Applied Clay Science 155 (2018) 30–36

Contents lists available at ScienceDirect

Applied Clay Science

journal homepage: www.elsevier.com/locate/clay

Research paper

Synthesis and characterization of novel nanocomposite by using kaolinite and carbon nanotubes

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ARTICLE INFO

Keywords:
Clay mineral
Nanocomposite
Kaolinite
Carbon nanotubes (CNT)
Adsorptive application

ABSTRACT

Environmental Pollution has increased tremendously since last few years as a result of urbanization leading to environmental, geological and global changes. There are numerous emerging pollutants which are toxic in nature. Since they may have critical environmental repercussions they have to be deactivated or mitigated by various technical means. Therefore, there is a need to develop new and novel smart material for sustainable environmental pollution control. Various types of clay and carbon materials are well known for their application in environmental protection using their properties of adsorption. However, they have been used separately. Keeping this thought, new material using kaolinite (kaol) and multiwalled carbon nanotubes (MWCNT) has been developed with the potential application in water treatment.

Kaol has been modified by chemical treatment method to get $-NH_2$ bondings as is evident from Fourier transform infrared (FT-IR) spectroscopy showing reflection at 1618 cm^{-1}. MWCNT were also modified by chemical route to develop −COOH bonding and it was confirmed by FT-IR reflection at 1726 cm^{-1}. Fabrication of nanocomposite by solution mixing method to get −CONH bonding was confirmed by the reflection at 1682 cm^{-1} on FT-IR spectrum. The structural properties of nanocomposites have been studied by X-ray diffraction (XRD). Mean size of crystalline structure was 32.46 nm for untreated kaol and 19.55 nm for nanocomposite. Simultaneously loss of intensity and widening of XRD reflection occurred due to change in crystalline to amorphous form. It was an indication of increase in amorphous form and reduction in crystallinity. It was evident from scanning electron microscopy (SEM), that clay- CNT nanocomposite was homogenized and has distinctive morphology. The average diameter of nanocomposite was ~20 nm, which was identified by high resolution transmission electron microscopy (HRTEM). Physical properties were also investigated using N_2 -adsorption isotherm study; pore size distribution and Brunauer–Emmett–Teller (BET) surface area. Adsorption properties of clay and nanocomposite were calculated and their BET surface area was found to be 9.13 $m^2 g^{-1}$ and 23.43 $m^2 g^{-1}$, respectively. Similarly, Langmuir surface area was 13.16 $m^2 g^{-1}$ and 33.95 $m^2 g^{-1}$, micropore area was 6.90 $m^2 g^{-1}$ and 22.11 $m^2 g^{-1}$ and total pore volume was 0.04 $cm^3 g^{-1}$ and 0.10 $cm^3 g^{-1}$, respectively.

1. Introduction

There is large addition of environmental contamination since last few decades subsequently because of industrialization, population migration from rural to urban areas, rapid growth of urban area, increasing traffic, agricultural, domestic activities and other environmental, geological and global changes. Everyday a challenge is recognized and attention is required for mitigation. Therefore, there is need to develop new and novel smart material for sustainable environmental pollution control. Various types of clay and carbon materials are well known for their application in environmental protection.

Kaolinite (kaol) was used as an adsorbent since long; it has triclinic crystalline two layer type equi-dimensional structure with the composition of SiO_2, Al_2O_3, H_2O with traces of elements like Mg, K, Fe, Ti, etc. (Murray, 2007; Choudhury, 2017). The presence of surface charge of clay was due to structure of clay, pH, cation exchange capacity (CEC) and swelling properties (Miranda-Trevino and Coles, 2003; Rouquerol et al., 2013). Clays are exceptionally fine particles containing chemical properties of colloids. The adsorption application of clay minerals was due to high specific surface area (SSA), high CEC, net negative charge on the surface of minerals, selectivity, regenerability, abundance, chemical and mechanical stability, and clay minerals layered structure, etc.

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https://doi.org/10.1016/j.clay.2017.11.043
Received 4 September 2017; Received in revised form 28 November 2017; Accepted 29 November 2017

Table 1
SSA and CEC of Kaol and its modified form.

Adsorbents	Origin of Kaol	SSA ($m^2 g^{-1}$)	Method of determination	CEC (m equiv. 100 g^{-1})	Method of determination	References
Untreated kaol	Columbia	3.8	Sears	11.30	Copper bisethylenediamine complex method	Bhattacharyya and Gupta (2006)
Acid activated kaol		15.6		12.20		
Kaol- TBA		14		3.9		
Kaol-ZrO		13.40		10.20		
Untreated kaol	Ubulu-Ukwu, Delta State, Nigeria	10.56	Sears	51.04	Ammonium acetate method of Chapman	Unuabonah et al. (2008)
Kaol-PVA		7.92		7.81		
Untreated Kaol	Kogi state, Nigeria	13.41	N_2 adsorption	11.26	Ammonium acetate method	Dawodu and Akpomie (2014)
		19.8	Methylene blue method			
Untreated Kaol	Rudnik, Slovakia	10.76	N_2 adsorption	–	–	Dankova et al. (2015)
Kaol- MnO_2		16.09				
Calcined kaol	Columbia	3.8	Sears	0.113	Copper bisethylenediamine complex method	Bhattacharyya and Gupta (2009)
Kaol- TBA		14.2		0.039		
Kaol	–	–	–	6.60	Copper EDTA complex	Bergaya and Vayer (1997)

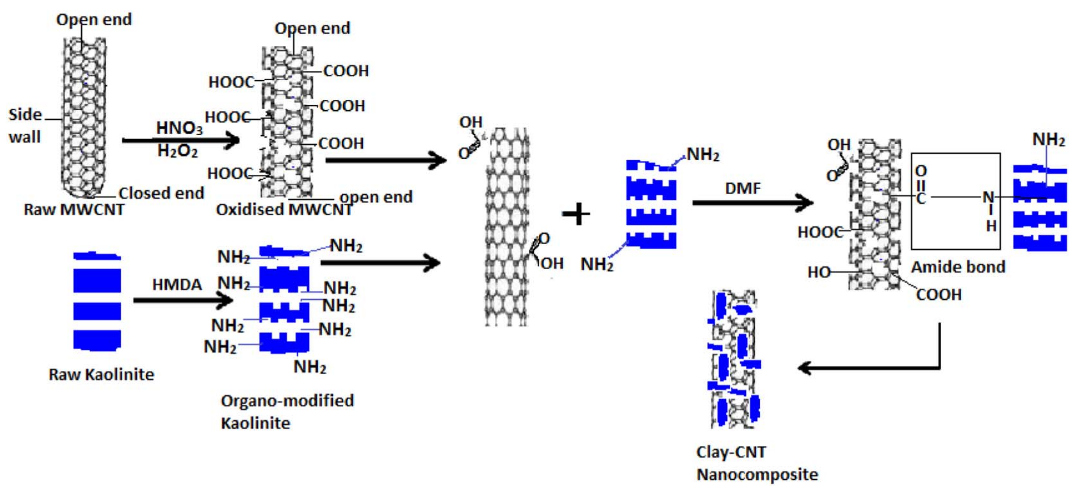

Fig. 1. Synthesis of nanocomposite by chemically treated clay and activated CNT.

(Murray, 2000; Gupta, 2009; Araujo et al., 2013; Ismadji et al., 2015; Mohellebi and Lakel, 2016; Soleimani and Amini, 2017; Rodrigues, 2003).

Rodrigues (2003) and Jiang et al. (2009) studied adsorption properties of kaol and showed that it increased to some extent after acid treatment. However, Jiang et al. (2009) concluded that there was 4.5 fold increase in adsorption capacity in comparison to untreated kaol. Recently, clay minerals have been modified by various methods to get enhanced adsorption properties such as SSA, surface functional group, CEC, pore volume, pore size, etc. (Suraj et al., 1998; Ijagbemi et al., 2010; Auta and Hameed, 2012; Yavuz and Saka, 2013; Adeyemo et al., 2017; Gao et al., 2015).

The SSA and CEC of untreated kaol and its modified form were reported by various researchers as tabulated in Table 1. SSA of untreated kaol increased from 3.8 $m^2 g^{-1}$ to maximum 19.8 $m^2 g^{-1}$ after modification. The CEC of kaol increased from 3.9 m equiv. 100 g^{-1} to maximum 51.04 m equiv. 100 g^{-1} (Dankova et al., 2015). The total pore volume of untreated kaol was 0.0037 $cm^3 g^{-1}$ and average pore diameter was 11.04 Å (Dawodu and Akpomie, 2014). Dankova et al. (2015) reported total pore volume to be 0.0775 $cm^3 g^{-1}$ with the adsorption capacity of 10–250 mg l^{-1}. Some researchers (Bhattacharyya and Gupta, 2006; Dawodu and Akpomie, 2014) have shown the maximum removal efficiency of Ni (II) from aqueous solution to be 166.67 mg g^{-1}.

However, in actual environmental conditions it is desirable to use well efficient material for removal of trace level pollutants. Lots of materials were already available for adsorption of various pollutants with their limitations. However, there was need to develop more efficient material to get better adsorption capacity when it is in trace (parts per trillion) levels.

Therefore, in the present study a new material has been developed with enhanced adsorption properties like SSA, average pore diameter, micropore area and total pore volume of clay. This has been achieved by using novel material like carbon nanotubes (CNT) along with clay, as it possesses high surface area after modification (Stafiej and Pyrzynska, 2007; Zhao et al., 2010; Gupta et al., 2014). Experimentation involved along with results have been discussed in this research paper.

2. Experimental

2.1. Materials

Kaol and CNT were purchased from M/s Sigma. Kaol and multi-walled carbon nanotubes (MWCNT) have average particle size of 5–45 nm and 10–50 nm, respectively, as mentioned by Sigma. Other chemicals (analytical grade) like acids, hexamethylenediamine, ethanol, hydrogen peroxide, dimethyl formamide (DMF) etc. were purchased from Merck and Fisher Scientific. Acids were further distilled to get high level of purity and used in these experiments.

2.2. Preparation of clay- CNT nanocomposite

Firstly, activation of kaol was carried out by chemical treatment as reported by Sedaghat (2013). 1.0 g clay was mixed in 40 ml of DI (De-ionized) water and heated to 80 °C for 15 min. After this 0.04 g of