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Research paper

Synthesis and characterization of Zn-Al layered double hydroxide nanofluid and its application as a coolant in metal quenching

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ABSTRACT

The current study comprises of the synthesis and characterization of Zn-Al layered double hydroxide (LDH) nanofluid for use as a potential coolant in the steel industry. Coprecipitation method was used in the preparation of Zn-Al LDH nanoparticles by using the nitrate salts of Zn, Al and Na, in the ratio of 2:1:2. The nanofluids were characterized based on their particle size, stability, surface tension, thermal conductivity and viscosity. TEM analysis revealed that the nanoparticles were needle-shaped with an average particle size of 45.61 nm and aspect ratio of 7.3. Different concentrations of the nanofluid, from 40 ppm to 240 ppm, were used to analyze the effect of particle concentration on the enhancement in thermal conductivity and viscosity. Stability analysis of the nanofluid exhibited good results, even without any stabilizer. An optimum cooling rate of 125 °C/s was obtained for a nanofluid concentration of 160 ppm, which is 1.29 times when compared to water.

1. Introduction

Taking into account the limited heat transfer potential of conventional coolants, and with the possibility of improved thermal efficacy requisite for various processes, nanofluids have become the focus of a rising number of heat transfer industries (Das et al., 2006). An improvement in the thermal conductivity of the base fluid is brought about by adding nanoparticles in small amounts (Chol, 1995; Eastman et al., 1996; Xuan and Li, 2000; Eastman et al., 2001; Murshed et al., 2008). Nanoparticles of metals, metal oxides, metal nitrides and metal carbides along with semiconductors and carbon nanotubes (CNT) have been added to conventional coolants like ethylene glycol (EG), water and poly- α -olefin oil (PAO) to substantially enhance the thermal conductivity (Yu et al., 2008). Researchers have estimated the effective thermal conductivity of nanofluids containing Al₂O₃ nanoparticles and found it to be enhanced by 30% at a relatively low volume fraction of 4.3% (Masuda et al., 1993). In the case of TiO₂ nanoparticles of diameters 10 nm and 15 nm having a maximum volume fraction of 5%, the enhancement was observed to be approximately 33% and 30%, respectively (Murshed et al., 2005). Authors have also investigated the temperature dependence of thermal conductivity for Al2O3 and CuO based nanofluids, with the results indicating an increase in enhancement characteristics. Particle volume fraction and temperature were found to have a direct effect on the thermal conductivity enhancement,

whereas particle size had an inverse relation on the effective enhancement (Mintsa et al., 2009). Studies have revealed that the effective thermal conductivity enhancement is primarily the result of the localized convection induced by the Brownian motion of the nanoparticles (Prasher et al., 2006). Suspended nanoparticles were found to enhance the rate of forced convection heat transfer (Trisaksri and Wongwises, 2007). Also, the interaction between the suspended nanoparticles and the plate surface had an effect on the heat transfer rate and CHF value (Pinto and Fiorelli, 2016).

A system which is relatively new in the field of heat transfer research and has not been analyzed for its thermal characteristics is layered double hydroxide (LDH). Layered double hydroxide (LDH) is defined by the general formula $[M_{II}^{II}_{-a}M_{a}^{III}(OH)_{2}]^{a+}$ ($A^{m}_{-})_{a/m}$ a bH₂O, where M^{II} represents a divalent cation such as Zn^{2+} , Cu^{2+} , Mg^{2+} , M^{III} a trivalent cation for eg., Al^{3+} , Cr^{3+} ; A^{m-} an anion like NO₃⁻, CO_3^{2-} and a and b the fraction constants (Seftel et al., 2008; Wang and O'Hare, 2012). The structure comprises of brucite-like layers containing edge-sharing metal hydroxide octahedral (Del Arco et al., 1999; Intissar et al., 2003). The positively charged metal hydroxide sheets have anions within the layers, to compensate the charge on the layers together with water molecules (Reichle, 1986; Choy et al., 1999). The dynamic chemical structure of LDH favours the combination of the divalent and trivalent cations together without any calcination (Chakraborty et al., 2015). The representative structure of LDH in

Abbreviations: LDH, layered double hydroxide; XRD, X-ray diffraction; DAQ, data acquisition; CHF, critical heat flux; HTC, heat transfer coefficient

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Fig. 1. Representative structure of LDH.

general is illustrated in Fig. 1, wherein the present study M^{II} represents Zn^{2+} , $M^{III} Al^{3+}$ and A^{m-} represents NO_3^{-} .

Jet impingement cooling has been widely used in several high heat flux applications, both in free-surface and submerged setups (Wolf et al., 1993). A major advantage of this cooling mechanism is the ability to dissipate heat flux at the higher temperature range of the cooling spectrum (Wang et al., 2012). In comparison to laminar cooling where low pressure impingement results in lower cooling rates (up to 80 °C/s), forced jet cooling is found to achieve higher cooling rates (Ravikumar et al., 2014b). The cooling rate depends on the thermo physical properties of the coolant, type of jet, and the surface condition of the specimen.

From the authors' previous work, LDH based nanofluid was found to display an appreciable increase in thermal conductivity, making it suitable for use in different cooling methodologies (Tiara et al., 2016). The present work deals with the heat transfer studies using Zn-Al LDH nanofluid jet in the case of an AISI 304 steel plate at different concentrations. As zinc and aluminum also have comparatively higher values of thermal conductivity in comparison to as compared to other low cost metals, it can be used as the di- and trivalent cations for LDH synthesis. Once optimized, the nanofluid can then be used along with different additives to study their effects on the rate of cooling. A coprecipitation method was adopted for the synthesis of Zn-Al LDH nanofluid, achieved by the gradual addition of two metal nitrates solutions, namely Zn(NO₃)₂·6H₂O and Al(NO₃)₃·9H₂O. Coprecipitation method employed in this work involves modification of LDH as a pretreatment step to make it more compatible (Sahu and Pugazhenthi, 2011).

2. Nanofluid characterization and experimental setup

2.1. Preparation and characterization of nanofluid

Zn-Al LDH nanofluids were prepared by co-precipitation method (Sahu and Pugazhenthi, 2011) by using two metal nitrate solutions (Zn $(NO_3)_2$ ·6H₂O and Al $(NO_3)_3$ ·9H₂O) and NaNO₃ in the mole ratio of 2:1:2, after which a 2 M NaOH solution was added to the solution dropwise and stirred continuously. Once the pH level reaches 10.7, the subsequent solution was stirred at room temperature for 16 h. It was then filtered, and the precipitate obtained was washed several times with distilled water till the pH level reaches 7 (Sahu and Pugazhenthi, 2011). The final nanofluid was prepared by dispersion of the neutral filtrate into the base fluid (water) and stirring it for 12 h. The nanofluid was sonicated (40 kHz ultrasonic bath) for around 15–30 min before its subsequent usage as a coolant. The concentrations of the final nanofluid



Fig. 2. XRD analysis of pristine Zn-Al LDH nanoparticles.

were varied from 40 ppm to 240 ppm to optimize its thermal properties.

2.1.1. XRD analysis

X-ray diffraction (XRD) analysis was carried out to characterize the crystalline nature of the sample (see Fig. 2). A High-Resolution XRD (PANalytical) with a scan speed of 0.5 s, where Ni-filtered Cu K α radiation was generated with a λ value of 1.5406 Å, was employed for the analysis. The crystalline size and the basal spacing based on the peak value (mentioned in Table 1) were calculated using the PANalytical X'Pert HighScore Plus software. The average crystallite size D_p was calculated using the Scherrer equation as given below (Monshi et al., 2012; Chakraborty et al., 2015).

$$D_{p} = \frac{0.94\lambda}{\beta\cos\theta} \tag{1}$$

where β is the line broadening at half the maximum intensity (FWHM), λ the X-ray wavelength and θ the angle of incidence ($2\theta = 5-70^{\circ}$).

The crystal size obtained from the table was compared with the data available from literature (JCPDS 037-0630) (Song et al., 2013). The crystallinity of solid state nanoparticles was found to affect its thermal conductivity (Zhu et al., 2006; Gangwar et al., 2014).

2.1.2. Size analysis

The shape of particles/aspect ratio significantly affects the thermal performance of the nanofluid solution (Yang et al., 2005; Ding et al., 2006). A heat transfer enhancement of over 290% was found in the case of needle-shaped nanoparticles in comparison to spherical particles (Xuan and Li, 2003; Wen and Ding, 2004; Ding et al., 2006). Also, rod-shaped nanoparticles were found to exhibit an enhanced thermal conductivity when compared to spherical particles (Murshed et al., 2005). Also, as the shape factor *n* (ratio of surface area of the particle to that of a spherical particle) influences the thermal properties of the nanofluid, elongated particles (n > 3) have an enhanced thermal conductivity (Keblinski et al., 2002). TEM image and particle size histogram of the nanoparticles at a specific location are shown in Fig. 3(a) and (b). ImageJ software (Tiara et al., 2016) was used for

 Table 1

 Crystal size analysis of Zn-Al LDH nanoparticles using XRD.

h k l	2θ (degree)	d-spacing (nm)	Crystal size (rounded off) (nm)
(003)	11.88	0.74	26
(006)	23.69	0.37	26
(211)	34.83	0.26	34
(015)	39.44	0.23	30
(802)	53.24	0.17	26
(110)	60.33	0.15	31
(113)	61.77	0.15	33

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