Synthesis and Characterization of Nanocrystalline Sodium Zinnwaldite Mica Mineral

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Abstract: Present study encompasses synthesized of nanocrystalline sodium zinnwaldite (ZWD) mica mineral. The material was synthesized by hydrothermal method (HM) and microwave method (MM). Compared with hydrothermal heating, microwave heating has the advantages of short reaction time, producing small particles with a narrow size distribution and high purity. Synthesized ZWD by solution route method and it was characterized by using different techniques such as X-ray diffraction, Energy dispersive analysis, scanning electron microscopy, transmission electron Microscopy and FT IR analysis. A computer program, called MINTEQA-2, which was developed by USA Geosciences, is used for predicting arsenic, cadmium and lead as functions of pH and solubility. The sorption data of pH may be specified as equilibrium values or MINTEQA-2 can calculate.

Keywords: MINTEQA- 2; TEM; XRD; FT IR; ZWD.

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I. INTRODUCTION

Mica group of minerals are natural, heterogeneous and colloidal particles having diameter less than 0.002 mm. This is an important property and the other properties are swelling, adsorption and ion exchange capacity. In addition to this, particle size and grain shape are the main properties that affect the usage of mica minerals. Human beings found various applications of layered clay mica minerals since prehistoric civilization due to their widespread distribution and a great diversity of reactions in nature [1]. A wide new application of mica minerals is also used to remove organic matter, drug residual, trace metal ions and lots of pollutant from the wastewaters due to their adsorption and ion exchange capacity. Recently, the development of nanoscience and nanotechnology ignited a new round of interest in clay minerals. Based on their nano-sized layers as well as the nano-sized interlayer space, clay mica group of minerals can act as naturally occurring nanomaterials or as nano-reactors for fabrication of nano-species, nanoparticles or nanodevices [2]. These mineral-based functional materials and nanocomposites show a great variety of applications in catalysis and adsorption, environmental remediation, polymers, electronics and fuel cells [3-8].

Mica minerals are similar to zeolites. Moreover, both are alumino-silicates. They differ, however, in their crystalline structure. Many types of micas have a layered crystalline structure (similar to a sheet) and are subject to swelling and shrinking as water is absorbed and removed between the layers. In contrast, zeolites have a rigid, 3-dimensional crystalline structure (similar to a honeycomb) consisting of a network of interconnected tunnels and cages. Micas, is a class of silicates having a prismatic angle 120o, eminently perfect basal cleavage, affording thin tough laminate or scales, colorless to jet black, transparent to translucent, of widely varying chemical composition and crystallizes in the monoclinic system. Sodium zinnewaldite micas are capable of removing pollutants from the environment and thereby control the spread of pollutants in soil, water and air.

Aluminosilicates exhibit good properties of sorption for the minimization of toxicological effects of toxic metal ions [9]. The Sorption processes are most attractive, since their application is simple and they require mild operating conditions. The amount of metal ion waste is increasing every year; they tend to accumulate in living organisms. Treatment processes for the removal of trace metal ions from wastewater include coagulation, adsorption, ion exchange, reverse osmosis, etc. [10, 11].

The aim of the present study involved is to synthesis of sodium zinnewaldite (Nanocrystalline). Here we demonstrate the synthesis and characterization of sodium zinnewaldite mica clay minerals. Sodium zinnewaldite are often reported to exhibit high sorption capacity and selectivity for divalent and trivalent ions, which makes the mica mineral attractive for the environmental applications, such as water softening or removing toxic elements from water [12]. Sorption of metal ions by sodium zinnewaldite mica is governed by ion exchange or chemisorptions.

II. MATERIALS

Materials and Methods

Aluminum nitrate, tetraethylorthosilicate TEOS (Aldrich), sodium hydroxide, tetramethylammonium hydroxide (TMA, 35% wt by in water) solution was purchased from (Across), lithium nitrate, magnesium nitrate and hydrofluoric acid. All the reagents and chemicals used were of analytical grade obtained from Merck and Aldrich.

Synthesis of Nanocrystalline Sodium Zinnewaldite

Sodium zinnewaldites was synthesized by under hydrothermal and microwave condition In this system.

III. RESULTS AND DISCUSSION

The nanocrystalline sodium zinnewaldite micas are extensively characterized by powder X-ray diffraction (XRD). Fig.1 showed that ZWD-5 has good crystallinity. The slightly arched baseline of commercial XRD spectra suggests in some cases the synthesized materials to be amorphous. Sodium zinnewaldite (ZWD) nanocrystal was recorded during the microwave synthesis; which proved it to be completely crystalline without the presence of any impurity phases.

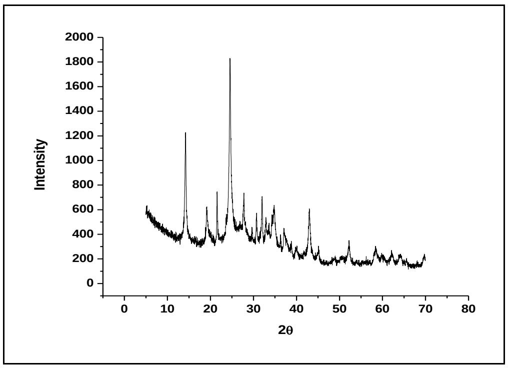

Fig.1 XRD patterns of synthetic analogues of sodium- zinnewaldite

Powder X-ray diffraction patterns of the analogue viz. ZWD synthesized from media of different alkaline concentration and with tetraethylorthosilicate as the silica source by maintaining the temperature and crystallization conditions are shown in Fig 1. The peak intensity and 2θ value increases with increasing the crystallization time, temperature and produces small particle size [15]. Finally, observed clearly peak intensity and 2θ values at ±1000C for 50 minutes in figure 1.

The chemical composition of the synthesized powder obtained from energy dispersive analysis (Fig.2) for the metals F, Na, Si & Al agree well with the weight percentage of these elements.

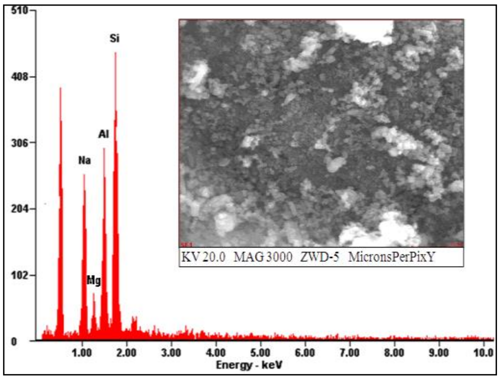

Fig.2 EDS analysis of synthesized for sodium - zinnewaldite

The chemical composition of the template-free Further, the synthesized material was characterized by electron microscopy as TEM and SEM analysis. The TEM studies show its ability to resolve and show individual particles clearly, in an aggregation of particle mass. Therefore, TEM studies were performed for ZWD type materials, in which nanocrystal size (35-100 nm) type crystals were obtained from microwave synthesis. The TEM images shown in Fig. 3 for ZWD show nanoparticle size (~50-150nm).. Mica crystals were clearly formed by microwave method (fig. 3, ZWD). TEM images confirm that sodium zinnewaldite nanocrystals synthesized by microwave heating are highly crystalline and have a narrow particle size distribution (about 35nm).

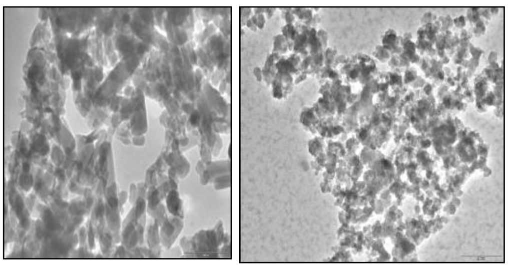
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Fig.3. TEM images for synthetic analogues of sodium zinnewaldite.

Also, scanning electron microscopy (SEM) images clearly show (Fig. 4) the size and crystal morphology of the synthesized samples for ZWD. The crystal sizes can be obtained by manually measuring crystals (10-200µm crystals averaged) in SEM images and taking the average as being representative of the mica mineral particle size, a shown in the SEM image. To clearly demonstrate the advantages of microwave method and hydrothermal method, XRD patterns of four selected samples are presented in figure 1 and the corresponding SEM micrographs are shown in figure 4. For the present microwave and hydrothermal method was selected, because each of these samples is one that is fully crystalline and has smallest crystal diameter in its own series.

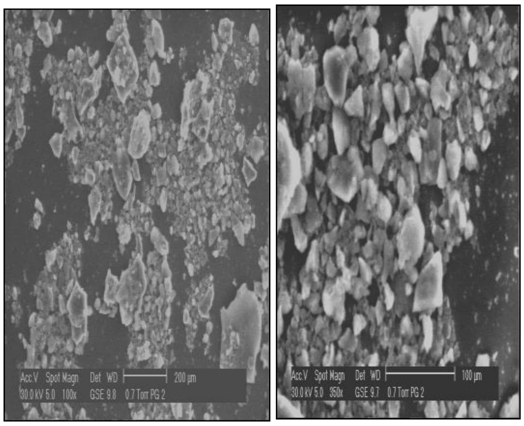

Fig.4. SEM images for synthetic analogues of sodium zinnewaldite.

The FT-IR spectrum of ZWD is shown by the Figure 5. The band at ~1036 cm-1 and ~ 835 cm-1 is due to Si-O-Si and Al-O-Al stretching respectively. A new broad band below 1639 cm-1can rea sonably be assigned to N-H deformation, vibration for amino salt appear as strong bands at ~ 1600-1574 cm-1 and also near 1640 cm-1. This suggests the presence of organic structure directing agents

4.0 Speciation of Metal Ions

The sorption behavior investigates MINTEQA-2 software of version 1.50 (Allison Geoscience) is used. Speciation has been carried out under normal simulated condition that is at zero ionic strength and at different pH (2-10) which was nearly meeting with the experimental conditions. From the speciation shows in Fig.12 shows it can be clearly seen that the dominating species at initial pH 2 and up to pH 8 are their cationic from that is Pb^{2+}, $Cd^{2+}$and As^{3+}. The formation of hydrolytic metal complexes such as Pb^{2+}, $PbOH^{+}$, $Pb_3(OH)_4^{2+}$, Cd^{2+}, $CdOH^{+}$, $Cd(OH)_2$ (aq) and H_3AsO_3, $H_2AsO_3^-$ etc may lead to significant decrease in solubility of metal ions over appropriate ranges at pH 6.0 & 8.0. This behavior may be due to the low complexing tendency of the metal ions. The main hydrolyzed species observed are Pb (OH)42-, Pb4 (OH)44+ and Pb(OH)2 (aq), Cd (OH)42-, Cd2(OH)3- and Cd (OH)3- as well as H4AsO3+, AsO3_ and HAsO3 2- respectively for Pb2+, Cd2+ and As3+ at the lower concentration of hydrolyzed species confirms that the sorbing species at entire pH range are cationic form of the metal ions shows in fig. 4 [18-22].

VI. CONCLUSION

Present study showed that the synthesized sodium zinnewaldite was found to be amorphous and crystalline in nature. XRD data show negligible effect of alkalinity. The particle size of the synthesized materials was analyzed by TEM analysis which corresponds to 35-100nm. The chemical composition of the synthesized powder was obtained from EDS which reflects the presence of K, F, Na, Si & Al which in turn with the weight percentage of these metals. Time of equilibration was found to be 10 h. with respect to all metal ions. Sorption was found to be pH dependent

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