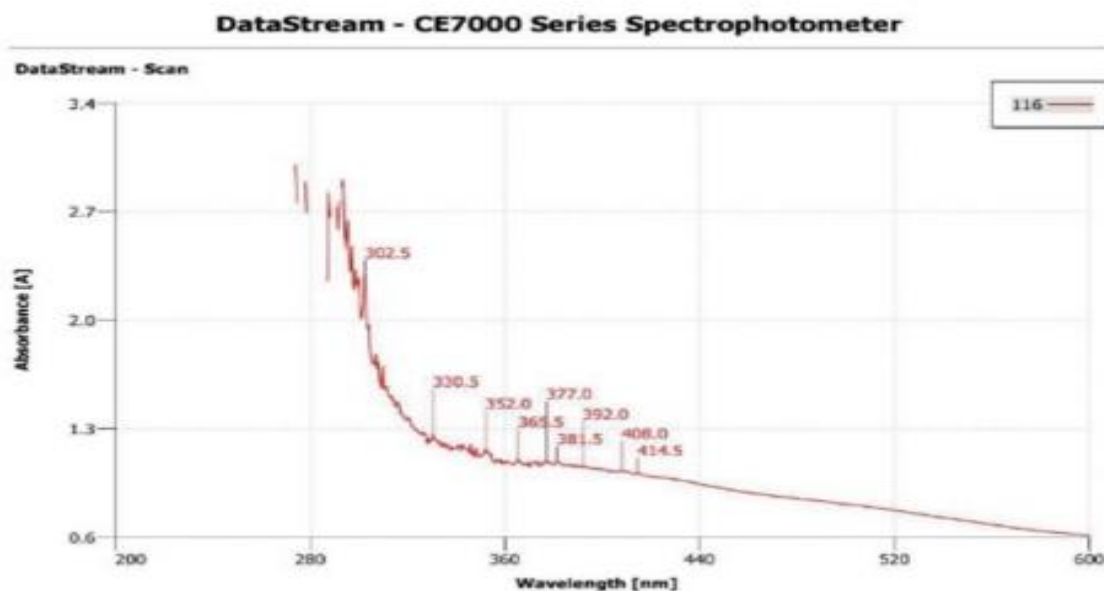


Figure 4.UV spectra of iron

Table 1.UV spectra of Fe NPs

Reported	Observed	NPs
300-400nm	352nm	Fe

UV spectra of Bismuth

The spectra obtained for the Bi nanoparticle comes at the range of 297-401nm. The lambda max appears at 351nm which is cleared by the reported value UV-Visible spectrophotometric analysis confirm the presence of Bismuth nanoparticle (McInnes, Sagu, & Wijayantha, 2014).

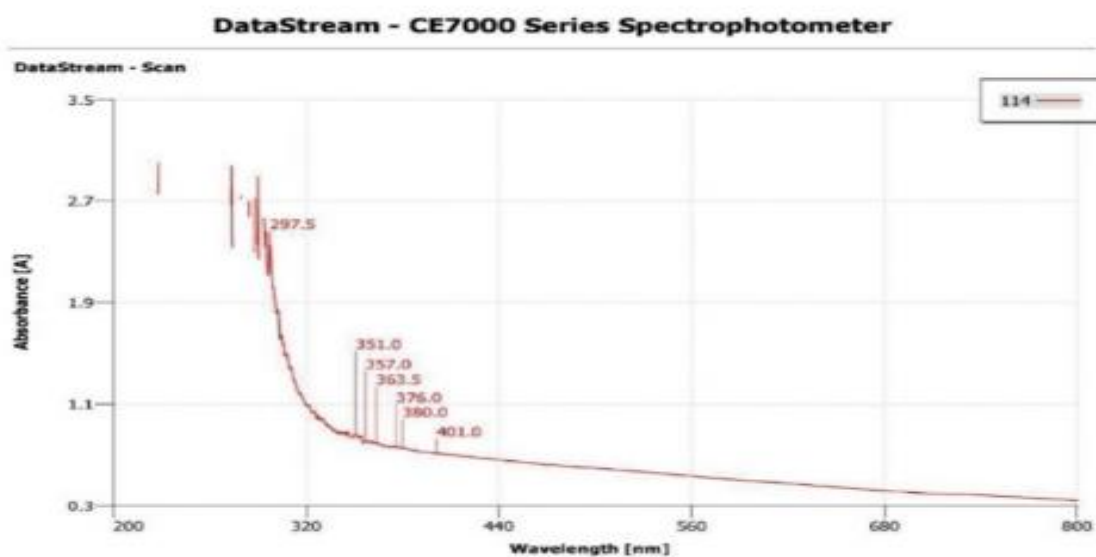


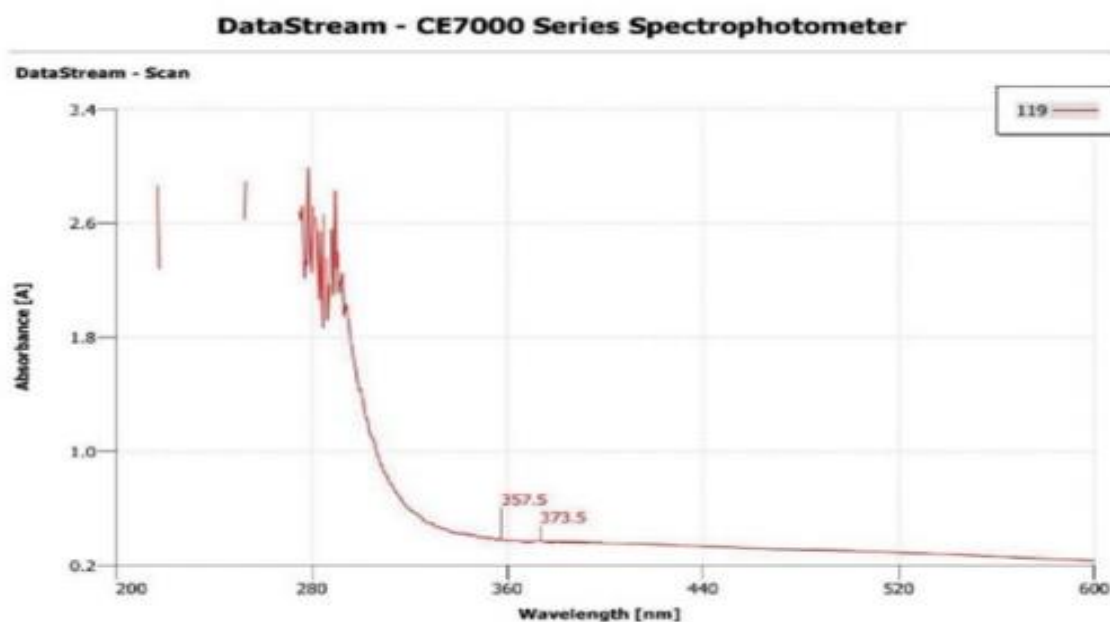
Figure 5.UV spectra of Bismuth

Table 2. UV spectra of Bi NPs

Reported	Observed	NPs
297-401nm	351nm	Bi

UV spectra of Bi-Fe nanocomposite

The nanocomposite appeared by Bi-Fe shows the peak at specific value as Bi and Fe individually which shows the formation of composite. So, the confirmation is done by the UV spectra of Bi-Fe nanocomposite at 357nm and 373nm which is the lambda max peak for composite and this peak is absence in the Bi and Fe individually. This is the confirmation of nanocomposite(McInnes et al., 2014)(Klačanová et al., 2013).

**Figure 6.** UV spectra of Bi-Fe nanocomposite**Table 3.** UV spectra of Bi-Fe NCs

Reported	Observed	NCs
350-400nm	357 and 373nm	Bi-Fe

SEM morphology

The image obtained by NOVA SEM 450 in LUMS Lahore Pakistan confirmed the particle size of CMC supported Bi-Fe nanocomposite up to 100nm. The images confirmed the presence of heterogeneous mixture of Bi-Fe. The surface of the nanoparticles is not smooth due to the interaction of particle with each other (Wei, Sun et al. 2018, Liu, Yu et al. 2020). So, this heterogeneous structure of the synthesized nanocomposite enables it to degrade any material with much accuracy.

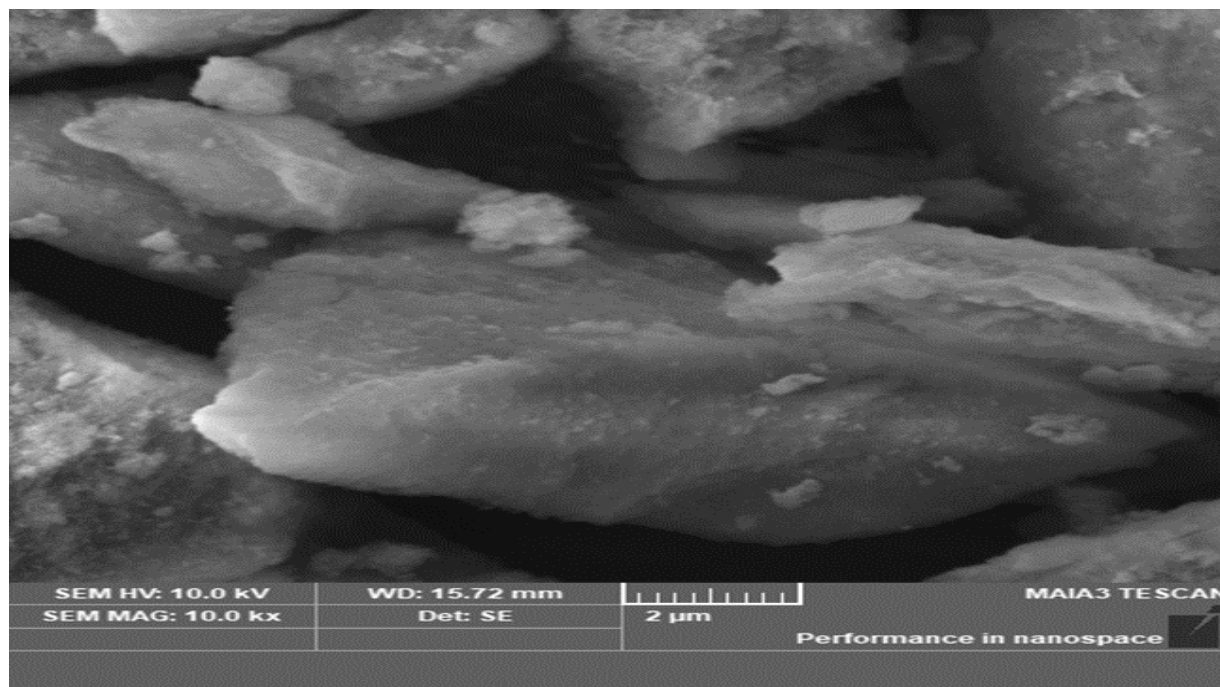


Figure 7. SEM pattern of Carboxymethyl cellulose (CMC) supported Bi-Fe nanocomposite

XRD

XRD spectra was investigated for the exact structure determination of the synthesized nanoparticle. Diffraction process appears at value for 2θ at 17.5° , 22° , 25° , 27.5° , 30° , 33° , 35° , 37.5° , 40° , 42° , 44° , 45.5° , 49° , 52.5° , 53.5° , 56° , 59° , 62° , 65° and 67.5° . the miller indexes for this value appear at 200,211,220, 031,222, 321, 400, 411, 024, 332, 422, 510,521, 035, 600,532, 541, 631, 710,721 respectively. The specific structure of almost rhombohedral type is observed in this way. So, in this way the structure elucidation was done by the help of XRD(McInnes et al., 2014)(Klačanová et al., 2013)(Zaahari et al., 2019).

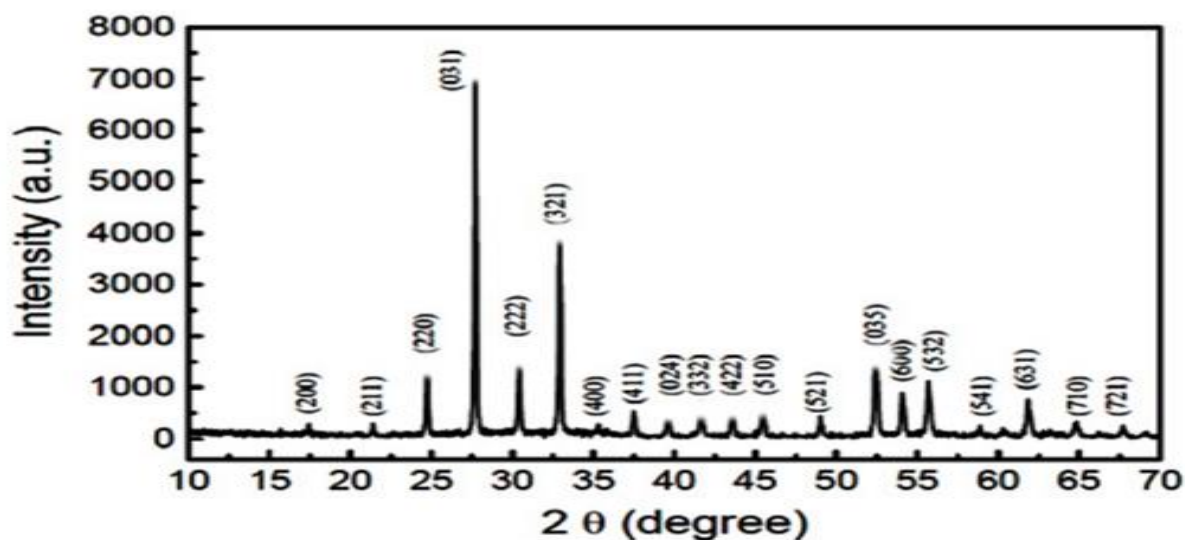


Figure 8. XRD pattern of Carboxymethyl cellulose (CMC) supported Bi-Fe nanocomposite

FTIR analysis

FTIR spectra of CMC

FTIR analysis done for the CMC confirmed the presence of different functional group attached with main compound. The peak at 3247.91 cm^{-1} showed the original solution of CMC for OH. The peaks at 2876.85 cm^{-1} shows the presence of C-H stretching for aldehyde. The strong peak at 1585.35 cm^{-1} is for N-H group and peak at 1412 cm^{-1} , 1321 cm^{-1} is for N=O. The peak at 1018 cm^{-1} shows specific peak for the C-O-C. The peak at 712.15 cm^{-1} is the characteristic peak for the absorption of C-H (Ghazali et al., 2014).

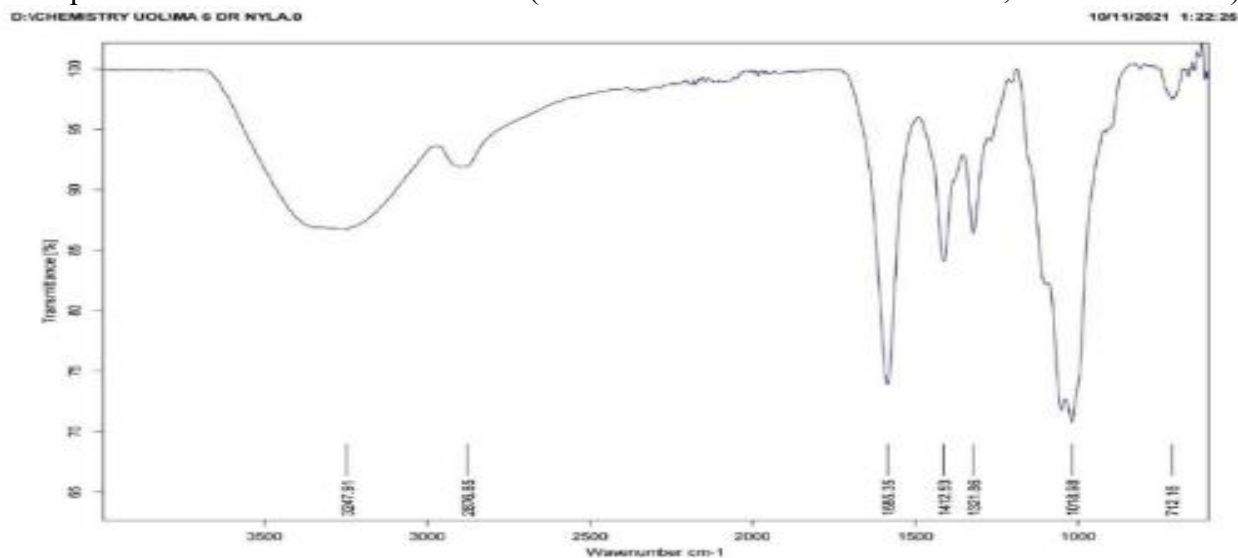


Figure 9. FTIR spectra of pure CMC

FTIR spectra of Bismuth nanoparticles

The functional group attach with the Bi nanoparticles was observed with the Fourier transform infrared spectrophotometer. The functional group observed at 615.40 cm^{-1} for $\text{Cl}^{-1}\text{ cm}^{-1}$ elongation, at 830 cm^{-1} , 875 cm^{-1} , 996 cm^{-1} , 1077 cm^{-1} was observed for amines, the peak at 1353 cm^{-1} and 1427 cm^{-1} was for ether, the peak at 1647 cm^{-1} was for $\text{C}=\text{O}$ and peak at 3450.13 cm^{-1} was for OH group (McInnes et al., 2014; Zaahari et al., 2019).

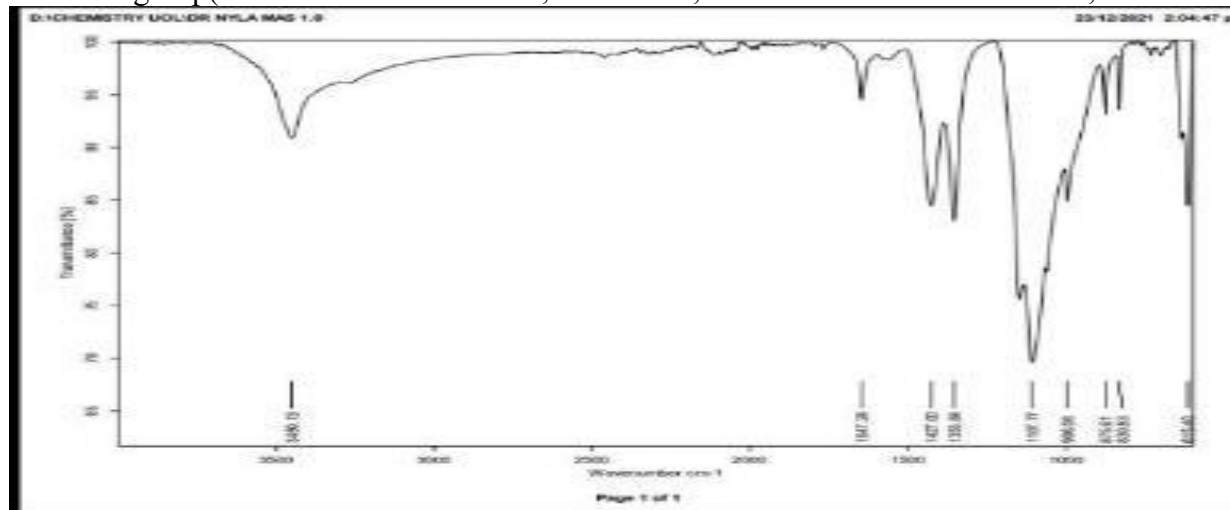


Figure 10. FTIR spectra of bismuth nanoparticles

FTIR spectra of Iron nanoparticles

FTIR observed at university of Lahore at Fourier transform IR spectrophotometer. The functional group at 607 cm^{-1} , 634 cm^{-1} , 718 cm^{-1} observed for the Cl^{-1} presence, at 795 cm^{-1} , 899 cm^{-1} observed for the ether, at 1098 cm^{-1} for C-O, at 1693 cm^{-1} for C=C, at 2105 cm^{-1} for $\text{C} \equiv \text{C}$ and at 3110.14 cm^{-1} for OH group (Klačanová et al., 2013).

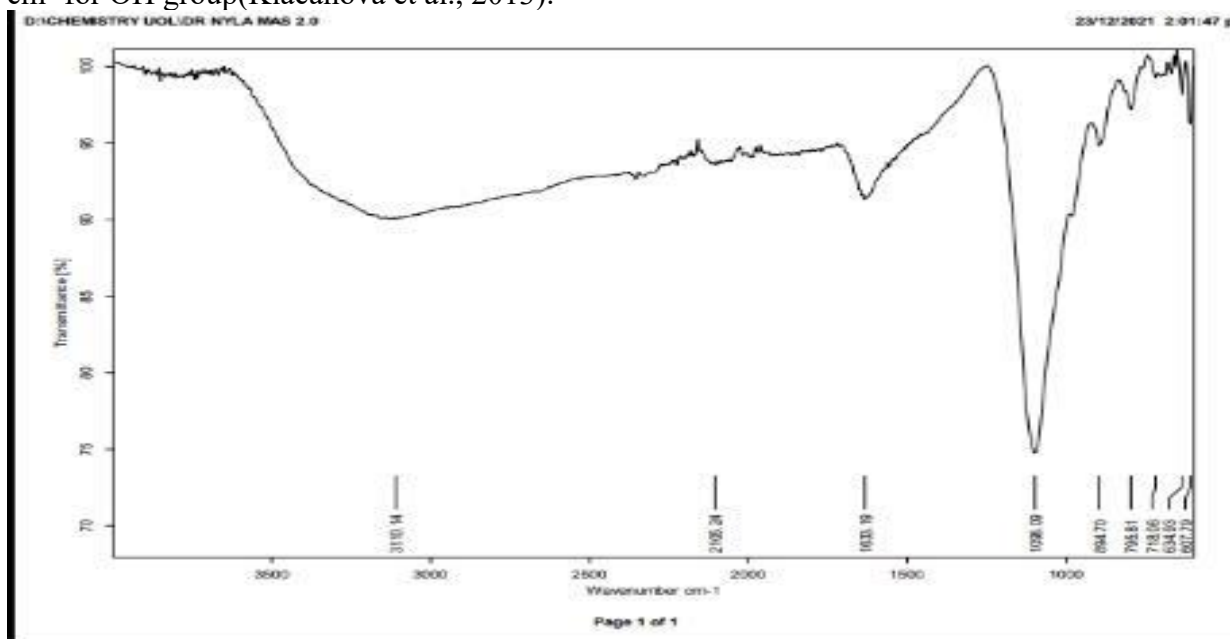


Figure 11. FTIR spectra of iron nanoparticles

FTIR spectra of Bismuth-Iron nanocomposite

For FTIR of Bi-Fe the almost same functional group was obtained except some cases at 611 cm^{-1} , 637 cm^{-1} for Cl^{-1} functional group was observed, at 875 cm^{-1} for ether group, at 1095 cm^{-1} for C-O, at 1440 cm^{-1} for N=O and at 3264 cm^{-1} for OH group show its presence (Klačanová et al., 2013; McInnes et al., 2014; Zaahari et al., 2019).

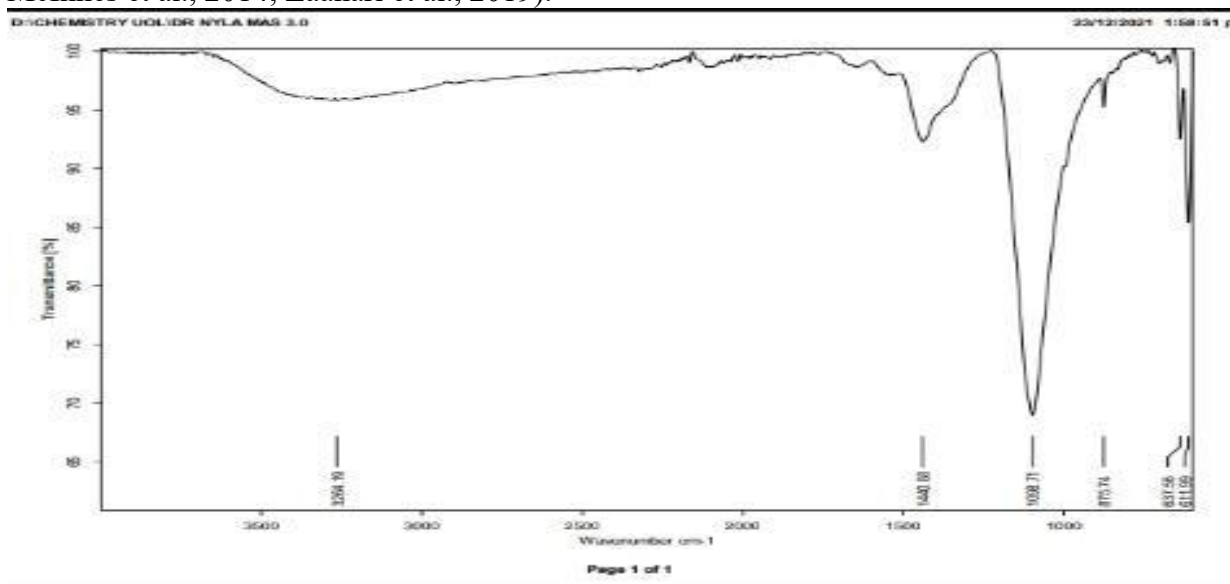


Figure 12. FTIR spectra of bismuth-iron NPs

FTIR spectra of CMC supported Bismuth nanocomposite

FTIR observed for the CMC supported Bismuth nanoparticles shows wavelength value at 692 cm^{-1} for Cl^{-1} , 1025 cm^{-1} for C-O, at 1333 cm^{-1} , 1420 cm^{-1} for $\text{N}=\text{O}$, at 1573 cm^{-1} for N-H, at 2074 cm^{-1} for $\text{C}\equiv\text{C}$ and at 3234 cm^{-1} for OH (Zaahari et al., 2019).

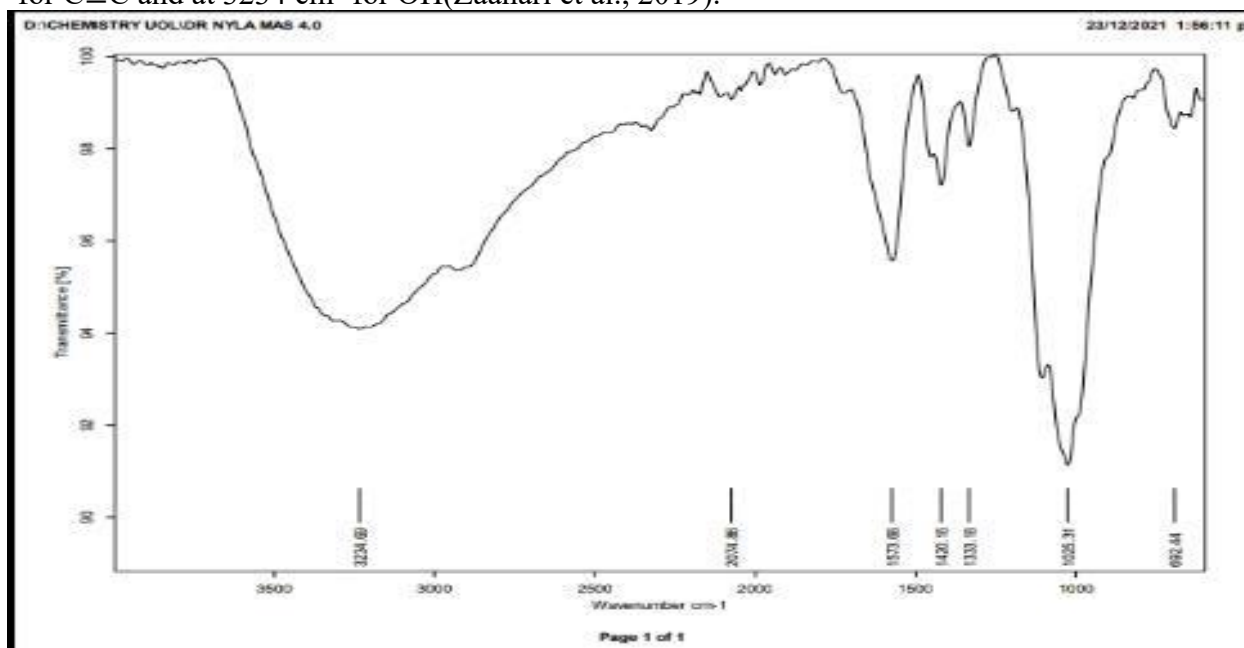


Figure 13. FTIR spectra of CMC supported bismuth NCs

FTIR spectra of CMC supported Ironnanocomposites

For CMC supported Fe NPs at 623 cm^{-1} for Cl^{-1} and 1005 cm^{-1} , 1095 cm^{-1} for C-O, at 1415 cm^{-1} for $\text{N}=\text{O}$, at 1573 cm^{-1} for N-H, at 2085 cm^{-1} for $\text{C}\equiv\text{C}$ and at 3186 cm^{-1} for OH (Zaahari et al., 2019).

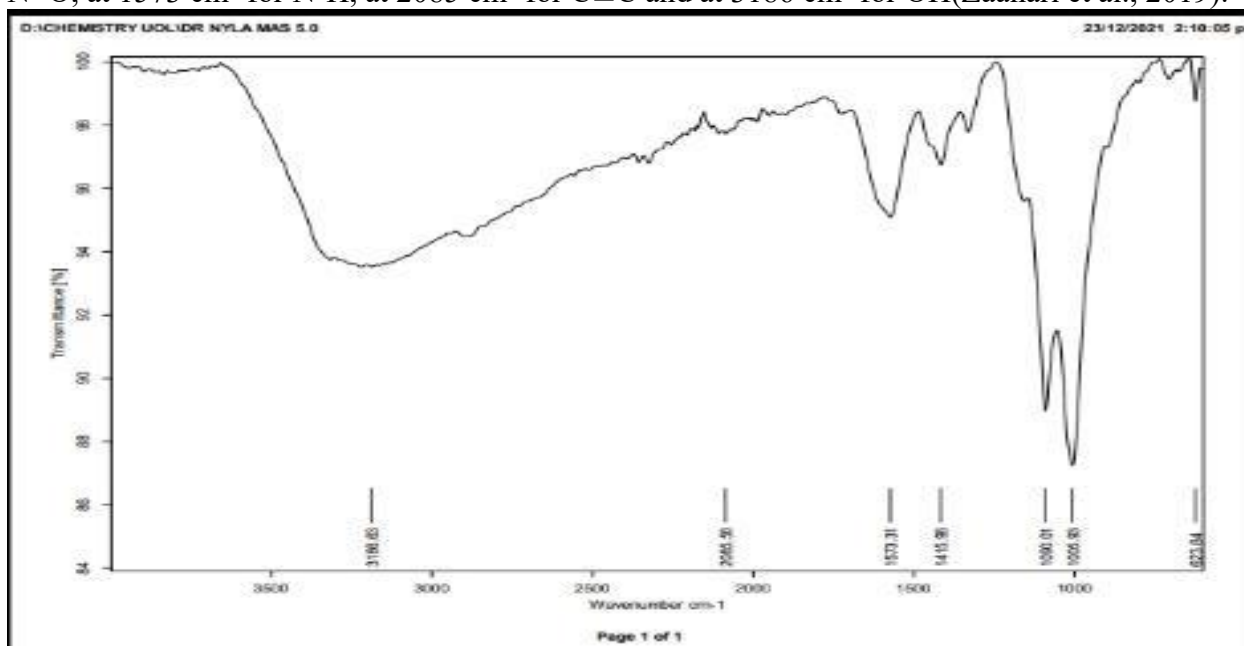


Figure 14. FTIR spectra of CMC supported iron NPs

FTIR spectra of CMC supported Bismuth-Iron nanocomposite

Fixed product obtained at CMC supported Bi-Fe nanocomposite was checked by FTIR. It shows functional group presence at 606 cm^{-1} , 622 cm^{-1} , 698 cm^{-1} for Cl^{-1} and at 1053 cm^{-1} for C-O, at 1333 cm^{-1} , 1447 cm^{-1} for N=O, at 1589 cm^{-1} for N-H, at 1981 cm^{-1} for $\text{C}\equiv\text{C}$, at 3202 cm^{-1} for OH group (Zaahari et al., 2019).

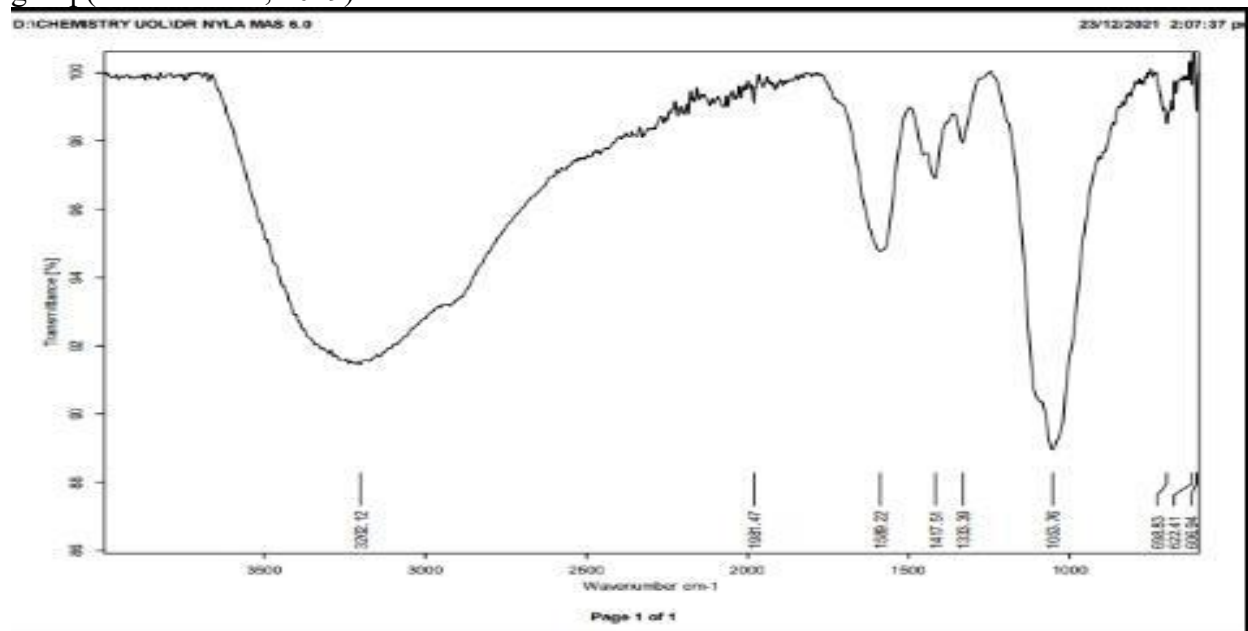


Figure 15. FTIR spectra of CMC supported Bi-Fe NCs

Photocatalytic reduction of Methylene Blue

Photo catalytic activities were performed at university of Lahore Punjab Pakistan. 0.01 mM solution were taken in five different test tubes and irradiated with the direct sunlight. It was noted that 10g of catalyst were added to each test tube and in 180 minutes off irradiation the degradation of MB was done up to 82% as confirmed by the UV visible spectrophotometer. The λ_{max} was checked at 667 nm. The degradation of MB was done by the help of CMC supported Bi-Fe nanocomposite. The degradation of MB with the help of photocatalyst was observed in direct sunlight (Indriyani, Yulizar, Yunarti, Apriandanu, & Surya, 2021). The 0.01 mM solution of MB was prepared and it shows the lambda maximum at 667 nm. The complete degradation up to 82% were done in the sun light in 180 min. time factor of this reduction was noted and observed in the UV-visible spectrophotometer from zero minute as shown in the graph. The solution of 0.5 mM, 0.1 mM and 0.05 mM were also prepared and their degradation pattern were also observed by the UV-Visible spectrophotometer. The graph shows that with the passage of time the degradation of MB increases. The catalytic effect of CMC supported Bi-Fe nanocomposite were also investigated by changing the concentration of catalyst but almost same pattern observed as the concentration increases in the sequence from 1 mg, 3 mg, 5 mg, 7 mg and 10 mg per 100 ml the degradation of the MB increases with concentration and time. The degradation of MB was noted by the help of following formula (Malathi et al., 2017).

$$\% \text{Degradation} = \frac{C_0 - C_p}{C_0} \times 100$$

By getting value of degradation at 667 nm with the passage of time on UV-VIS spectrophotometer. It was calculated at up to 82%. The MB solution and its degradation was observed from 0, 10, 20, 30, 40, 50, 80, 0, 120 and 180 minutes respectively as given by the graph clearly.



Figure 16. Degradation of Methylene Blue (x, y)

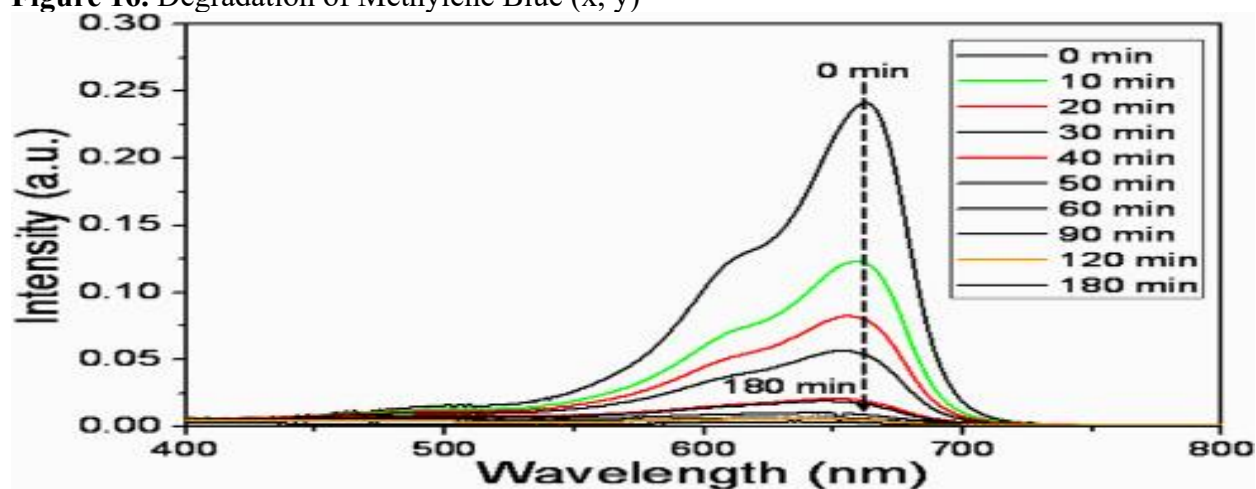


Figure 17. Degradation pattern of Methylene Blue (MB) by CMC supported Bi-Fe nanocomposite

Degradation of Methyl orange

The photo catalytic activity for methyl orange was done in university of Lahore. The solution which was selected for the photo catalytic activity were 0.05mM. The solution was taken in five different test tubes with 1, 3, 5, 7 and 10 mg off catalyst in each test tube. It was noted that in 180 minutes of irradiation of visible light from sun the degradation of MO was up to 85% which was confirmed by UV visible spectrophotometer. The λ_{max} was checked at 467 nm. MO is an anionic dye (Noori & Gholizadeh, 2020). The degradation of MO was done by the help of CMC supported Bi-Fe nanocomposite. The degradation of MO with the help of photocatalyst was observed in direct sunlight (Zaahari et al., 2019). 0.05mM solution of MO was prepared by adding 0.0327g of MO in 100ml of water to prepare 1mM solution of MO then following the concentration formula 0.1mM and 0.05mM solution was prepared. The standard solution and five other solution each containing 1mg of catalyst in 10ml solution was irradiated directly in the sun light. The λ_{max} was observed at 467 nm for MO. MO was degraded up to 85% in 150 minutes as confirmed by the UV-Visible spectrophotometer.



Figure 18. Degradation of methyl orange (x, y)

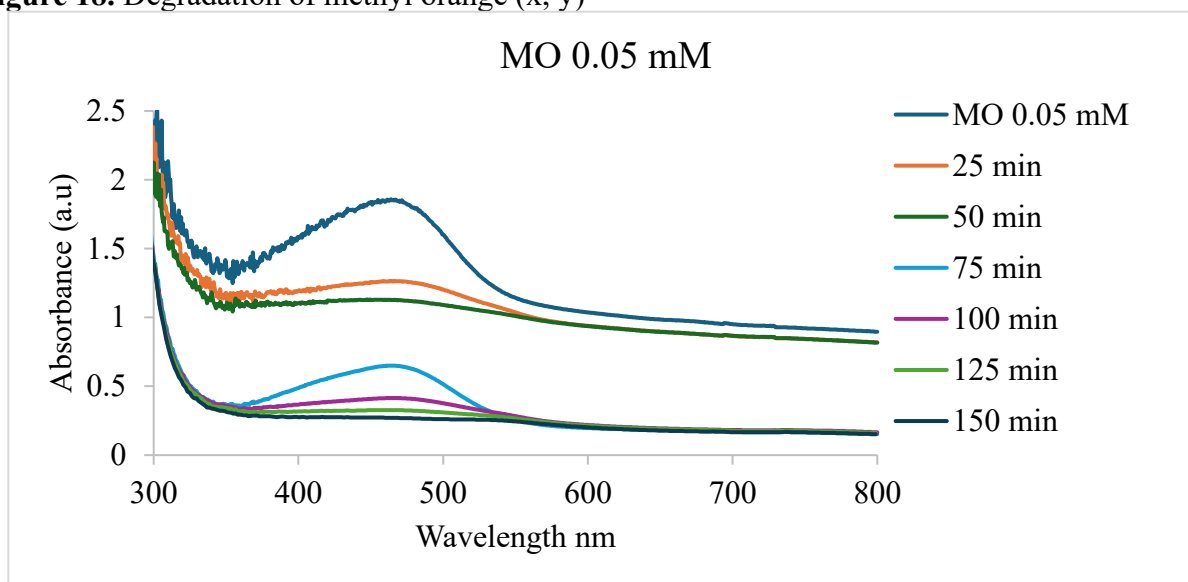


Figure 19. MO photo catalytic activity

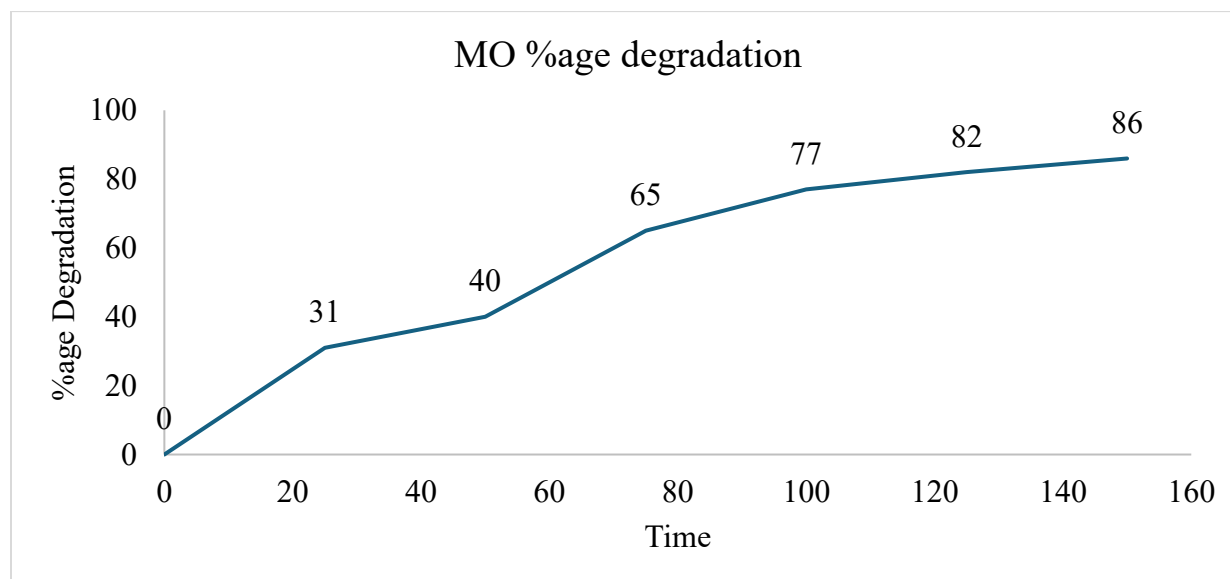


Figure 20. MO %age photo catalytic activity

Degradation of ortho-nitrophenol

1 mM solution of ortho nitrophenol was taken for the photocatalytic activity at department of chemistry university of Lahore Pakistan. The solution was taken in six different test tubes and irradiated with direct sunlight. After just 10 minutes of radiation of visible light the degradation of ortho nitrophenol was up to 70% as confirmed by the UV visible spectrophotometer. The maximum degradation of ortho nitrophenol was done by 10 mg of catalyst at λ_{\max} of 401 nm. 1mM solution of ortho-nitrophenol was prepared by adding 0.0139g of ortho-nitrophenol into 100ml of distilled water.

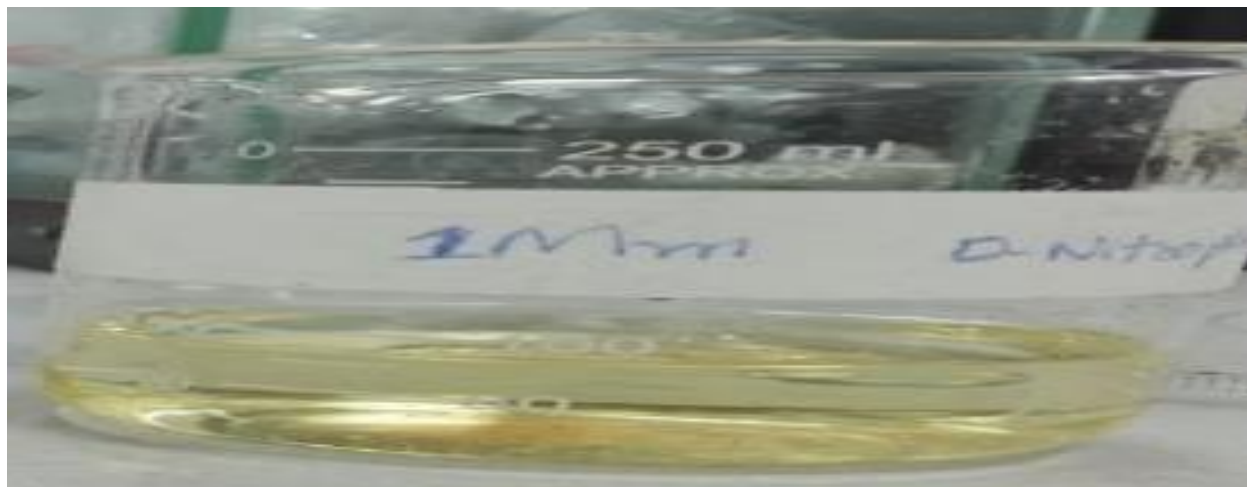


Figure 21. 1mM solution of O-Nitro Phenol

The lambda max was observed at 401nm for ortho-nitrophenol. The solution was irradiated in the presence of sun light directly. The concentration of catalyst was taken as 1mg per 10 ml of ortho-nitrophenol. It was degraded up to 70 percent as confirmed by the UV-visible spectrophotometer in the Department of chemistry University of the Lahore.



Figure 22. Degradation of O-Nitro phenol (x, y)

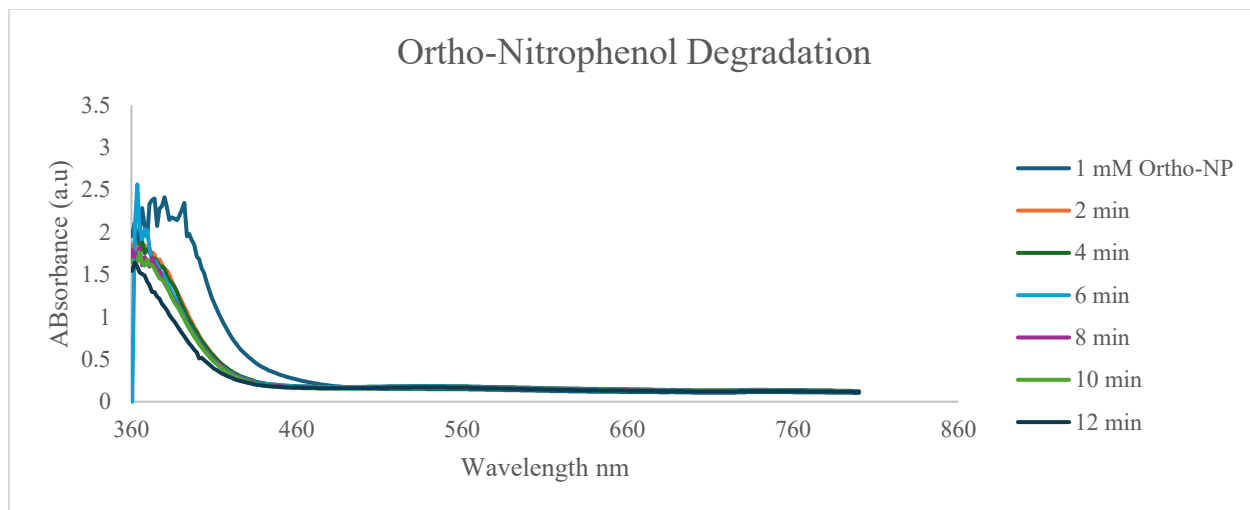


Figure 23. Degradation of O-nitrophenol by CMC supported Bi-Fe nanocomposite

Conclusion

The carboxymethyl cellulose (CMC) supported Bi-Fe nanocomposite synthesized by very cost effective and environment friendly co-precipitation method. It has excellent ability for the degradation of Methylene blue (MB), Methyl orange (MO) and ortho-nitrophenol. The Methylene blue (MB) was degraded up to 82% in 180 minutes, Methyl orange was degraded up to 85% in 150 minutes and ortho-nitrophenol was degraded up to 70% in 30 minutes. The X-ray diffraction (XRD) indicated the crystalline structure of the prepared nanocomposite. The Fourier transform infrared spectrophotometry (FTIR) confirmed the presence of Bi, Fe and CMC in sample having specific value at different wavelength. The scanning electron microscope (SEM) morphology confirmed the presence of heterogeneous structure of synthesized material. The UV-visible spectrophotometric analysis confirmed the presence of Bi and Fe respectively at specific value and the degradation of Methylene blue (MB), Methyl orange (MO) and Ortho-Nitrophenol under direct sunlight. The degradation of Methylene blue (MB) was done in 120 minutes. The concentration of MB is taken as 0.01mM and in every 10 ml solution 1mg of catalyst was added, and 0.05mM of MO was mixed by 1mg of catalyst and 1mg added to 1mM solution of ortho-nitrophenol. The degradation done by the Carboxymethyl cellulose (CMC) supported Bi-Fe nanocomposite was up to 82% for MB, 85% of MO and 70% for ortho-nitrophenol as confirmed by UV-visible spectrophotometer. So, it is concluded that CMC supported Bi-Fe nanocomposite have excellent ability to be used as photocatalyst for degradation of dyes and other material in the wastewater.

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