***This work aims to prepare magnesium oxide MgO nanopowder using the coprecipitation method and prepare nanocomposites by mixing MgO prepared nanopowder with epoxy resin by weight percentages (0.5, 1, 1.5, 2, and 2.5) using hand lay up molding. These prepared chemical materials are added to many consumer products to meet fire safety codes and prevent these items from catching fire quickly. If the flame retarded material or an adjacent material has ignited, the flame retardant will slow down combustion and often prevent the fire from spreading to other items. Especially some of these chemicals can accumulate in parts of electrical equipment, cars, airplanes, and building components. Using non toxic nanofillers in polymers to achieve flame retardancy is a viable option. The prepared powder has a cubic structure, space group, and 4.2165 Å unit cell parameters according to X-ray diffraction XRD data and using Dicvol 91 indexing program. The grain size of the prepared powder was measured using Sherrer's equation to be 12.45 nm. The scanning electron microscope SEM micrograph of MgO nanopowder showed a spherical shape. The effect of MgO on flame retardancy of epoxy resin was investigated using limiting oxygen index LOI, rate of burning RB, and maximum flame height HF tests. According to the results of the three standard tests, the best flame retardancy with a strong and well intumescent char is obtained from the sample with 2 wt. % of MgO nanopowder, which has the highest LOI value of 21.95, RB value of 1.65 cm/min, and HF value of 5.44 cm. This data of using MgO nanopowder as flame retardant was valuable and necessary because it showed MgO nanopowder help prevent and slow fires of epoxy resin, therefore, protecting property and saving lives***

***Keywords: flame retardant, magnesium oxide, nanocomposites, epoxy resin, nanopowder, coprecipitation method***

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# IMPLEMENTATION OF THE MGO/EPOXY NANOCOMPOSITES AS FLAME RETARDANT

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## 1. Introduction

Modern fire precautions guarantee a high level of fire safety and virtually exclude catastrophic fires which razed entire towns only one century ago. However, even today, fire statistics show a high death toll and huge losses to the economy [1]. Where FRs have been introduced in the manufacturing of many goods to meet fire safety requirements. Since the 1980s, there has been a growth in polymeric material utilization, which has enhanced the risk of fires caused by the flammability of polymeric materials [2]. To address this weakness, several FR treatments and techniques have been introduced, such as nanofillers, halogenated and nonhalogenated FRs, grafting layered silicates, copolymerization, and the synergistic use of natural fiber and FRs [3]. There are a number of FRs available that serves a vital purpose in protection against textile related fires and are used throughout the world in various applications such as textiles, building supplies, plastic molding materials, mattresses, clothing, furniture, and a variety of other materials. FRs are being applied to the material to make them noninflammable or self-extinguishing Where the principle of FRs is to decrease its propensity to burn when subject to a heat source or open flame [4]. From the above can be defined flame retardants are chemical materials that can withstand direct flame where it works to increase ignition resistance and reduce the rate of flame spread and it is added to the material that can't resist flame to improve its properties [5–7]. These materials are either additives called external flame retardants, which are ineffective chemical additives added and mixed with polymeric materials without any chemical reaction with them or as part of the polymer structure called internal flame retardants [8]. Two methods are known to inhibit the flame, the first method is preventing the oxygen from reaching to flame zone by generating noncombustible gases which cause poisoning of the flame by free radicals and extinction on it, the second method depends on the thermal flame theory which states that the flame retardants need to thermal energy to disintegrate which leads to the reduction of the heat surface material to temperature less than the temperature of ignition and thus the burning will stop [9–11]. Four primary materials work to retard flame in various ways. These materials include nitrogen, phosphorus, halogenated, and inorganic flame retardants [12, 13].

Therefore, studying the dependence of change in oxygen index and rate of burning on the amount of flame retardant fillers and the chemical composition is a relevant task.

## 2. Literature review and problem statement

An analysis of the scientific literature demonstrates that one of the ways to reduce the flammability of polymeric materials based on magnesium oxide MgO nanopowder is the introduction of flame retardant fillers to the polymeric composition. the effect of nanomagnesium oxide on the mechanical and flammability features (flexural strength and modulus, flammability strength including the amount of char

residue, time to ignition, total smoke production, and heat release rate) of composites made of wood flour and high density polyethylene was studied. The addition of nanomagnesium oxide increased the ignition time and char residue, and it decreased the total smoke production, heat release rate, and burning rate, SEM and EDX pictures taken from samples including nanomagnesium oxide showed improper distribution and accumulation of nanomagnesium oxide, which weakened the mechanical features of nanocomposites [14]. MgO and microencapsulated red phosphorus (MPR) were incorporated into high impact polystyrene matrix (HIPS) by melt compounding and were prepared MgO-MRP/HIPS composites. The composite (MgO: MRP: HIPS) with this composition (35:15:100) exhibits excellent flame retardancy because both MgO and MRP can promote charring which acts as a barrier against fire and gives rise to increased flame retardancy when the polymer is degraded or burned in the air [15]. A precipitation aging method with and without calcination used to synthesize the flower-shaped hydromagnesite (MgO-P) and magnesium oxide (MgO) micro spheres with the thickness (~ 20 nm) was used as flame-retardants for the electrospun cellulose acetate (CA)/MgO-P or CA/MgO nanofiber. Three evaluation methods including thermography during heating, broken time after heating, and combustion observation were adopted to compare the flame retardancy of composited nanofibers. Doped CA nanofibers showed best flame retardancy, no smoke and size shrinkage could be observed during the combustion of CA/MgO-P or CA/MgO nanofibers [16]. Using an inorganic-organic composite (MCN or MgO/g-$C_3N_4$) synthesized by incorporating magnesium oxide (MgO) combined with graphitic carbon nitride (g-$C_3N_4$) to prepare flame-retardant polyamide 6 (PA6). The results of differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) revealed that the introduction of MCN efficiently enhanced thermal stability of PA6 because of the MCN possessed a laminate structure, more holes, and a larger specific surface area. It could effectively improve the flame retardancy of PA6 [17]. Colloidal magnesium hydroxide nanoflake $Mg(OH)_2$ with high dispersion stability was prepared by one step surfactant assisted preparation and paper based relics protection with long term anti acidification and flame retardancy. The surfactant acts as both a structure-direct agent to confine the growth of $Mg(OH)_2$ with rich active sites and a surface modifier to enhance its solvent adaptability and dispersion stability. The as-obtained $Mg(OH)_2$ presents the superior paper protection performance characterized by its safer pH than the original aged paper and the excellent long-term anti-acidification effect. Furthermore, it endue them as an improved flame retardant for multifunctional paper protection [18]. The effects of talc ($SiO_2$, MgO and CaO)/intumescent flame retardant additions (ammonium polyphosphate and pentaerythritol) on thermal, burning, and mechanical characteristics of the rigid polyurethane foams were investigated. The best fire resistance was achieved with a composition that includes 10 wt. % the intumescent flame retardant and 5 wt. % talc. The addition of this composition resulted in a 32 % reduction in the total heat release value [19]. A kind of novel aerogel composites were prepared by incorporation of $Mg(OH)_2$ coated hollow glass microspheres (HGM) into chitosan (CSA) matrix and then cross-linking by glutaraldehyde (abbreviated as CSA-HGM-$Mg(OH)_2$). The as-prepared composite aerogel exhibits high porosity, excellent thermal insulation, and flame retardancy with a high limit oxygen index (LOI) value up to 50.8 [20].

## 3. The aim and objectives of the study

The aim of the study is to identify the mechanism of coprecipitation method of (NaOH and $Mg(NO_3)_2.6H_2O$) in preparing the MgO nanopowder and identify the influence of the concentration of MgO prepared nanopowder used as flame retardant fillers on flame retardancy of epoxy resin that does not support combustion based on the values of LOI, RB, and HF.

To achieve the set aim, the following objectives have to be solved:

– establish the phase formation, unit cell parameters, space group, and surface morphology of prepared MgO nanopowder;

– study a dependence of the oxygen index of epoxy polymeric compositions on their formulations composition and properties of the ingredients.

## 4. Materials and methods of research

We studied flame retardancy of epoxy resin, as well as flame retardant fillers, which were magnesium oxide nanopowder. The synthesis process of the MgO nanopowder and the fabrication process of the MgO/epoxy resin nanocomposites are shown in the following methods:

The magnesium oxide MgO nanopowder was prepared by the coprecipitation method. The (0.1 M) concentration of $Mg(NO_3)_2{\cdot}6H_2O$ dissolved in 200 mL of distilled water and (0.1 M) concentration of NaOH also dissolved in 200 mL of distilled water, the NaOH solution adds to $Mg(NO_3)_2{\cdot}6H_2O$ solution by dropwise with magnetic stirring for 6 hours. The mixture was filtered and washed several times using distilled water to get the final product, and then dried to remove the moisture at 100 °C for 8 hours. The dried powder was calcinated in a furnace at 550 °C for 5 hours to remove the impurities present in the powder.

The MgO/epoxy nanocomposites were fabricated as follows: The prepared MgO nanopowder (used as flame retardant material) was added with percentage weight (0.5, 1, 1.5, 2, and 2.5) by hand lay-up molding to Epoxy resin and hardener type (amine) (Sikadur-52) of (2:1) for ratio curing. Molded samples as a sheet shape in the dimensions of ($13\times13\times0.4$ $cm^3$). The samples are cut by laser and have smooth edges according to the test method. For limiting oxygen index (L.O.I), rate of burning (R.B), and maximum height of flame (HF) testing, the specimens were chosen and each one has a length of ((130±5), (130±5), and (125±5)) mm, the width of ((6.5±0.5), (12.5±0.5) and (10±0.1)) mm, respectively and thickness of (3.0±0.1) mm.

To characterize magnesium oxide MgO nanopowder and prove the particle size in a nanoscale; XRD and SEM tests were performed. To determine the flammability of MgO/Epoxy nanocomposites; limiting oxygen index (L.O.I) (ASTM:D-2863), the rate of burning (R.B), the average extent of burning (A.E.B), and the average time of burning (A.T.B) are measured according to (ASTM:D-635), and the maximum height of flame (HF) and the amount of loss in weight of polymer are measured according to (ASTM:D-3014) were performed.

L.O.I is the most widely used test to determine the resistance to ignition and the relative flammability of polymeric materials. It was measured by (ASTM:D-2863) method and calculated from the following equation [21]:

$$n\%=(V_{O2}/(V_{O2}+V_{N2}))\times 100, \quad (1)$$

where $n\%$ – limited oxygen index;

$V_{O2}$ – volumetric speed flow of oxygen (mL/sec);

$V_{N2}$ – volumetric speed flow of nitrogen (mL/sec).

A. T. B in minutes and A. E. B were measured by (ASTM:D-635) method and calculated from the equations (2) and (3), respectively [22]:

$$ATB=\sum(t-30)/(\text{No. of samples}), \quad (2)$$

$$ABE=\sum(100-X)/(\text{No. of samples}), \quad (3)$$

where $t$ – time of burning (min);

$X$ – length of the unburned part of the model when self-extinguishing occurs (cm).

Other variables were calculated from (ASTM: D-635) method in addition to A. E. B. are:

– R. B. – rate of burning (cm/min);

– S. E. – self-extinguishing;

– N. B. – the ignition non-continuity of the specimen after the removal of the heat source.

The weight percentage for the residual of the burning material was measured by (ASTM: D-3014) method and calculated by the following relationship [23]

$$PWR\%=((W_1-W_2)/W_1)\times 100, \quad (4)$$

where $W_1$ – weight of the sample before combustion (gm);

$W_2$ – weight of residual material after combustion (gm);

$PWR\%$ – percentage weight of the residual burning material;

$H_F$ – maximum height of flame reached (cm).

## 5. Results of the Studying Implementation of the MgO/Epoxy Nanocomposites as Flame Retardant

### 5. 1. Synthetic Tests for the Prepared Powder

#### 5. 1. 1. X-ray Diffraction Test

The crystallography and phase formation of prepared MgO nanopowder were investigated using X-ray diffraction (Shimadzu XRD-6000) with Cu Kα1 radiation (λ=1.540 Å) and 2θ values from 30° to 110°. The peaks in the XRD pattern of MgO nanopowder (Fig. 1) were in accordance with JCPDS No. 75–1525 which expressed the cubic structure [16]. The results of indexing for MgO nanopowder performed by using (Dicvol 91) program are (unit cell parameters (a)=4.2165 Å with $Fm\bar{3}m$ space group and angels (α)=90º).

The existence of sharp peaks in the XRD pattern of MgO nanopowder (Fig. 1) established the formation of nanoparticles, and an increase in the peak width represented a decrease in the size of nanoparticles. Also, the absence of extra peaks in the synthesized nanopowder established their high purity. The average grain size for synthesized MgO nanopowder was determined by Scherrer's equation, which was found to be about (12.45 nm).

#### 5. 1. 2. Scanning Electron Microscopy Test

The surface morphology of MgO nanopowder was studied by scanning electron microscopy (Fig. 2).

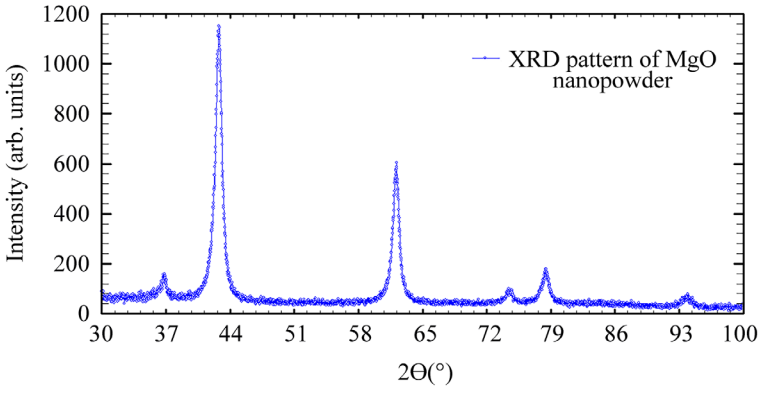


Fig. 1. X-ray diffraction pattern of MgO nanopowder

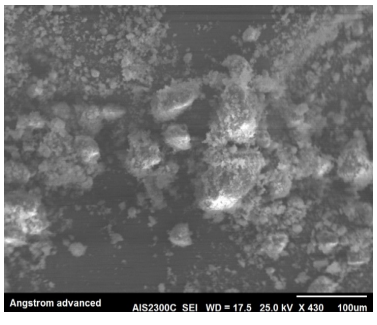


Fig. 2. Scanning electron microscopy image of MgO nanopowder

SEM micrograph (Fig. 2) showed agglomeration among MgO nanoparticles fine particles with the spherical shape of the nanoparticles.

### 5. 2. Flame Retardancy Tests of Prepared Nanocomposites

#### 5. 2. 1. Limiting Oxygen Index (L.O.I) test

The results obtained are represented in Table 1 show the L.O.I increased with the increase in the reinforcement ratios of the added MgO nanopowder to the epoxy (directly proportional) because MgO nanopowder catalyzed the thermal-oxidative degradation and accelerated a thermal protection/mass loss barrier at the burning surface; on the other hand, the filler decreased activation energies in the initial step and improved thermal stability in the final period [24].

Table 1

Results of LOI for the epoxy without and with reinforcement ratio of MgO nanopowder

| Type of additive | LOI vs. Additives% | | | | | |
|---|---|---|---|---|---|---|
| | Non | 0.5 | 1 | 1.5 | 2 | 2.5 |
| Neat Epoxy | 19.59 | – | – | – | – | – |
| Epoxy+MgO | – | 20.23 | 21.1 | 21.56 | 21.95 | 21.3 |

The value of the LOI in (Table 1) decreases with the increase of the reinforcement ratio at (2.5 % MgO) because the use of a large reinforcement ratio leads to the heterogeneous mixing and the occurrence of agglomeration of nanoparticles, thus the microvoids are converted to macro voids which can affect as oxygen concentration zones, which helps increase ignition.

**5. 2. 2. Rate of burning (R. B.) test**

The results of the rate of burning of epoxy with additive show a large reduction in (R. B). The (R. B) of epoxy samples was measured without and with additive in addition to the values of (ATB) and (AEB) were calculated by using (2), (3), respectively, as listed in Table 2.

Table 2

(A.T.B), (A.E.B), (R.B), (S.E) and (N.B) for epoxy without and with reinforcement ratios of MgO nanopowder

| Test | Additives % | | | | | |
|---|---|---|---|---|---|---|
| | Non | 0.5 | 1 | 1.5 | 2 | 2.5 |
| A. T. B. | 5.35 | 5.81 | 6.08 | 6.9 | 4.5 | 5.61 |
| A. E. B. | 10.3 | 10.3 | 10.3 | 10.3 | 4.12 | 6.88 |
| R. B. | 1.92 | 2.02 | 1.91 | 1.83 | 1.65 | 1.74 |
| S. E. | – | – | – | – | – | – |
| N. B. | – | – | – | – | – | – |

The decrease in the R. B. is inversely proportional to the increase in the reinforcement ratios of the added additive to the epoxy because the additive forms an inert atmosphere which reduces and inhibits the oxygen from reaching a flame zone that is required for maintaining the ignition.

**5. 2. 3. Maximum height of flame (HF) test**

Table 3 shows the results of the measurement of flame height for Epoxy with various amount weight percentages of MgO nanopowder.

Table 3

(HF) with reinforcement ratios of MgO

| Test | H vs. Additives % | | | | | |
|---|---|---|---|---|---|---|
| | Non | 5.0 | 1 | 5.1 | 2 | 5.2 |
| $W_1$ (gm) | 4.56 | 4.76 | 4.85 | 5.23 | 5.31 | 5.16 |
| $W_2$ (gm) | 1.37 | 0.8 | 0.86 | 1.28 | 1.4 | 1.07 |
| *PWR* % | 69.95 | 83.19 | 82.26 | 75.52 | 73.63 | 79.26 |
| *HF* (cm) | 12.3 | 11.3 | 8.8 | 7.43 | 5.44 | 6.52 |

The results showed that the increasing reinforcement ratios of the added additive lead to the decrease of ($H_F$) (inversely proportional) and an increase in effectiveness and retardancy (directly proportional) for the same reason mentioned in the LOI test.

## 6. Discussion of results of the studying implementation of the MgO/epoxy nanocomposites as flame retardant

Based on the data and the observations made throughout the report, it is clear that the flame retardants tested had significantly different effects on the flammability, heat release rate, and mass loss rate of epoxy resin during exposure to the heat radiation source.

Generally, The coprecipitation method of NaOH and $Mg(NO_3)_2.6H_2O$ was successful in preparing magnesium oxide nanopowder with a cubic structure, $Fm\bar{3}m$ space group, and 4.2165 Å unit cell parameters according to XRD data (Fig. 1) and a spherical shape according to SEM micrograph (Fig. 2), as well as, the grain size of the prepared powder was measured using Sherrer's equation to be 12.45 nm according to XRD data (Fig. 1).

Fire resistance of the polymeric epoxy resin, filled with flame retardants by weight percentages (0.5, 1, 1.5, 2, and 2.5), was determined using a method of oxygen index. The results of the research, shown in Table 1, demonstrate that the oxygen index increases with an increase in the content of flame retardant fillers. It should be noted that the oxygen index of the nanocomposite with 2 wt. % (21.95) grows faster than that of the other weight percentages. In other words, the content of MgO affects the values of the oxygen index. That is, degradation products of the polymeric matrix also affect the inhibiting process of combustion of the polymeric composition. The MgO/epoxy treated samples did show little or degradation in improvements in fire safety properties, such as increasing the time to ignition or reduction in heat release rate. In some cases at certain heat fluxes, the MgO/epoxy performed close than the untreated samples, e.g. the MgO (0.5 wt. %) decreased the ignition time, increased the average heat release rate, and the effective heat of combustion of epoxy resin at high heat fluxes. It is important to remember that the effectiveness and usability of MgO as a flame retardant should not solely be based on a heat radiation exposure test. In order to get a grasp of the electiveness of these flame retardants, experiments such as a flame spread test or as the first item to ignite should be considered, because of the numerous ways for a fire to occur and spread.

The limitation of this study is employing the flame retardant fillers with different compositions exerts a certain effect on the physical properties. It was established that an increase in the content of flame retardant fillers in polymeric compositions results in weak of the samples.

## 7. Conclusions

1. The coprecipitation method of NaOH and $Mg(NO_3)_2{\cdot}6H_2O$ was successful in preparing magnesium oxide nanopowder. The XRD peaks of the prepared powder revealed good crystalline of the nanoparticles with a mean particle size of 12.45 nm. The SEM micrograph of the prepared powder showed the prepared MgO nanopowder has a spherical shape with the agglomeration of fine particles.

2. Flame retardant fillers inhibit the combustion process more effectively, under conditions of filling with various flame retardants, where increase the oxygen index of polymeric compositions (19.59–21.95 %) at the concentration of fillers from 0 wt. % by weight to 2 wt. % by weight depending on their composition and dispersion. The increase in the reinforcement ratios from 0 wt. % to 2.5 wt. % leads to decrease in the R. B. from (1.92 cm/min) to (1.74 cm/min); the best result (1.65 cm/min) was at 2 wt. %. The increased reinforcement ratios of the added additive lead to the decrease of ($H_F$) and the best result of HF was 5.44 cm at 2 wt. %.

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