IJARSCT

ISSN (Online) 2581-9429

International Journal of Advanced Research in Science, Communication and Technology (IJARSCT)

International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal

Impact Factor: 7.53

Volume 4, Issue 5, April 2024

Preparation and Characterization of Scolecite/ZnO Nanocomposite by a Novel Chemical Route

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Abstract: *This research work has proposed preparation of Scolecite/ZnO nanocomposites (NCs) by a novel aqueous route using Zinc Nitrate Hexahydrate (Zn $(NO_3)_2.6H_2O$) as a precursor and distilled water were used as solvent without any requirement of calcinations step at high temperature. The comprehensive structural studies carried out using Powder X-Ray diffraction (PXRD) and Fourier Transform Infrared Spectroscopy (FT-IR) PXRD spectrum showed that the ZnO Nanoparticles exhibited crystalline structure. The average crystallite sizes of the prepared NCs calculated by using Debye-Scherrer equation. The sharp peak in the FT-IR spectrum determined the purity of ZnO Nanoparticles.*

Keywords: Nanocomposite (NCs), Natural Zeolite, Scolecite, Novel Chemical Route, ZnO, XRD, FTIR

I. INTRODUCTION

Recently, nanomaterials are considered as the world's most attractive materials due to their potential applications particularly for environmental challenges such as in water treatments [1]. Additionally, the use of nanostructured materials for water treatment processes is a reality and hot issue and thus there are long lists of nanomaterials in the markets [2]. In several studies nanoparticles have been addressed as efficient and effective adsorbents for removal of toxic metals from water systems. Among the available adsorbents, metal oxides nanoparticles (NPs) such as; titanium oxide, ferric oxide, magnesium oxide, alumina oxide, cesium oxide [3-4], manganese oxide [5], copper oxide, zinc oxide [6-7] and graphite oxide [8] which are classified as the promising and desirable sorption materials for toxic metals removal from aqua systems due to their high selectivity, capacity and efficiency to remove these metals to meet the strict regulations [9-10]. To improve the applicability of metal oxide nanoparticles for water treatment, nanocomposite (NCs) materials have emerged as suitable alternatives to overcome limitations of growth nanoparticles by employing porous supports materials of large area as a matrices or stabilizers to obtain hybrid nanocomposite adsorbents [11].

Natural zeolites are characterized by well-developed and ordered micro porous crystalline structure, molecular-sieve, adsorptive and ion-exchange properties that conditions the perspective of their practical application [12]. Natural zeolites are successfully used in such technological processes where the application of synthetic zeolites is not economically approved. Special attention is paid to the inexpensive, unique by their properties minerals which simultaneously possess selective, ion-exchange and adsorptive properties and are associate with modern actual problems of wasteless technology and environmental protection [13].Taken into account a great perspective of application of natural zeolites, investigation of physico-chemical properties of natural zeolites becomes necessary. Scolecite sample under investigation was taken from the vicinity of village Dhanora (Umarkhed Dist. Yavatmal (M.S.) India). Idealized formula of the unit cell of scolecite is as follows: $Ca_8(Al_{16}Si_{24}O_8).24H_2O$. By its chemical composition (%) - SiO_2-46.24; Al_2O_3-26.41; Fe_2O_3-0.04; CaO-12.2; Na_2O-1.2 and K_2O-0.46. This zeolite is close to the theoretical formula of this mineral [14]. On the other hand, zinc oxide (ZnO) as a useful and efficient catalyst has been explored for organic conversions. In practical applications, the use of ZnO nanoparticles with a broad surface area has led to the development of their catalytic activities. However, these nanosized particles have limitations, including the problem of the separation of catalysts at the end of the processes and the aggregation of nanoparticles [15-17]. To solve these problems, one interesting alternative is using ZnO supported on inert or active materials like zeolites that improves the catalytic efficiency and shows easy recovery by simple filtration and avoidance of aggregation of metal oxide nanoparticles. Based on thatpresent work suggested, the Novel chemical route synthesis of Scolecite/ZnO Nano-Composites (NCs) was discussed.

II. EXPERIMENTAL METHODS AND PREPARATION

Chemicals:

All chemical materials were used without any further purification. Zinc Nitrate Hexahydrate ($Zn(NO_3)_2.6H_2O$) was purchased from Sigma Aldrich. HCl purchased from MERCK. 99% Ethanol, Natural Zeolite (Scolecite) obtained from the well mining from village Dhanora, (Umarkhed, Yavatmal dist, Maharashtra(MS), India) and distilled water (DW) used as a solvent.

Preparation Scolecite/ZnO NCs:

1. Pretreatment on Scolecite crystal:

Take naturally occurring Zeolite that is Scolecite crystal. Using top-down process extracts macro material crystal and makes fine powder of Scolecite crystal using mortar pestle. Take 5gm of natural scolecite and wash with distilled water for one hour using a magnetic stirrer at room temperature and filter the pure Scolecite powder and dry overnight at room temperature.

2. Synthesis:

The etching solution of 0.1N HCl was made. The dried Scolecite powder is treated with 0.1N HCl to activate Scolecite. The activation process using 0.1N HCl which influences the adsorption capacity of zeolite [18].After activation scolecite powder were reacted with 1M (29.74gm) of zinc nitrate hexahydrate ($Zn(NO_3)_2.6H_2O$) dissolved in 50 ml deionized water and make it 100ml solution and the etched powder of natural scolecite were added to zinc nitrate hexahydrate solution. The reaction was carried out at room temperature by using a magnetic stirrer for 2 hours. Switch off the magnetic stirrer after 2 hours and keep it constant for the next 22 hours. Filter the product and dry under sunlight. In an aqueous solution, ions can exchange into and out of the Scolecite framework structure.

Characterization of Scolecite/ZnO NCs:

The crystalline structures of the prepared NCs were investigated using PXRD with the Tabletop XRD, Miniflex 600, RIGAKU instrument in the wide angle range of 2θ (10 – 90̊). The particle sizes of the prepared NCs were calculated by the Debye-Scherrer equation,

$$D = \frac{k\lambda}{\beta \mathrm{Cos}\theta}$$

Where, k = 0.94 is a coefficient, λ = 0.15418 nm is the X-ray wavelength, β is the full width half maximum (FWHM) of the sample and θ is the diffraction angle.

The FT-IR spectra were recorded in the range of 400–3500 cm^{-1} using FT-IR Spectrophotometer (FT-IR) -Shimadzu, Japan spectrophotometer.

III. RESULTS AND DISCUSSION

Powder X-ray diffraction:

Powder X-ray diffraction patterns for Scolecite and the Scolecite/ZnO NC are shown in Fig.1 below:

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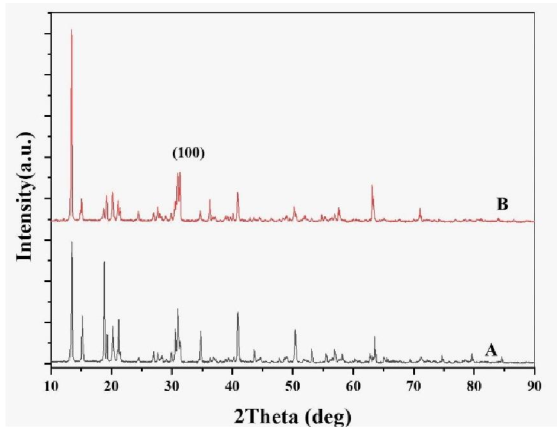

Fig.1. A and B X-ray diffraction pattern of Scolecite and Scolecite/ZnO NCs respectively.

The patterns suggest that the pure scolecite peaks (A) which shows the typical monoclinic crystal system [21] and the patterns of Scolecite/ZnO NCs (B) peaks are mostly similar with the pure scolecite peaks (A). Moreover, new peaks appearing in the Scolecite/ NCs (B) belong to ZnO. The changing of the relative peaks intensity and small shift to the higher diffraction angles (Fig.1) indicate the small compression of the Scolecite crystalline structure in the presence of ZnO NPs without changing in the cubic structure shape. New peaks appeared at (34.31°) which assigned as (100) related to the Zinc Oxide which were in accordance with ZnO [19].while some other ZnO peaks (002) at 34.45°, (101) at 36.43° cannot be observed in the XRD pattern of Scolecite/ZnO NCs, meaning that; the ZnO NPs are confined inside the scolecite, same reason was reported [20].The average crystallite sizes of the prepared NCs calculated by Debye-Scherrer equation and found 36.85nm. Moreover, there are no other diffraction peaks appearing in the product except the diffraction peaks of scolecite and ZnO NPs, which means the prepared product is pure.

Fourier Transform Infrared

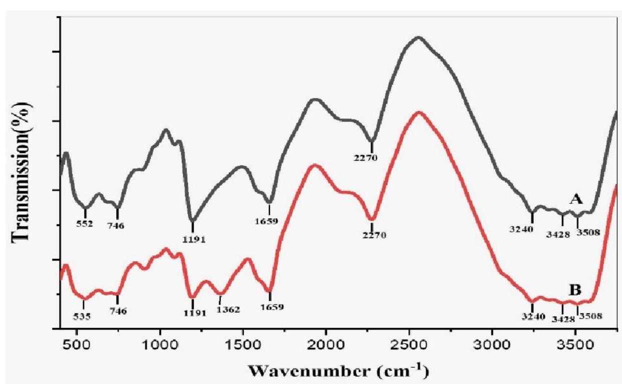

Fig.2. Fourier transform infrared (FT-IR) spectra of; A: Scolecite and B: Scolecite/ZnO NCs respectively.

Fig.2. illustrates FT-IR spectra of pure Scolecite and the prepared Scolecite/ZnO NCs. In pure Scolecite the broad band of 1191 cm^{-1} caused by the stretching vibration in Si (Al)-O group. The band at 1659 cm^{-1} is related to the remaining H_2O molecules bending vibration in Scolecite voids. The broad band at 1362 cm^{-1} indicates the stretching of the O-H group. The broad band centered at 535 cm^{-1} observed in the spectrum of synthesized ZnO is due to the vibrations of Zn–O bonds, which confirm the formation of ZnO.

IV. CONCLUSION

In this research work, Scolecite/ZnO NCs were carefully prepared and characterized successfully a Novel Chemical route and FT-IR and PXRD confirm synthesis of Scolecite/ZnO nanoparticles. The synthesized ZnO nanoparticles

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exhibit good crystallinity which was approved by PXRD spectra. The prepared NCs can be examined for toxic metal removal water.

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