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RESEARCH PAPER

Sonochemical Preparation of Magnesium Hydroxide and Aluminum Hydroxide Nanoparticles for Flame Retardancy and Thermal Stability of Cellulose Acetate and Wood

Mahya Tamiji [1], Ali Reza Ahmadian-Fard-Fini [2], Manouchehr Behzadi [1], Davood Ghanbari[1,*]

[1] Department of Science, Arak University of Technology, Arak, Iran

[2] School of Built Environment, University of Technology Sydney, Sydney, Australia

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ABSTRACT

In this work firstly magnesium hydroxide ($Mg(OH)_2$) and aluminum hydroxide ($Al(OH)_3$) were prepared using sono-chemical reaction at solvent of water without applying any surface active agent. Effect of various sono-chemistry parameters such as power, cycles, time and volume on the size and shape of nanostructures were investigated. Secondly nanoparticles were modified and coated by ethyl cellulose capping agent. Modified nanoparticles were added to cellulose acetate and surface of wood for investigation of flame retardancy. Thermal stability were characterized by thermal gravimetric analysis (TGA). Flame retardancy were examined by UL-94 and heat release tests.

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INTRODUCTION

Various flame retardants were used in the previous decades while application of a lot of aromatic and effective flame retardants were limited because of release of toxic gases and aromatic residuals. Magnesium hydroxide ($Mg(OH)_2$) and aluminum hydroxide ($Al(OH)_3$) were applied as a green and biocompatible flame retardants, but bulk metal hydroxide are not very effective, for suitable effectiveness about 60 percent of these fillers are needed. High percent of bulk additives reduce mechanical stability. Nanoparticles because of high ratio of volume to surface can solve this problem, so instead using of high amount of bulk fillers we can use nano dimensions additives. In front of heating and flame both aluminum hydroxide ($Al(OH)_3$) and magnesium hydroxide ($Mg(OH)_2$) release water around 300 and 400 ºC respectively [1-7]. The interesting result is that both of them with endothermic reactions release water. Water vapor dilute and cold the environment of the flame zone and adsorb heat [8-12]. In this work these bio compatibles nano flame retardants were synthesized by applying ultra sound irradiation in a water medium. For modification of the nano cores ethyl cellulose was applied. Finally these nano additives were sprayed on the surface of the wood. The results confirm appropriate thermal stability and flame retardancy of both nano additives in compare to pristine wood. Thermal stability was approved around 10-20 ºC, flame retardancy of wood also improved.

* Corresponding Author Email: ghanbarichemist@gmail.com

MATERIALS AND METHODS

All the materials were obtained from Sigma-Aldrich or Merck Company. For preparation of magnesium hydroxide firstly 1g of $Mg(NO_3)_2$ $6H_2O$

was dissolved in 200ml of water, then under ultrasonic irradiation (100W, 60 min) ammonia solution (0.1M) was added to the solution until reaching pH to 11. After that white precipitate was centrifuged and was washed by distilled water and ethanol.

For synthesis of aluminum hydroxide, 1g of $Al(NO_3)_3$ $6H_2O$ was dissolved in water and under sono-irradiation (100W) sodium hydroxide solution (0.1M) was added to the solution (pH to 11). The prepared product was rinsed by distilled water and ethanol. For preparation of sample for UL-94 test, 1g was added to 5 g of cellulose acetate and after 5 h stirring and ultrasonic irradiation the solution was casted and the final product with 130*13*3 mm .

RESULTS AND DISCUSSION

Schematic of magnesium and aluminum hydroxide preparation is depicted in Fig. 1. Phase of product was investigated by X-ray diffraction pattern, XRD pattern in Fig. 2 show pure hexagonal phase of magnesium hydroxide with JCPDS 78-0316, space group : P-3m1). Fig. 3 illustrate XRD pattern of the aluminum hydroxide that approve formation of pure Monoclinic phase (Mineral name: Gibbsite) with JCPDS 33-0018, space group P21/n, space group number 14 (a: 8.6552 , b: 5.0722, c: 9.7161) . Scanning electron microscopy images of $Mg(OH)_2$ is illustrated in Fig. 4 that

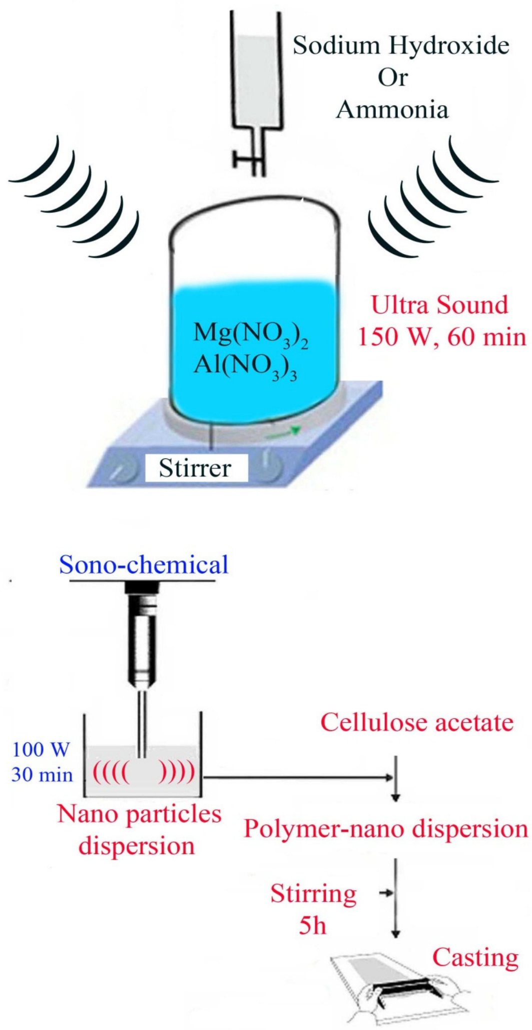

Fig. 1. Schematic of nanoparticle and nanocomposites preparation

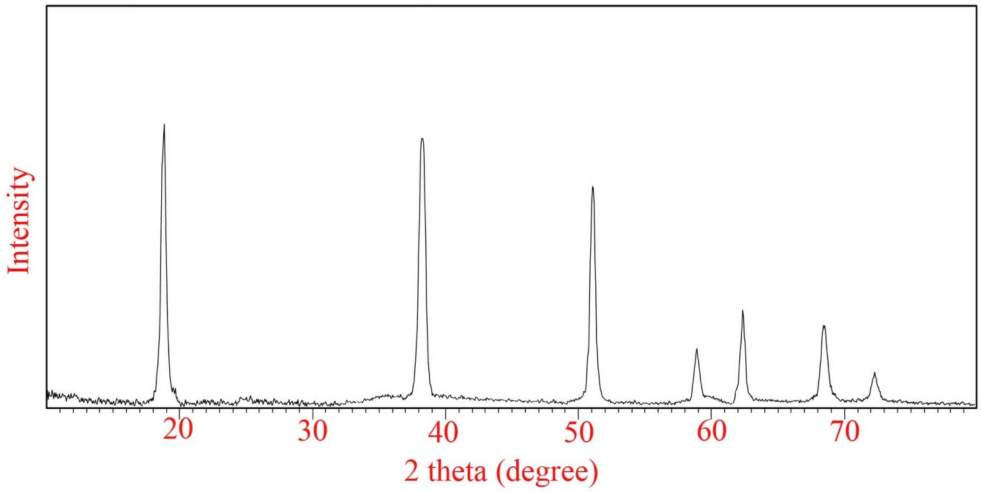

Fig. 2. XRD pattern of the $Mg(OH)_2$ nanoparticles

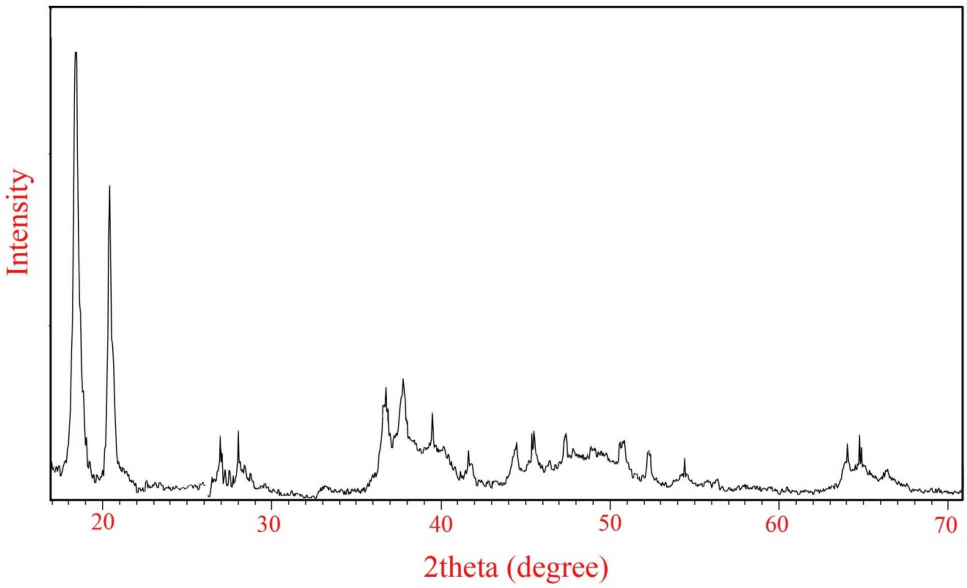

Fig. 3. XRD pattern of the $Al(OH)_3$ nanoparticles

approve formation of nanoparticles with average size less than 30 nm. By applying ultrasound irradiation nano bubbles are created and after final growth they will blow. By these explosions micro-jets are created and hot spots are produced, these hot spots are very effective for breaking of bonds and agglomerations. By using sono-chemical waves mono-disperse nanoparticles were prepared. SEM images of the $Al(OH)_3$ are depicted in Fig. 5 , that approve formation of nanoparticles with mediocre size around 30 nm.

Fig. 6 illustrates the FT-IR spectrum of cellulose acetate-$Mg(OH)_2$ nanocomposite and show absorptions at 550 cm^{-1} related to the Mg–O bonds in $Mg(OH)_2$. Peak around 2950 cm^{-1} is because of aliphatic C-H bonds. Absorptions around 1400 and 1600 cm^{-1} are responsible to C=O bonds and 1106 cm^{-1} is responsible to C-O bond.

Fig. 7 show thermal gravimetric analysis (TGA) of the nanocomposites, by addition of nanoparticles to the polymer or wood matrix, thermal stability and flame retardancy were

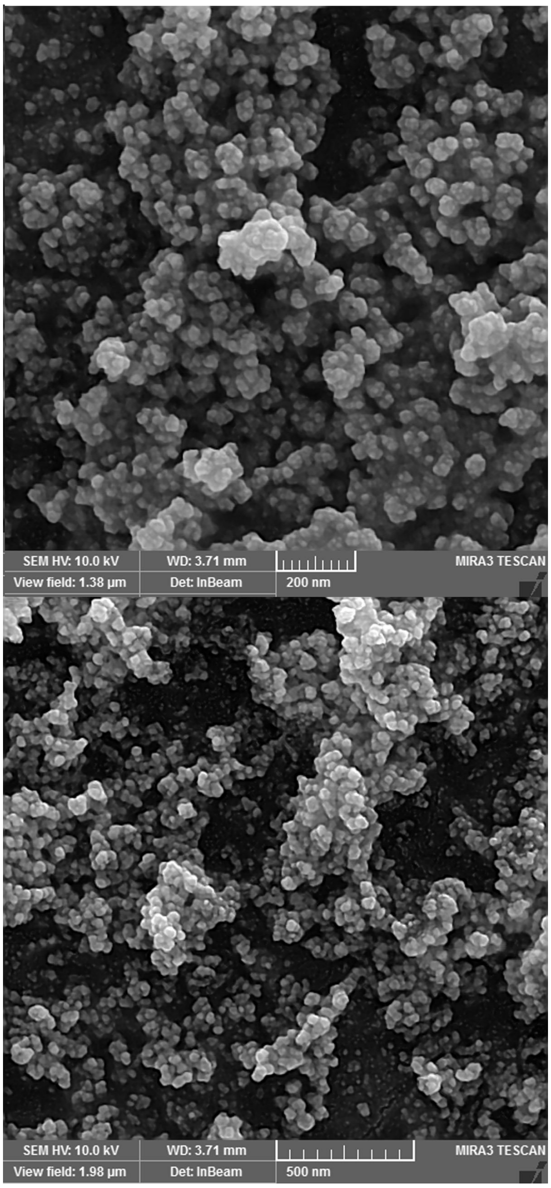

Fig. 4. SEM images of the $Mg(OH)_2$ nanoparticles

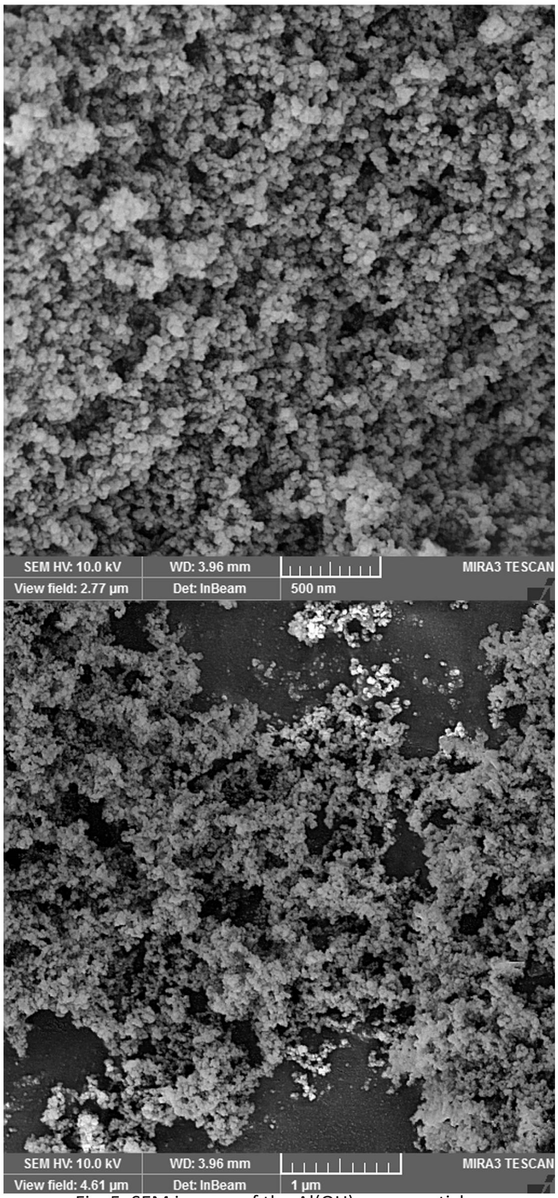

Fig. 5. SEM images of the $Al(OH)_3$ nanoparticles

approved. In temperatures around 300-400 ºC both magnesium and aluminum hydroxides with endothermic decomposition release water vapor.

Due to present of nanoparticles thermal decomposition of the nanocomposite was shifted towards the higher temperatures. nanostructures can act as barriers which slow down product volatilization and thermal transport during decomposition of the polymer.

The influence of $Mg(OH)_2$ and $Al(OH)_3$ on the fire retardancy of the cellulose acetate and has been considered using UL-94 test and is depicted in Fig. 8. In UL-94 a sample 130 × 13 × 1.6 mm is used. A flame (1.5 cm) is applied to the specimen (10 s each) twice. If sample quench in less than 10 second after any fire contact classified as V-0, particles drips are allowed as long as they are not inflamed. A V-1 type is given to a sample with maximum time less than 30 s (drips are like V-0). V-2 has time condition like V-1 while flaming drips are allowed. When the total flaming time is above 50 s. it is not classified (NC), finally horizontal burning with rate less than 76 mm/min is HB [5-10].

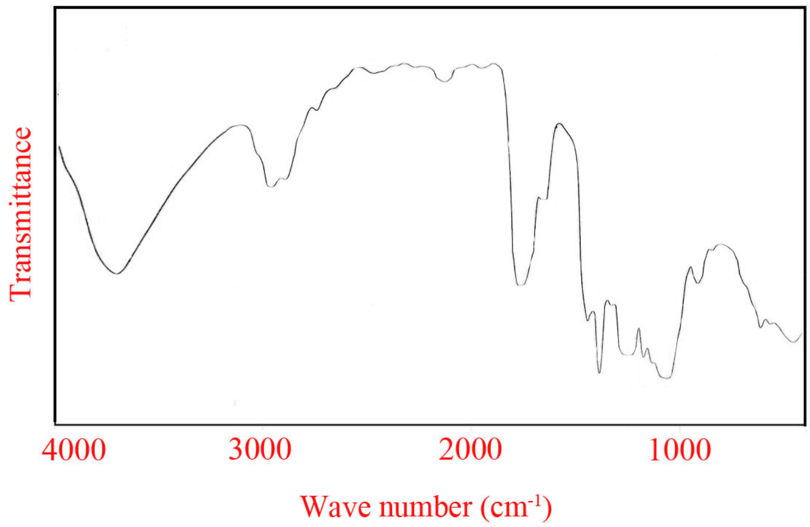

Fig. 6. FT-IR spectrum of the $Mg(OH)_2$ nanoparticles

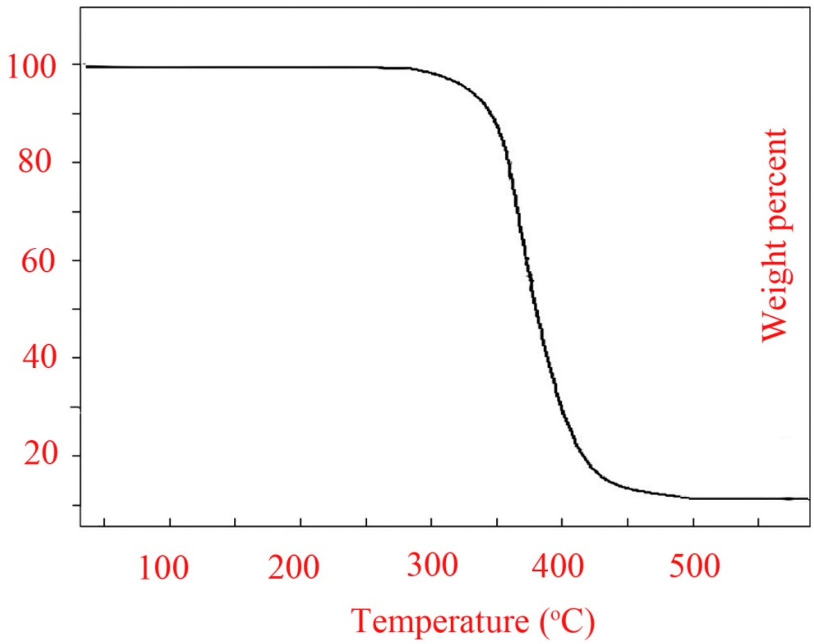

Fig. 7. TGA spectrum of the CA-$Mg(OH)_2$ nanocomposite

UL-94 tests for pure CA is N.C while CA-$Mg(OH)_2$ and CA-$Al(OH)_3$ show V-0 classification. Flame retardancy of nano-composite is because of higher surface to volume which can disperse into the matrix homogeneously, and formation of a dense char during the combustion. Hydroxyl groups on the surface of these nanoparticles have suitable interaction with cellulose acetate. These modified nanoparticles with ethyl cellulose were sprayed on the wood samples, during decomposition of magnesium and aluminum hydroxide H_2O is released, this water vapor dilute combustion environment of the flame zone, also with an endothermic reaction absorb heat from the flame zone. After decomposition of aluminum hydroxide, Al_2O_3 and char remain as a barrier

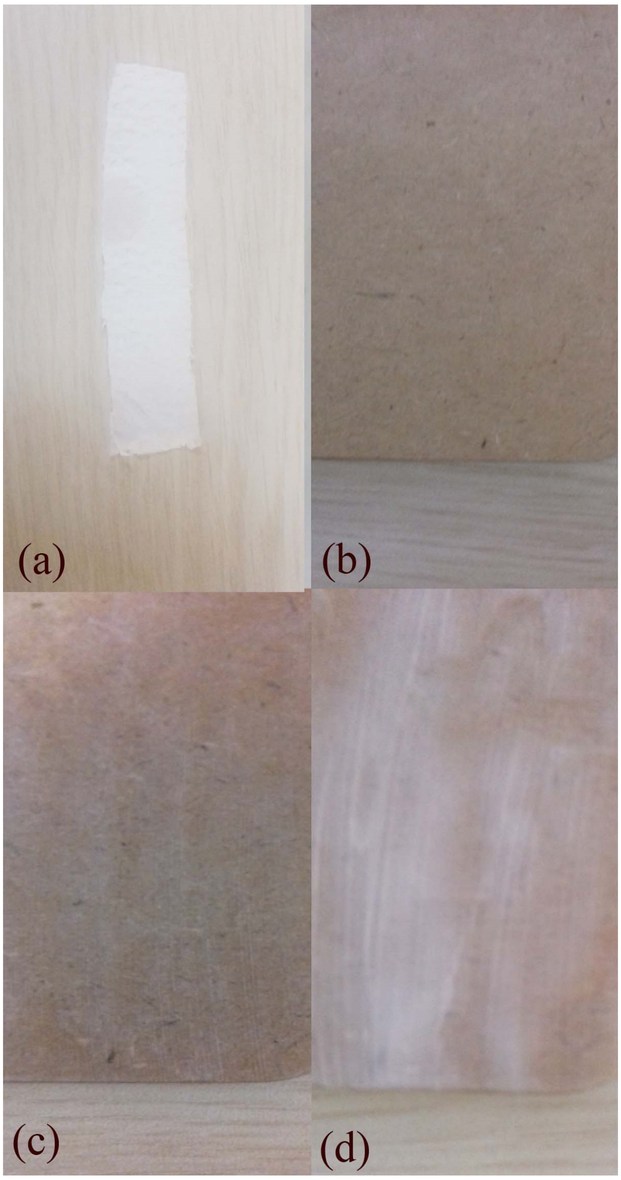

Fig. 8. UL 94 test of the (a) CA-$Mg(OH)_2$ nanocomposite (b) pristine wood (c) wood-$Mg(OH)_2$ nanocomposite (d) wood-$Al(OH)_3$ nanocomposite

layer. This obstruction slows down evaporation of polymeric segments and prevents reaching oxygen, heat and flame to the product [8-9].

CONCLUSION

Magnesium hydroxide and aluminum hydroxide were synthsized using ultra-sonic at water without applying surfactant. The influence of power, cycles, time and volume on the particle size of nanostructures were investigated. Nanoparticles@ ethyl cellulose were added to cellulose acetate and wood surface for improvement of flame retardancy. Thermal stability was improved about 20ºC and flame retardancy was got better from NC to V-0. In temperatures around 300-400 ºC both magnesium and aluminum hydroxides with endothermic decomposition release water vapor.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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