



Continuous flow formulation and functionalization of magnesium di-hydroxide nanorods as a clean nano-fire extinguisher



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ABSTRACT

The particle size of magnesium di-hydroxide (MDH) was reported to have a significant impact on its flammability performance. Consequently, there have been many classical trials to produce ultra fine MDH for polymer flame retardancy. This paper reports on a novel continuous flow hydrothermal method for the instant synthesis of MDH nanoparticles. The hydrothermal conditions including: temperature, pressure, and flow rate were precisely controlled to achieve consistent product quality. MDH nanorods of 120 nm length and 20 nm diameters were reported by TEM. The tailored MDH crystalline phase and its phase transition with temperature (during its endothermic heat sink action) were investigated with XRD. The effectiveness of MDH as an endothermic heat sink material was evaluated by TGA and DSC. MDH was surface modified with organic polymeric surfactant in a continuous manner via post synthesis surface modification approach. Organic modified MDH exhibited complete change in surface properties and the nanoparticles were harvested from water phase to organic phase. Organic modified MDH exhibited smaller particle size with an increase in specific surface area and surface porosity compared with uncoated MDH. The synergism between MDH and an intumescent flame retardant (FR) agent commercially known as Exolit AP750 was investigated in order to achieve self extinguish multi-component epoxy nanocomposite at 10 wt.% total solid loading. This is the first trial to synthesize and to functionalize ultra fine MDH continually; this approach might extend MDH usage as a clean and nontoxic FR agent that can be effectively surface modified. Therefore, enhanced flammability performance can be achieved at low solid loading level.

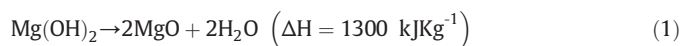
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1. Introduction

Inorganic hydroxides represent more than 50% of flame retardants (FRs) sold globally [1,2]. They act as heat sink materials by consuming energy during their thermal decomposition, releasing water, and forming an oxide layer [3,4]. Therefore they cool down the burning polymer surface, dilute the combustion gasses, and shield the polymer under-layers via the formed oxide layer [5]. The formed oxide layer also absorbs soot leading to low smoke levels [2]. In accordance with ISO 4589, limiting oxygen index (LOI) is still the most commonly used test to describe the flammability performance [5]; LOI values of at least 30% oxygen are necessary to fulfil the basic flame retardancy in wide applications [2]. Fig. 1 gives the relationship between LOI and different hydrated mineral loading levels in poly(ethylene-co-vinyl acetate) (EVA) copolymer [2].

The high processing temperature is a challenge for hydrated minerals particularly for aluminium tri-hydroxide (ATH) which starts decomposition at 180 °C [1]. This is why much attention has been directed to magnesium di-hydroxide (MDH), which provokes an

endothermic dissociation reaction at temperature higher than 300 °C and is very effective up to 400 °C [6]. MDH endothermic degradation accompanied with heat absorption and water release with the formation of MgO as a protective oxide layer [2] (Eq. (1)).



This is why it is very interesting with respect to the extrusion and injection moulding processes of some polymers [5]. Yet, high solid loading level is required to achieve the required LOI value. The particle size of MDH was reported to have a significant impact on its flammability performance; which can be ascribed to the rate of filler decomposition and/or the formation of more stable ash [7,8]. The replacement of 50 wt.% micrometric (2–5 μm) MDH in EVA with nanometric MDH showed improved fire performance; the LOI value increased from 24% to 38.3% [5]. Consequently, there have been many trials to produce MDH nanoparticles for flame retardant applications [9]. MDH with versatile morphological structures can be prepared by several methods, such as sol–gel technique [10], precipitation [11–17], hydrothermal [13], solvothermal [18], preparation using a bubbling setup [19], and microwave assisted synthesis [20].

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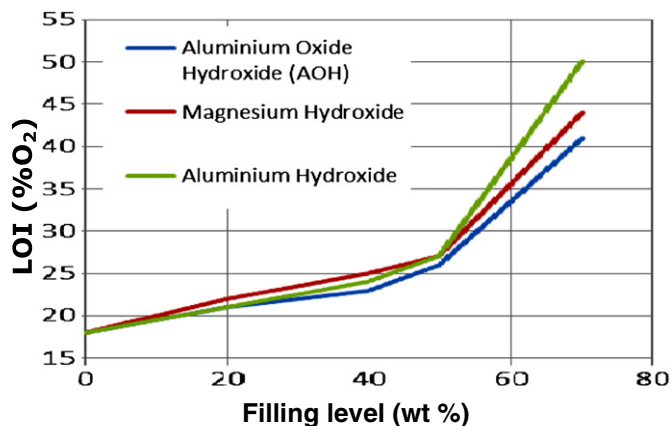


Fig. 1. LOI values of hydrated minerals with different loading levels in EVA.

Recently, hydrothermal synthesis is becoming one of the up-and-coming nanomaterial fabrication technology [21,22]. In the past century, hydrothermal synthesis was performed in batch mode, which has several drawbacks such as: time consuming, low yield, and poor reproducibility. These factors limited its application on the industrial scale [23]. Many attempts have been performed to establish continuous hydrothermal synthesis techniques [21,24,25]. In 2006, Lester and Blood established a new continuous nozzle reactor design, which involved counter-current mixing of an aqueous metal salt stream with a super-critical water (ScW) stream. This design exploited the density difference between the two feed streams; therefore instantaneous and strong mixing was achieved. The resultant turbulent micro mixing eddies were streamlined toward the reactor outlet with no stagnation zones [26]. Fig. 2 shows the counter-current reactor design and the fluid mixing simulation.

Nanoparticles of narrow size distribution are expected to form when instantaneous nucleation occurs and subsequent growth takes place at the same rate for all particles [27]. Through hydrothermal synthesis, the nanoparticle surface properties can be significantly altered from hydrophilic to hydrophobic and vice versa by the proper selection of

the capping agent (surface coating agent) [23]. Therefore continuous hydrothermal synthesis (CHS) offers a relatively simple route which is inherently scalable and chemically much more benign than many other nano-production technologies [26]. This approach could be an ideal scenario for nanoparticle synthesis near the critical and super-critical conditions. Furthermore, CHS offers highly controlled diffusion, size and shape control, minimized grain boundary effect, dense particles, high crystallinity, and phase purity [23,28]. CHS also facilitate on line surface modification of nanoparticles during their manufacture. Adschiri group reported that the use of surfactants to control the surface morphology; the size of the particles can be effectively controlled by appropriately maintaining the molar ratio of the modifier to the starting material and the type of surfactant (Fig. 3) [27].

So far MDH is manufactured in batch techniques, which are mainly precipitation processes (using batch stirred reactor vessel); these classical techniques are laborious, time consuming with no control of the particle morphology [17]. For instance, in 2013 Pilarska reported poly-dispersed MDH with particle size ranging from 0.6 to 600 nm; with no control of the particle morphology. Furthermore, the classical attempts for MDH surface modification with fatty acids and poly(ethylene glycol) [9] could not secure effective surface modifications due to the lack of anchoring groups, and functional group similarity respectively [29].

This paper reported on a novel continuous flow formulation and surface modification of MDH nanoparticles. Surface modification of MDH was achieved via post synthesis approach using poly(ethylene-co-acrylic acid) which can offer strong covalent bonding to MDH surface via many anchoring carboxylic groups along the polymeric chains. Organic modified MDH exhibited smaller particle size with complete change in surface properties from hydrophilic to hydrophobic, as well as an increase in specific surface area and surface porosity. Surface modification was reported to have the potential to enhance the dispersion characteristics of inorganic nanoparticles into the hosting polymers (dispersion to the molecular level with minimum aggregation) [28]; consequently enhanced flammability performance can be achieved at low solid loading level [1,5,30]. The proper combination between MDH and an intumescent FR agent was vital to achieve self extinguish multi-component epoxy nanocomposite that was able to resist a flame at 1700 °C and to self extinguish after the flame was removed.

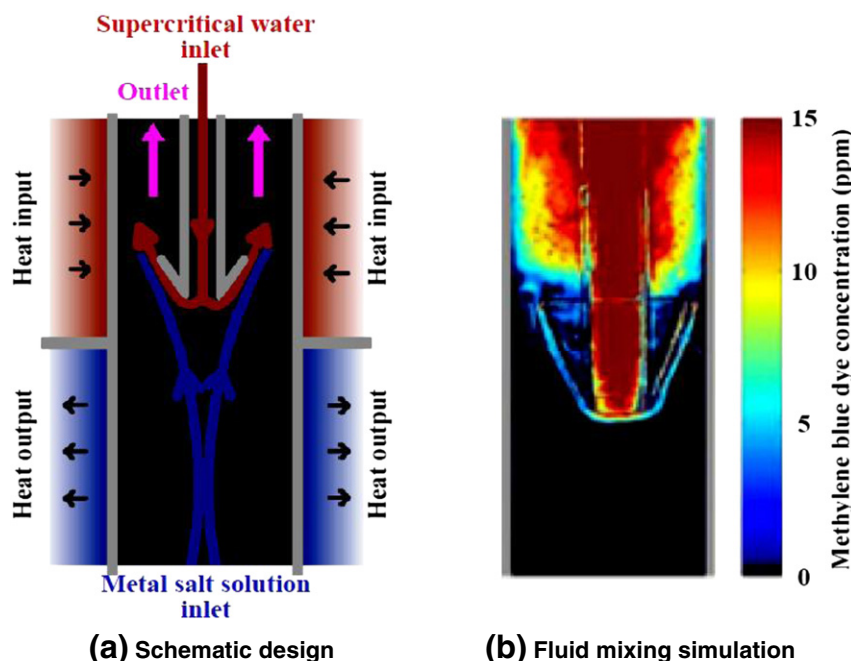


Fig. 2. Schematic for counter-current reactor design and fluid mixing.

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