Effect of processing conditions on sonochemical synthesis of nanosized copper aluminate powders

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ABSTRACT

Nanosized copper aluminate (CuAl$_2$O$_4$) spinel particles have been prepared by a precursor approach with the aid of ultrasound radiation. Mono-phasic copper aluminate with a crystallite diameter of 17 nm along the (3 1 1) plane was formed when the products were synthesized using Cu(NO$_3$)$_2$·6H$_2$O and Al(NO$_3$)$_3$·9H$_2$O as starting materials, with urea as a precipitation agent at a concentration of 9 M. The reaction was carried out under ultrasound irradiation at 80 °C for 4 h and a calcination temperature of 900 °C for 6 h. The synthesized copper aluminate particles and the effect of different processing conditions such as the copper source, precipitation agents, sonochemical reaction time, calcination temperature and time were analyzed and characterized by the techniques of powder X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), atomic force microscopy (AFM) and Fourier transformation infrared spectroscopy (FT–IR).

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1. Introduction

In general, aluminate spinels show high thermal stability, high mechanical resistance, hydrophobicity and low surface acidity [1–4]. These properties make them interesting materials as catalysts and carriers for active metals to replace current technology. Copper aluminate spinel is known to be active in the degradation of some organic compounds, and is commonly prepared by high temperature calcination of mixed aluminium and copper oxides [5,6], or is the product of impregnating a porous alumina having a high surface area with a solution of copper compound [7,8]. However, to attain complete reaction, a temperature of above 1000 °C has to be maintained for several days. Recently, MAl$_2$O$_4$ (M = Cu, Zn, Mg, Ni and Li) has been prepared by the following methods: co-precipitation [9,10], hydrothermal synthesis [11–13] and sol–gel methods [14–16]. The disadvantages of solid-state routes, such as inhomogeneity, lack of stoichiometry control, high temperature and low surface area, are improved when the material is synthesized utilizing a solution-based method. The high surface area of CuAl$_2$O$_4$ is of prime importance for catalytic purposes. Typically, high surface areas go along with small particle sizes. Hence, the fabrication of nanosized copper aluminate particles will be worthwhile to study.

The extreme conditions such as high temperatures (>5000 K), pressures (>20 Mpa) and cooling rates (>10^{10} Ks^{-1}) attained during acoustic cavitation lead to many unique properties in the irradiated solution, and these conditions have been effectively used to prepare nanoscale metals, metal oxides and nanocomposites [17–20]. The sonochemical method has some advantages, including uniformity of mixing, reduction of crystal growth and morphological control. Ultrasound can also fracture agglomerates to produce a uniform composition of products [21]. The use of ultrasound radiation during the homogeneous precipitation of the precursor is expected to reduce the precipitation time of the precursor and to ensure homogeneity of the cations in the precursor [22].

Previous authors [23–27] have synthesized a series of aluminates or ferrites using the sonochemical method. In the present study, nanosized copper aluminate (CuAl$_2$O$_4$) spinel particles have been prepared by a precursor approach with the aid of ultrasound radiation. The synthesized copper aluminate particles and effect of different processing conditions, such as the copper source, precipitation agents, sonochemical reaction time, calcination temperature and time, were analyzed and characterized by powder X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), atomic force microscopy (AFM) and Fourier transformation infrared spectroscopy (FT–IR). The structural changes of the aluminate materials were investigated by the XRD methods, which were applied in the assessment of nanosized copper aluminate powders preparation as well.

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2. Experimental

All the reagents were of reagent grade purchased from Guangzhou Chemical Reagents Factory (Guangzhou, China), and were used without further purification. The synthesis of the precursor to nanosized $CuAl_2O_4$ was carried out with the aid of ultrasound radiation. First, the precipitation agents, Cu-salt and $Al(NO_3)_3 \cdot 9H_2O$ in stoichiometry were added in a sonication flask, and formed a solution. Secondly, the solution was purged with argon for 15 min and irradiated with high-intensity ultrasound radiation under argon at 80 °C for a pre-determined time by employing a direct immersion horn. The pH of the solution changed from 6.1 prior to the sonication to 7.0 at the end of the precipitation. After the precipitation reaction was complete, the precipitate was separated from the solution by centrifugation, washed repeatedly with distilled water and ethanol, and was finally dried in a vacuum for 10 h at 110 °C to be the precursor for $CuAl_2O_4$. The dried precursor was calcined at a pre-determined temperature for a pre-determined time to become nanosized $CuAl_2O_4$ particles.

Sonication was performed using a SC-4 high-intensity ultrasonic reactor (Chengdu Jiuzhou Ultrasound Factory, China; 0.6 cm tip diameter, with a titanium horn operating at 20.81 kHz at a power of 100 Wcm^{-2}) at room temperature. The powder X-ray diffraction patterns were recorded on a Bruker X-ray diffractometer (Model D/max-3A, CuKα, λ = 1.5418 Å). The morphologies of the products were examined by scanning electron microscopy (SEM, JSM-5910), transmission electron microscopy (TEM, JEM-100 CXII) and atomic force microscopy (AFM, CSPM5000, Being Nano-Instruments, China). The Cu/Al atomic ratios of the products were examined using a SEM with electron dispersive X-ray (EDX) analysis. A carbon layer was vacuum–vapor-deposited onto the samples to provide a conducting surface. FT–IR spectra were recorded on a Perkin–Elmer 1730 (American) infrared spectrophotometer with KBr pellets in the range of 400–4000 cm^{-1}.

3. Results and discussion

3.1. Powder X-ray diffraction (XRD) analysis

Fig. 1 shows the XRD pattern of the nanosized copper aluminate which was synthesized using $Cu(NO_3)_2 \cdot 6H_2O$ and $Al(NO_3)_3 \cdot 9H_2O$ as starting materials, with urea as a precipitation agent at a concentration of 9 M, ultrasound irradiation at 80 °C for 4 h and a calcination temperature of 900 °C for 6 h. It shows the characteristic XRD pattern of $CuAl_2O_4$. If the precursor is heated at a temperature lower than 900 °C, a mixture of CuO and $CuAl2O_4$ is obtained. The principal d values taken from the JCPDS 01-078-0556 for $CuAl_2O_4$ are 2.8564, 2.4359, 2.0198, 1.6491, 1.5548, 1.4282, 1.2774 and 1.1661 Å. In this study, XRD peaks for the $CuAl_2O_4$ nanoparticles have been observed at d = 2.8504, 2.4328, 2.0148, 1.6463, 1.5547, 1.4259, 1.2763 and 1.2284 Å, which are in agreement with the JCPDS values. On the basis of the Debeye–Scherrer equation, the crystallite size was calculated to be ca. 17 nm using the (3 1 1) reflection at d = 2.4328 Å. The average crystallite size in this case was found to be ca. 17 nm. No peaks attributable to alumina are present in the XRD pattern of the nanoparticles of the copper aluminate produced.

3.2. Scanning electron microscopy (SEM)

Fig. 2 shows the results of scanning electron microscopy of the precursor of $CuAl_2O_4$. From Fig. 2, the precursor exits lots of aggregation, and the mean pore size is above 3 μm. Fig. 3 illustrates the results of scanning and transmission (inset Fig. 3) electron microscopic observation of the nanosized $CuAl_2O_4$. The SEM and TEM micrographs show that the particle size is ca. 18 nm with little aggregation. This value is in accordance with that obtained from XRD measurements.
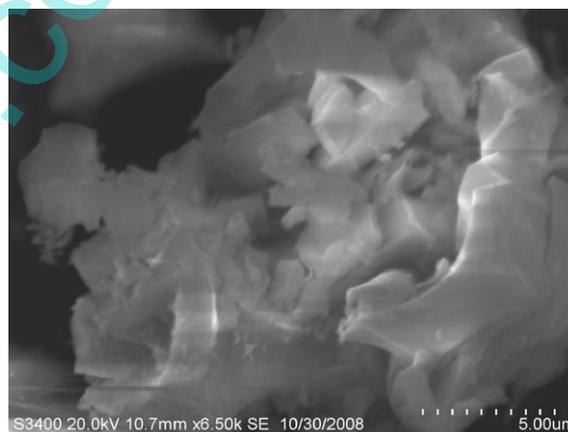

Fig. 2. SEM image of the precursor of $CuAl_2O_4$ nanosized powders.

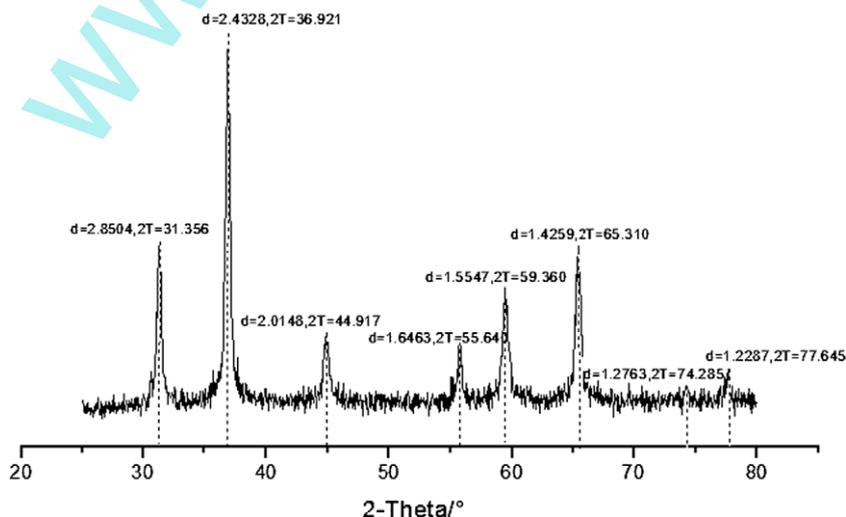**Fig. 1.** XRD patterns of $CuAl_2O_4$ nanosized powders synthesized using $Cu(NO_3)_2 \cdot 6H_2O$ and $Al(NO_3)_3 \cdot 9H_2O$ as starting materials, with urea as a precipitation agent at a concentration of 9 M, ultrasound irradiation at 80 °C for 4 h and calcination temperature 900 °C for 6 h.
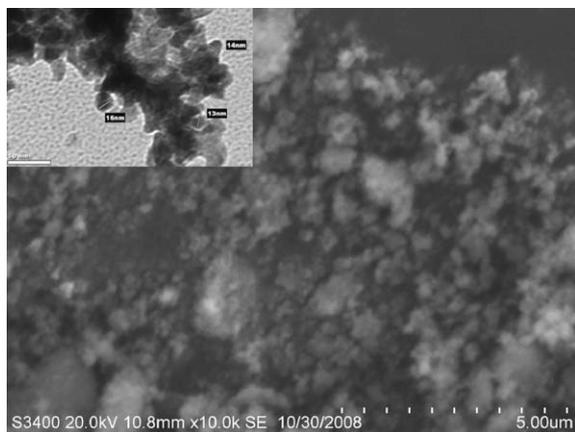
Fig. 3. TEM image of the CuAl$_2$O$_4$ nanosized powders (the inset shows the TEM image of product).

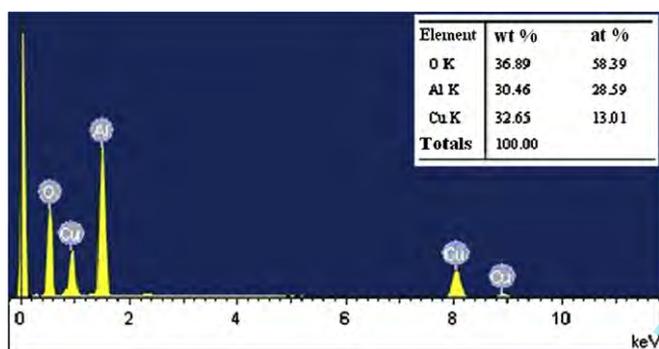**Fig. 4.** EDX of CuAl$_2$O$_4$ nanosized powders.

Fig. 4 shows the results of the EDX (energy dispersive X-ray). From the elemental analysis in combination with the results obtained from EDX, the stoichiometry of the precursor heated at 900 °C for 6 h was found to be 13.01% (atom percent) of Cu, 28.59% of Al and 58.39% of O. This result theoretically agreed with the atom percent (Cu 14.29%, Al 28.57% and O 57.17%). From the EDX analysis, the atomic ratio of aluminium to copper of the CuAl$_2$O$_4$ produced was found to be 2.18. The non-stoichiometry of the nanoparticles may be accounted for by the formation of the surface CuO. As no bulk CuO was observed by XRD, it is presumed that the domains of CuO are very small or located at the surface of the CuAl$_2$O$_4$ particles.

3.3. Atomic force microscope (AFM)

The nanosized CuAl$_2$O$_4$ powders were characterized by atomic force microscopy in tapping mode. The scanning area of the sample was 5×5 μm. The sample was prepared by employing diffusion in 95% ethanol for 15 min under ultrasonic radiation and then coating it on a muscovite substrate and dried for 30 min in the atmosphere. Fig. 5 shows the AFM results of nanosized CuAl$_2$O$_4$. Fig. 5A depicts a clear topographic image of the nanosized CuAl$_2$O$_4$ powders. It is seen that a clear topographic image was obtained, and that the CuAl$_2$O$_4$ products are nanosized powders and the particles size is uniform at about 20 nm. The three-dimensional view of the same sample (Fig. 5B) illustrates that the particles size of the CuAl$_2$O$_4$ powders were in the range of 10.8–21.5 nm with a narrow size distribution. The morphological characteristics of CuAl$_2$O$_4$ powders
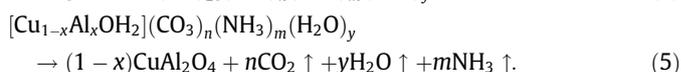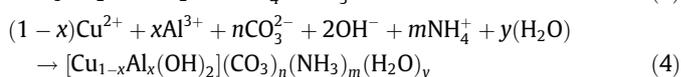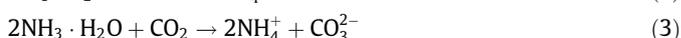
observed from the AFM images were consistent with their TEM images.

3.4. Infrared spectroscopy

Fig. 6 shows the IR spectra of the precursor as well as the CuAl$_2$O$_4$ nanoparticles. The products were synthesized using Cu(NO$_3$)$_2$· 6H$_2$O, Al(NO$_3$)$_3$·9H$_2$O as starting materials, with urea as a precipitation agent at a concentration of 9 M. The reaction was carried out at 80 °C for 4 h under ultrasound irradiation and a calcination temperature of 900 °C for 6 h, with a urea concentration of 9 M. The as-formed precursor shows the spectral features (Fig. 6a) typical of LDH, which are described in detail elsewhere [28]. The broad band at ca. 3417 cm^{-1} can be attributed to the hydroxyl groups which are extensively hydrogen bonded. The band at ca. 1645 cm^{-1} is assigned to the bending vibrational mode of the interlayer water molecules. The weak absorption band at ca. 2190 cm^{-1} has been attributed to the existence of intercalated NH$_3$ species as reported in the literature [29]. The IR spectrum provides the evidence for the presence of intercalated carbonate ions. The strong absorption band at ca. 1390 cm^{-1} originates from the v_3 mode of CO$_3^{2-}$ ion. The IR bands observed below 1000 cm^{-1} can be attributed to the v_2 mode of carbonate and the M–OH modes (M = Cu and Al). The precursor after heating at 900 °C shows IR peaks at 3412, 582 and 461 cm^{-1} (Fig. 6b). Additionally, there are two peaks between 450–800 cm^{-1}. This indicates that the product obtained by sonochemical method was a spinel.

3.5. The mechanism of the formation of copper aluminate

According to the results of infrared spectroscopy, the mechanism of the formation of copper aluminate is given by Eqs. (1)–(5). The process includes four steps. First, urea was hydrolyzed and ammonia was obtained. Secondly, OH$^-$ was formed from the ammonia. Then CO$_3^{2-}$ and [Cu$_{1-x}$Al$_x$(OH)$_2$](CO$_3$)$_n$(NH$_3$)$_m$(H$_2$O)$_y$ were yielded in turn. Finally, nanosized CuAl$_2$O$_4$ was synthesized after the calcination of [Cu$_{1-x}$Al$_x$(OH)$_2$](CO$_3$)$_n$(NH$_3$)$_m$(H$_2$O)$_y$.

$$CO(NH_2)_2 + 3H_2O \rightarrow CO_2 \uparrow + 2NH_3 \cdot H_2O \tag{1}$$

$$NH_3 \cdot H_2O \rightarrow OH^- + NH_4^+ \tag{2}$$

$$2NH_3 \cdot H_2O + CO_2 \rightarrow 2NH_4^+ + CO_3^{2-} \tag{3}$$

$$(1-x)Cu^{2+} + xAl^{3+} + nCO_3^{2-} + 2OH^- + mNH_4^+ + y(H_2O)$$
$$\rightarrow [Cu_{1-x}Al_x(OH)_2](CO_3)_n(NH_3)_m(H_2O)_y \tag{4}$$

$$[Cu_{1-x}Al_xOH_2](CO_3)_n(NH_3)_m(H_2O)_y$$
$$\rightarrow (1-x)CuAl_2O_4 + nCO_2 \uparrow + yH_2O \uparrow + mNH_3 \uparrow. \tag{5}$$

3.6. Effect of precipitation agents

The precipitation agents are used in all preparations of aluminates by the chemical precipitation method. Many researchers have used alkali metal hydroxides, namely KOH or NaOH [30–34]. To compare the composition of copper aluminate using the different precipitation agents, we chose NaOH, NaCO$_3$ and urea as the precipitation agents, respectively, without changing any other parameters (as in Section 3.1). Fig. 7 shows the XRD patterns of copper aluminate prepared by using the different precipitation agents. The result shows that the XRD pattern of copper aluminate prepared by using aqueous urea solution as a precipitation agent agrees well with the standard data of JCPDS (JCPDS 01-078-0556). The principal d values taken from the JCPDS 01-078-0556 for CuAl$_2$O$_4$ are 2.8564, 2.4359, 2.0198, 1.6491, 1.5548, 1.4282, 1.2774 and 1.1661 Å. In this study, XRD peaks for the CuAl$_2$O$_4$ nanoparticles have been observed at d = 2.8504, 2.4328, 2.0148, 1.6463, 1.5547, 1.4259, 1.2763 and 1.2284 Å, which are in close
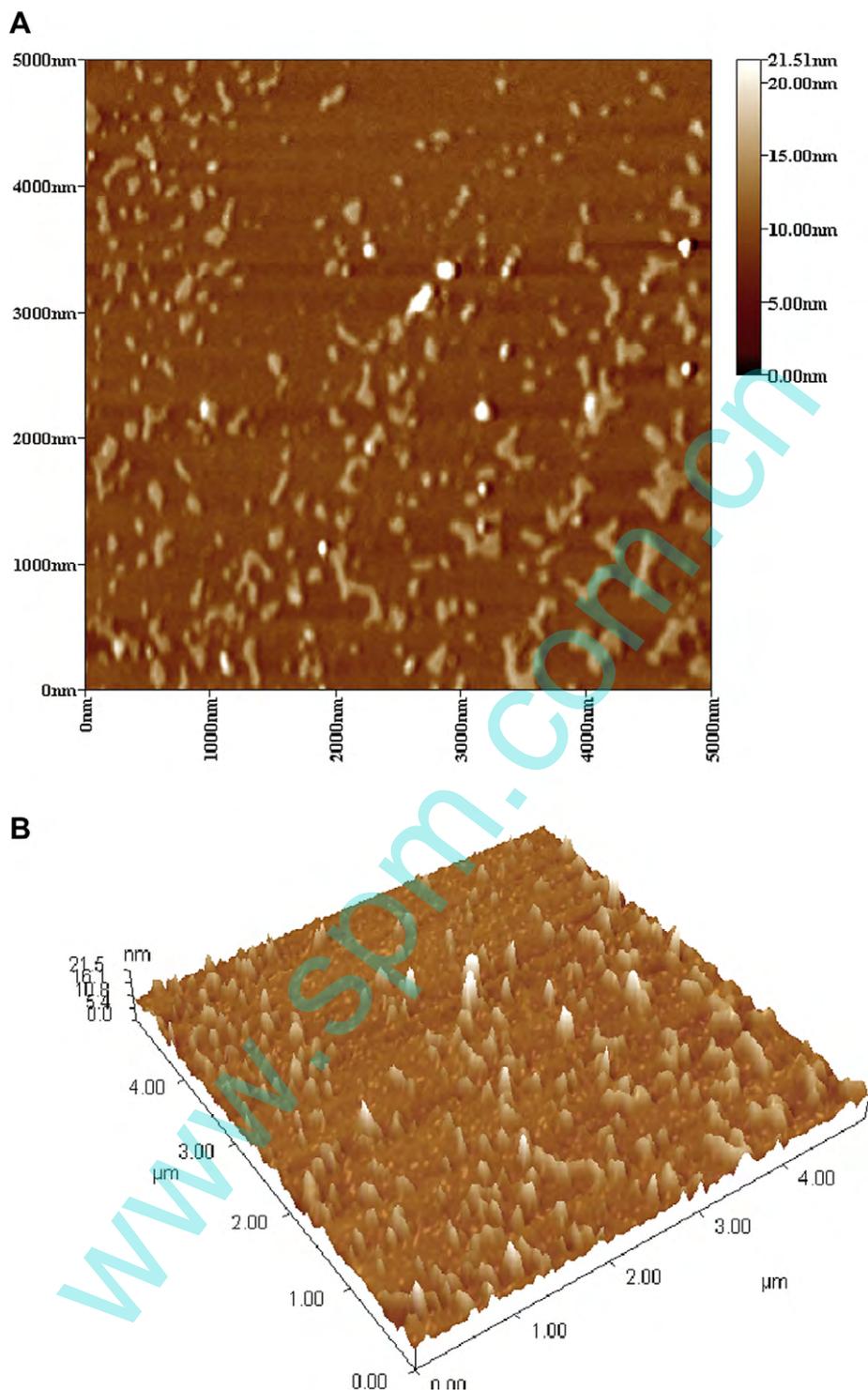
Fig. 5. AFM images: (A) topographic view of nanosize $CuAl_2O_4$ and (B) three-dimensional view of nanosize $CuAl_2O_4$.

agreement with the JCPDS values. However, the XRD pattern of copper aluminate prepared using NaOH or NaCO₃ as a precipitation agent did not match with the standard pattern.

Use of aqueous urea solution in place of KOH or NaOH can be advantageous because (a) urea does not have a tendency to get incorporated into the oxide matrix and (b) any residual urea entrained in copper aluminate powder can be easily driven off while drying the powder at 110 °C. Based on these results, the copper aluminate was synthesized using aqueous urea solution as a precipitation agent in this research.

3.7. Effect of copper source

To compare the composition of copper aluminate from different copper salts, we chose $Cu(NO_3)_2 \cdot 6H_2O$, $CuCl_2 \cdot 2H_2O$ or $CuSO_4$ as copper sources, respectively, without changing any other parameters. The XRD patterns of the $CuAl_2O_4$ particles prepared using different copper salt as raw materials are shown in Fig. 8.

Fig. 8 shows that the XRD pattern of nanosized copper aluminate prepared using $Cu(NO_3)_2 \cdot 6H_2O$ as the copper source is in better agreement with the standard data. But most of the peaks are

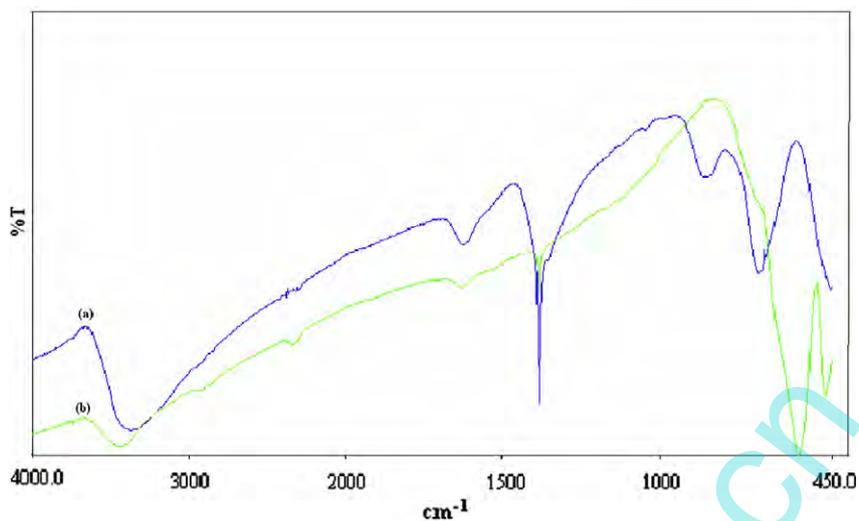**Fig. 6.** IR spectrum of: (a) as-formed precursor and (b) $CuAl_2O_4$ nanosized powders obtained by heating the precursor at 900 °C.
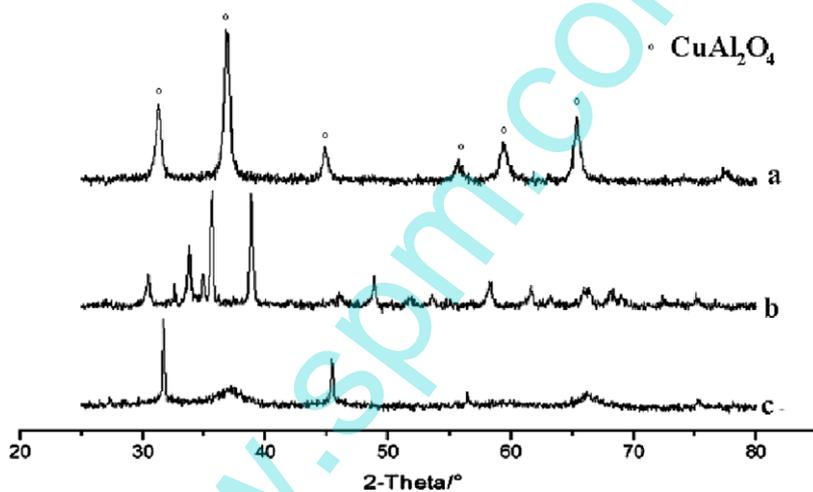

Fig. 7. XRD patterns of $CuAl_2O_4$ nanosized powders synthesized using: (a) urea; (b) NaOH; (c) $NaCO_3$ as the precipitation agents.
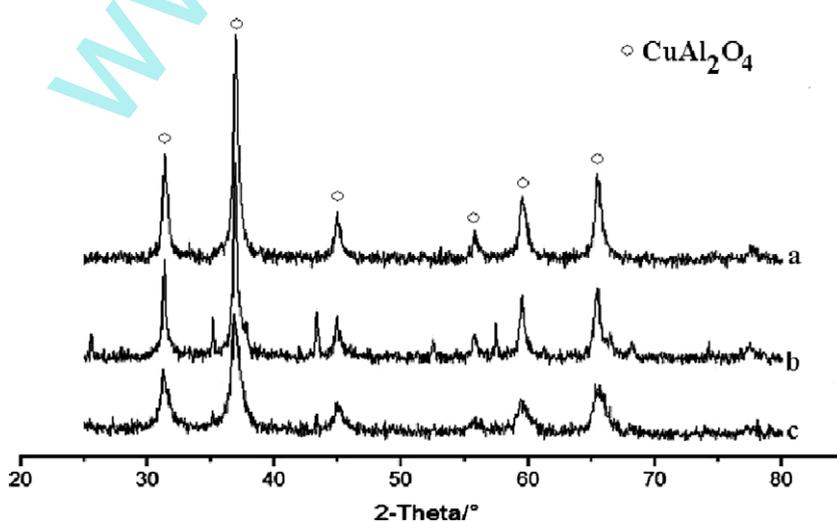

Fig. 8. XRD patterns of $CuAl_2O_4$ nanosized powders obtained from: (a) $Cu(NO_3)_2\cdot6H_2O$; (b) $CuSO_4$ and (c) $CuCl_2\cdot2H_2O$.

weak and broad when using $CuCl_2 \cdot 2H_2O$ as a copper source. There are many impure peaks utilizing $CuSO_4$ as a copper source. Hence, $Cu(NO_3)_2 \cdot 6H_2O$ was aptly chosen as a copper source in the present study.

3.8. Effect of sonochemical reaction time

The outcome of the sonochemical method is similar to that which would be achieved by chemical co-precipitation. The sonochemical method has some advantages, however. For example, there is a uniformity of mixing, reduction in crystal growth, morphological control, reduction in agglomeration, etc. The sonochemical reaction times are the key factors to control aggregation and uniformity of the product particle size. The present work studies the effect of the sonochemical reaction times on the product particle size and composition. We chose sonochemical reaction times of 2, 3 or 4 h, respectively, without changing any other parameters. The XRD patterns of copper aluminate powders were shown in Fig. 9. From Fig. 9, with the increase of the sonochemical reaction times from 2 to 4 h, crystallinity increased accordingly. The peaks of copper aluminate obtained at a calcination time of 2 h were broad and weak. Mono-phasic copper aluminate with a crystallite diameter of 17.3 nm along the (3 1 1) plane was formed when carried out for 4 h under ultrasound irradiation at 80 °C and a calcination temperature of 900 °C for 6 h, with urea as a precipitation agent at a concentration of 9 M.

The effects of ultrasound radiation on chemical reactions are due to the very high temperatures and pressures, which occur during cavity collapse by acoustic cavitation. There are two regions of sonochemical activity, as postulated by Suslick and co-workers [35,36], namely, the inside of the collapsing bubbles and the interface between the bubble and the liquid which extends to about 200 nm from the bubble surface. If the reaction took place inside the collapsing bubble, as is the case for transition metal carbonyls in organic solvents, the temperature inside the cavitation bubble could reach 2300–5100 K depending on the vapor pressure of the solvent. If water were utilized as the solvent, the maximum core temperature that could be attained would be close to 4000 K [37]. The product obtained would be close to a result of the high cooling rates ($>10^{10}$ K s^{-1}) obtained during the collapse. On the other hand, if the reaction took place at the interface with a tem-

perature of 1900 K, nanocrystalline products are expected to form. If the solute is ionic with a low vapor pressure, then during sonication the amount of the ionic species would be very low inside the bubble and little product would be expected to occur inside the bubbles. Since the solutes are ionic in the present study, we propose that the formation of LDH would occur at the interface between the bubble and the liquid.

3.9. Effect of calcination temperature

Calcination is a necessary step in the preparation of aluminate by chemical co-precipitation. The calcination temperature plays a key role in the crystalline type and particle size of the copper aluminate. The copper aluminates were synthesized at calcination temperatures of 500, 700 or 900 °C, respectively. All other parameters were kept the same as previously described. Fig. 10 shows different XRD patterns for the precursor calcined at different temperatures. With the increase in the calcination temperature, crystallinity increased and the particle sizes also increased correspondingly. There were no typical peaks when the calcination temperature was 500 °C. According to the standard data of copper aluminate, the precursor calcined at 700 °C already showed a copper aluminate phase. Little CuO and Al_2O_3 phases existed in powders calcined at 700 °C. The powders calcined at 900 °C showed the pure copper aluminate phase.

3.10. Effect of calcination time

Calcination time has an obvious effect on the product of composition and particle size. Fig. 11 shows different XRD patterns for the powders calcined at 900 °C for 2, 4 or 6 h, respectively, without changing any other parameters. With the increase of the calcination times, crystallinity increased and particle size increased accordingly. The result shows that the three XRD patterns are characteristic of copper aluminate. But the peaks of the product obtained at a calcination time of 2 h were comparatively broad and weak. The peaks of the products obtained at a calcination time of 4 h showed one little impure peak. There exist a lot of impurities when the calcination times are below 2 h, including CuO and Al_2O_3. Optimized copper aluminate formation occurs at a calcination time of 6 h.

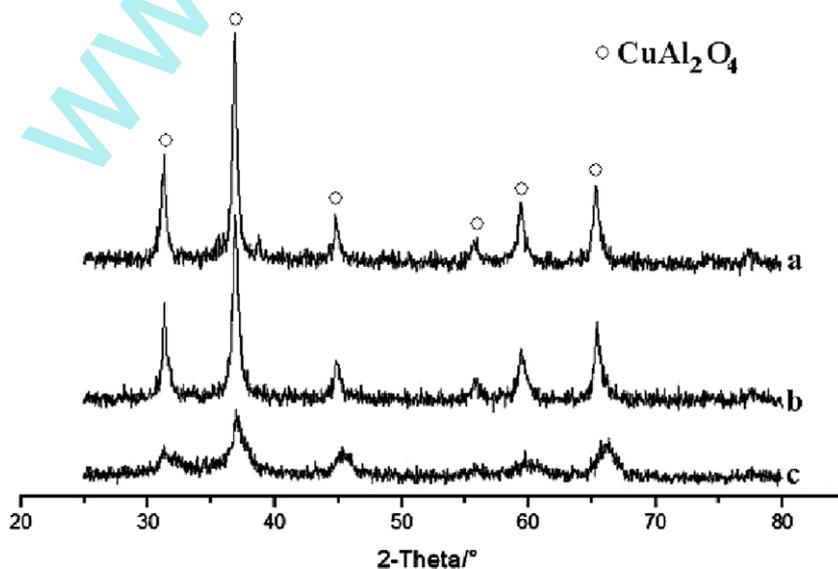**Fig. 9.** XRD patterns of $CuAl_2O_4$ nanosized powders obtained at different sonochemical reaction times (a) 2 h; (b) 3 h; (c) 4 h.
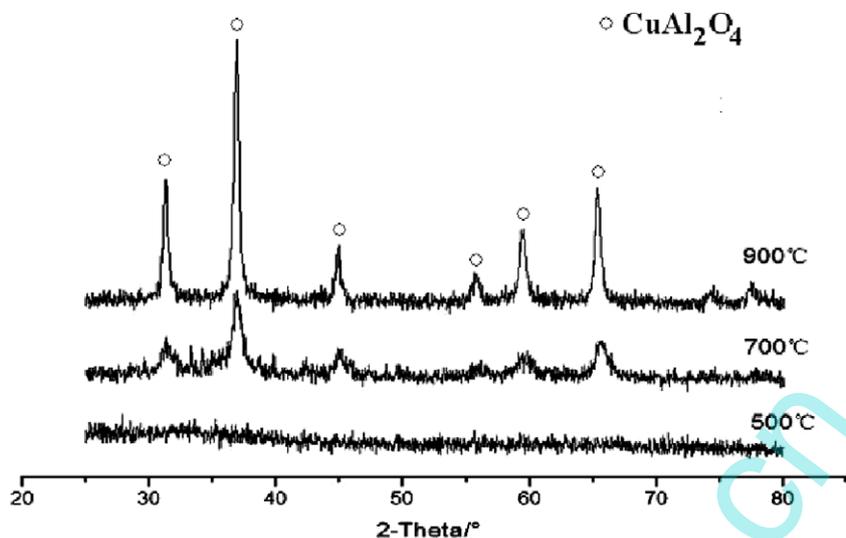
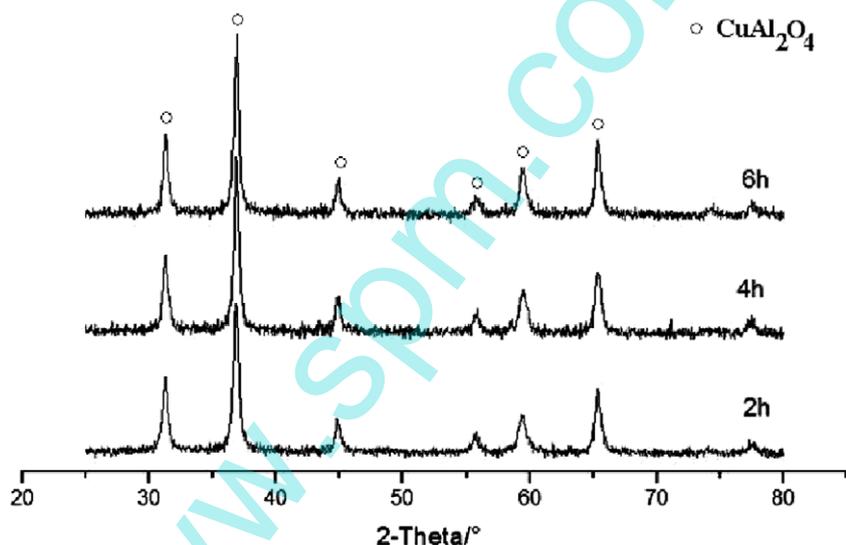**Fig. 10.** XRD patterns of CuAl$_2$O$_4$ nanosized powders obtained at different calcination temperature.

Fig. 11. XRD patterns of CuAl$_2$O$_4$ nanosized powders obtained at different calcination times.

4. Conclusion

In summary, nanosized copper aluminate spinel particles may be prepared by a precursor approach with the aid of ultrasound radiation. The effect of different processing conditions such as the copper source, precipitation agents, sonochemical reaction times, calcination temperature and time on the synthesis of nanosized copper aluminate particles was analyzed and characterized by a series of techniques: powder X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), atomic force microscopy (AFM) and Fourier transformation infrared spectroscopy (FT–IR). Mono-phasic copper aluminate with a crystallite diameter of 17 nm along the (3 1 1) plane was formed when the products were synthesized using Cu(NO$_3$)$_2$·6H$_2$O and Al(NO$_3$)$_3$·9H$_2$O as starting materials, with urea as a precipitation agent at a concentration of 9 M. The reaction was accomplished under ultrasound irradiation at 80 °C for 4 h and a calcination temperature of 900 °C for 6 h.

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