Applied Clay Science 107 (2015) 98–108

Contents lists available at ScienceDirect

Applied Clay Science

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

Research paper

# Synthesis of Cu–Al layered double hydroxide nanofluid and characterization of its thermal properties

Samarshi Chakraborty [a], Ishita Sarkar [a], Krishnayan Haldar [a], Surjya Kanta Pal [b], Sudipto Chakraborty [a,*]

[a] Department of Chemical Engineering, Indian Institute of Technology, Kharagpur, India
[b] Department of Mechanical Engineering, Indian Institute of Technology, Kharagpur, India

ARTICLE INFO

*Article history:*
Received 22 September 2014
Received in revised form 8 January 2015
Accepted 12 January 2015
Available online 31 January 2015

*Keywords:*
Cu–Al LDH
Nanofluid
Thermal conductivity enhancement
Stability analysis

ABSTRACT

Synthesis of pristine Cu–Al layered double hydroxide (LDH) nanofluid via one step method and study of its thermal properties are the core essence of the current work. Nitrate salts of Cu, Al and Na were mixed in a particular molar ratio at constant pH to produce desired Cu–Al LDH. Different dispersion techniques were utilized to uniformly disperse Cu-Al LDH in water to obtain Cu–Al LDH nanofluids. Broadly used characterization techniques were implemented to identify and characterize pristine Cu–Al LDH nanoparticle. These techniques were used to determine crystallite size, composition, morphology and characteristics vibration of interlayer anion present in the nanoparticle. The nanofluids were characterized for particle size, cluster size, surface tension and thermal conductivity. Particle size analysis was carried out to confirm the formation of nanofluid. Dynamic light scattering (DLS) method had been employed to measure the clustering tendency of nanofluid. Effect of nanofluid loading on thermal conductivity was studied in depth. Influence of particle size, shape and composition on thermal conductivity of nanofluid had also been selected as an essential topic of investigation. Zeta potential and visual phase separation study were carried out to measure the stability of concerned nanofluid.



## 1. Introduction

Among the few selected positively charged layered compounds, layered double hydroxide, also known as hydrotalcite, had achieved a lot of attention. Layered Double Hydroxides (LDH) known as a brand of ionic layered material composed of positively charged brucite type sheets. It contains anionic counter ions and solvent molecules in the interlayer space. LDH is denoted by the common chemical formula: $[M^{2+}{}_{1-x} M^{3+}{}_{x}(OH)_2]^{x+}(A^{m-})_{x/m}$ x $yH_2O$, where, $M^{2+}$ is a divalent cation (such as $Ni^{2+}$,$Mg^{2+}$,$Cu^{2+}$,$Zn^{2+}$), $M^{3+}$ is a trivalent cation (such as $Cr^{3+}$, $Al^{3+}$), A is an interlayer anion with m− charge, x and y are fraction constants (Wang and O'Hare, 2012). LDH is also identified as anionic clay comprising of closely filled planes of hydroxyl anion that lies on top of triangular lattice. LDH could be prepared via many ways such as co-precipitation (Sahu and Pugazhenthi, 2011), anionic exchange (Kooli et al., 1997; Rocha et al., 1999), reconstruction method based on memory effect (Chibwe and Jones, 1989) and hydrothermal/microwave treatment (Benito et al., 2006). Application of LDH includes a much diversified spectrum due to its tuneable properties such as acid shielding effect, superior adsorption ability, anion exchange skill, thermal stability, flame retardant and gas barrier potential. Pharmaceutical and cosmetic application (Choy et al., 2007), and use of LDH for polymer nanocomposite synthesis (Chen and Qu, 2003; Krishna and Pugazhenthi, 2012) were owed to such properties. It was also used for several other purposes such as heterogeneous catalysts (Rives et al., 2003; Jinesh et al., 2010), drug delivery hosts (Rives et al., 2014). According to this literature survey, potential of LDH as a heat transfer fluid was unexplored. Thermal properties of LDH such as thermal conductivity, surface tension and stability were overlooked so far.

Nanofluids are colloidal solutions of nanoparticles (with an average particle size of 100 nm or less in at least one dimension) suspended in base fluid to improve the thermal performance (Mahbubul et al., 2014). Use of nanofluids in heat transfer application was widely investigated by many researchers. Stephen U.S. Choi (Choi, 1995) first invented nanofluid as energy efficient heat transfer fluid via dispersing metallic nanoparticles in traditional base fluid. The resulting fluid had displayed excellent improvement in thermal conductivity when compared with common base fluid. Scientists working on the field of heat transfer used nanoparticles in traditional base fluids like water, ethylene glycol and engine oil due to their potential benefit and applications in microelectronics, energy supply, transportation and metallurgical applications. Thermal conductivity of nanofluids changes with shape, size and composition of nanoparticle. Wang and his co-workers (Wang et al., 1999) determined the thermal conductivity of CuO and $Al_2O_3$

*Abbreviations:* DLS, Dynamic Light Scattering; EDS, Energy Dispersive X-Ray Spectroscopy; FTIR, Fourier Transform Infrared Spectroscopy; JCPDS, Joint Committee on Powder Diffraction Standards; LDH, Layered Double Hydroxide; PDI, Polydispersity Index; SEM, Scanning Electron Microscope; TEM, Transmission Electron Microscope; XRD, X-Ray Diffraction.

* Corresponding author at: Chemical Engineering Department, IIT Kharagpur, Kharagpur-721302, West Bengal, India. Tel.: +91 3222 283942 (office).
*E-mail address:* sc@che.iitkgp.ernet.in (S. Chakraborty).

http://dx.doi.org/10.1016/j.clay.2015.01.009
0169-1317/

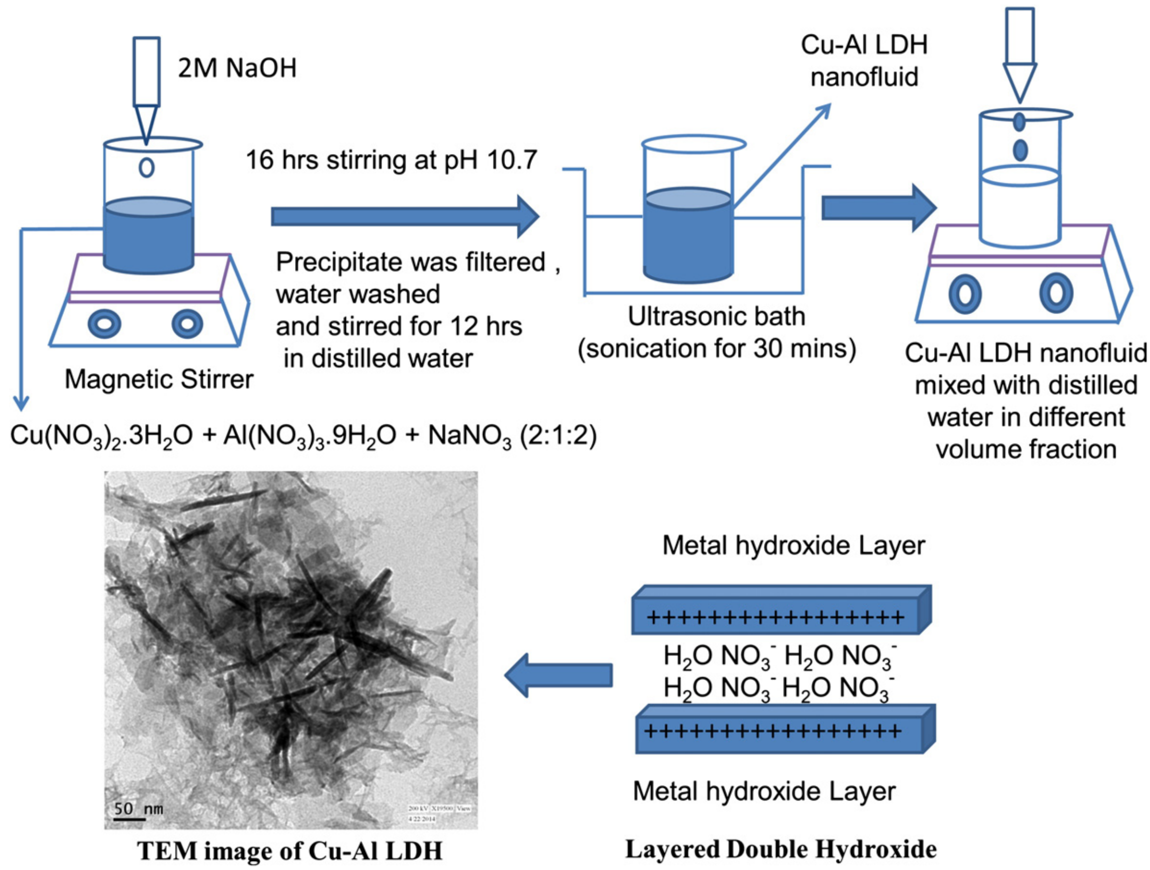


**Fig. 1.** Schematic diagram of Cu–Al LDH nanofluid preparation.

nanoparticle in different base fluids. They studied the influence of particle size and dispersion method on thermal conductivity. Similarly, Xie et al. (Xie et al., 2002) evaluated thermal conductivity of $Al_2O_3$ nanofluid having particle size in the range of 12–302 nm. Metal oxide itself does not display extraordinary thermal conductivity although it can improve the thermal conductivity of base fluids in nano suspension. Most of the work reported on the nanofluid synthesis had mainly employed two step synthesis techniques. In step by step techniques, nanoparticle synthesis was carried out first. Finally, nanoparticle was dispersed into the base fluid to prepare the nanofluid. This method had a major drawback because it leads to particle agglomeration. Particle agglomeration was found to be detrimental for nanofluid stability and its thermal properties (Zhu et al., 2004).

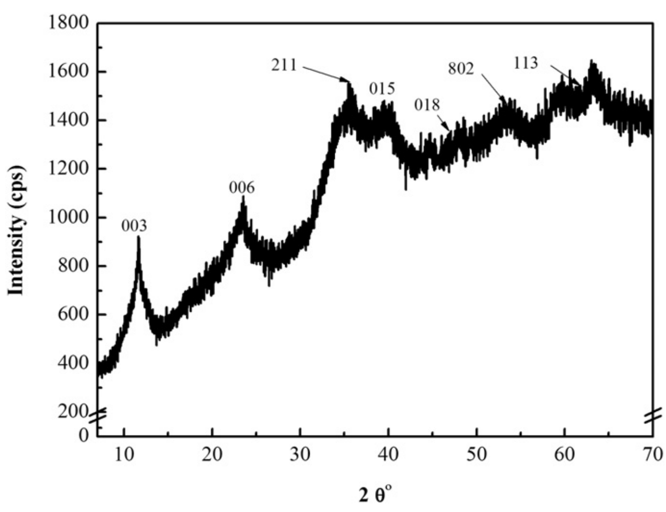


**Fig. 2.** XRD analysis of pristine Cu–Al LDH nanoparticle.

According to existing literature, no attention had been given on the potential of LDH as nanofluid. Thermal conductivities of copper (Cu) and aluminum (Al) are significantly high as compared to other cheap metals. Previously heat transfer studies had been carried out on CuO and $Al_2O_3$ nanoparticles separately. LDH possesses a dynamic chemical structure, which allows combining Cu and Al together, without any calcination. In this paper, Cu–Al LDH nanofluid was prepared via one-step method using the nitrate salts of Cu and Al in the molar ratio of 2:1, respectively. Different characterizations were carried out to determine crystal size, composition, dispersion, particle size, shape, stability, surface tension and thermal conductivity of Cu–Al LDH nanofluid and nanoparticle.

## 2. Experimental facility

### 2.1. Raw materials

Copper nitrate ($Cu(NO_3)_2.3H_2O$), Aluminum nitrate ($Al(NO_3)_3.9H_2O$), Sodium nitrate ($NaNO_3$) and Sodium hydroxide (NaOH) of analytical grade were procured from Merck, India. Distilled water had been used throughout the preparation period.

### 2.2. Synthesis of Cu–Al LDH nanofluid

One step method was implemented for nanofluid synthesis. Co-precipitation method was used to prepare pristine Cu–Al LDH solution. Fig. 1 shows Cu–Al LDH preparation technique. In this present work, two solutions were initially prepared. Solution 1 comprised of aqueous solution of $Cu(NO_3)_2.3H_2O$, $Al(NO_3)_3.9H_2O$ and $NaNO_3$ had been prepared by mixing the nitrate salts in the molar ratio of 2:1:2, respectively.