Flame retardancy properties and thermomechanical behavior of the nanocomposite of thermoplastic Polypropylene/ Linear Low-density Polyethylene blend filled with nano calcium carbonate

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Abstract. The flammability of thermoplastic polymers, especially of polypropylene, considers one of the crucial global concerns because of the numerous applications of these materials in different aspects of modern life. So, the efforts of scientists are focused on developing the effective flame retardants especially with the progress in the nanotechnology. In this work, Fire retardancy performance, mechanical properties, thermomechanical behavior and thermal stability of the blend of polypropylene (PP) - linear Low density polyethylene (LLDPE) filled with nano calcium carbonate (NCC) were examined. The blend was contained on 90% PP and 10% LLDPE with different loadings of nano calcium carbonate (1.5%, 3%, 6%, 9% of PP weight). The samples were prepared by the extrusion method. The mechanical tests were tensile and bending tests while fire retardancy tests were limited oxygen index (L.O.I), U-94 protocol tests and ash content. The thermomechanical tests were thermal expansion coefficient, and thermal E-modulus. Thermal stability was examined using differential scanning calorimetry (DSC) and Differential Thermal Analysis (DTA). The results of tests showed the positive role of using nano calcium carbonate as filler for this blend. The mechanical properties, thermal expansion coefficient and thermomechanical behaviour were increasing and the highest values were recorded for 6% sample. While the maximum values of thermal stability, and fire resistance properties were recorded for 9% sample. The other nano calcium carbonate addition ratios showed varies responses. The fire retardancy properties were enhanced with using NCC filler. The results of the present work showed the success of using nano calcium carbonate with PP-LLPE blend as a fire retardant.

Keywords: nano CaCO₃, polypropylene, fire retardancy, LLPE, blend, thermomechanical

1. Introduction:
Polypropylene (PP) is the outstanding polymer which it has numerous applications in different fields. PP is a non-polar thermoplastic polyolefin polymer which is manufactured from propylene monomer by a Ziegler-Natta catalyst or using a metalloocene as a catalyst which is increasingly used in propylene polymerization [1]. PP characterizes with ease formulation using processing methods, good resistance for chemical solutions, heat insulating, high stiffness and recyclability. In return, PP has defects like high thermal expansion coefficient; low resistance for UV-rays, weak bonding as paint, very sensitive for oxidation and the most important defect is its high flammability. PP flammability is classified as sever flammable polymer [2]. These defects contribute to limit PP applications. One of the effective
solutions to improve the properties of PP is blended with other polymers to create copolymers with suitable characteristics in order to achieve advantageous properties inherent to the combination of both resins. Many studies were fulfilled in recent years where PP was blended with HDPE [3,4], LDPE [5,6], PET [7,8], PS [9], EVA[10], PVA [11].

Linear low-density polyethylene (LLDPE) is produced by copolymerization between ethylene and an alfa-olefin co-monomer such as 1-butene, 1-hexene or 1-octene. It results in an ethylene/alfa-olefin copolymer with many short chain branches along the polymer backbone. Ultimate developments in metallocene catalysts allowed adding LCB on LLDPE structure during copolymerization [12]. It is characterized by high impact strength, good properties barriers against water and alcohols, good chemical resistance against mild and strong buffers. Besides, the linear low-density polyethylene (LLDPE) is used in many industries because of its unique melt flow properties and less shear sensitivity [13]. In return, main defects of LLDPE are a weak gas barrier, difficult to formulate in the processing process not like LDPE. But, the most important defect is high flammability.

Besides, PP composites in which are synthesized by using solid fillers is considered the best solution to improve the PP properties like mechanical, thermal stability, optical, hygienic and fire retardancy properties. PP mixed with different types of nanofillers recently like nano clay [14], Titanium dioxide [15], Zinc oxide [16], and calcium carbonates [17].The main reason to add a small portion of LLDPE is to achieve the acceptable improving in mechanical, optical properties and other physical properties in addition to reduce the cost of the production process [18, 19, 20]

The technique of synthesis copolymers filled with nanofillers is a very promising solution to get on the composites with outstanding characteristics. The other solution to improve the PP properties is the three components systems which give the scientists the opportunity to treat the defects of polymers and to design engineering composites for different engineering applications. Examples of three components systems works are like Nano silica with blending of PP/elastomer [21], PP/EVA/ n- SiO₂, [22], PP/EVA/ Fullerene[23], PP/PET/MWCNT [24],PP/LLPE/n-TiO₂[25]. The attempts are still in the beginning but the results are very encouraging ones.

The aim of this work is to assess the fire retardancy and to investigate the mechanical and thermomechanical of a polymeric blend of linear low-density polyethylene LLDPE/PP (10%-90%) filled with different concentrations of nano calcium carbonate.

2. Experimental works:

2.1 materials and chemicals: all materials and chemicals which were used in this work are listed in below:

a) Polypropylene: PP was provided by Sumitomo chemical company(Tokyo, Japan.), its melt flow 10g/min, Density is 0.9 g / cm³.

b) Linear low-density polyethylene (LLDPE) :it was provided by Sabic company (Jeddah, KSA). It is characterized by density 0.91 g/cm³ melting flow 2g/10 min

c) Nano calcium carbonate NCC: the physical properties of nano CaCO₃ are listed in table 1

d) Coupling agent: it is used to increase the compatibility between nanoparticles and polymer matrix PP. Its properties are listed in table 1

e) Ethanol: high-quality Ethyl Alcohol (purity = 99,9% ) to the pharmaceutical and medical industry applications, Sasma supplier, Netherlands
Table 1 physical properties of chemicals

| Chemical name               | Purity | Bulk Density | Color        | Molecular weight | Particles size | Boiling point | Manufacturer  | Country  |
|-----------------------------|--------|--------------|--------------|------------------|----------------|---------------|---------------|----------|
| Calcium carbonate CaCO₃    | >97.5  | 0.68 g/mL    | White powder | 100.0869 g/mol   | 30-50 nm       | -             | Sky Spring    | USA      |
| Dimethyl dichloride saline C₂H₆C₂Cl₆ | >98%   | 1.07 g/mol.  | Transparent liquid | 129 g/mol | -             | 68-70°C       | Fluka         | Germany  |

2.2 Preparation methods: the preparation methods include two parts. The first part is a modification of nanoparticles to increase the compatibility between polymeric blend and the solid particles. The second part is to synthesize nanocomposites samples.

2.2.1 Modification of nano Calcium carbonate (NCC): to improve the compatibility of nanoparticles. NCC was mixed with solution consisted of 3% of coupling agent in ethanol. The mixture was stirred for 3 hours using mechanical stirrer and then using an ultrasonic probe (Heilscher.UP200/400, probe diameter 9 mm and 85% amplitude, Germany) for 1 hour. The obtained suspension left at room temperature for 24 hours to dry.

2.2.2 Preparation of nano-blend composite: polypropylene pallets were mixed well with LLDPE at a constant ratio (90% - 10%). After that, the blend was mixed with different concentrations of modified NCC as it is shown in table 2. All components of blends were fed into twin screw extruder (Chinese type with 20/25 L/D and motor speed 0-100 rpm. It has three thermal processing (180, 220,250) °C. The molten was compressed by a mechanical heating press (10 bars for 10 min and temperature 180 °C in the steel mold with dimension (15 cm x 10 cm x 0.6 cm). The prepared specimens were left for 24 hours at room temperature. After, samples were cured at 80 °C for 2 hours. The blend composites samples were cut in different shapes according to the test requirements.

Table 2 the composition of prepared samples

| Sample code | PP % | LLDPE% | NCC % |
|-------------|------|--------|-------|
| PP0         | 100  | 0      | 0     |
| LLDPE       | 0    | 100    | 0     |
| PLL         | 90   | 10     | 0     |
| PPLL1.5     | 88.5 | 10     | 1.5   |
| PPL3        | 87   | 10     | 3     |
| PPL6        | 84   | 10     | 6     |
| PPL9        | 81   | 10     | 9     |

2.3 characterization tests:

1. Thermal stability:
   a. DSC: Thermal behavior of samples was characterized by differential scanning calorimetry DSC (TA- 8 model, Shimadzu, Japan). The rate of heating was 10 °C/min in the air for thermal range from 20-250°C.
   b. DTA: The degradation temperatures were investigated using Differential thermal analysis (DTG-60, Shimadzu, Japan). Thermal range is 25-450 °C with 10 C/min. heating rate.
2. Mechanical tests: tensile (ASTM-D-638) and Flexural strength (ASTM D-790) were tested by using the mechanical testing machine (H50KT model, Tinius- Olsen, UK).

3. Thermomechanical tests: The thermomechanical assessment of prepared samples was done using thermomechanical analyzer (TMA – PT1000, Linseis Company, Germany).

4. Fire retardancy tests:
   A. Limited oxygen index (L.O.I): this index was tested according to ASTM D 2863
   B. U(94 Horizontal: the rate of burning was done according to (ASTM-D 635- 03) for three specimens. The specimen dimension was (100 mm L*13 mm W *6 mm Thick). In the test, the time of the first drip was recorded.
   C. Ash residue test: it was done according to (ASTM D5630). The samples were heated in a furnace at 600 °C with heating rate 10C/min for 1 hour.

3. Results and discussions:
3.1. Mechanical properties of nanocomposites:
Figures 1, 2 show the results of tensile strength and E-modulus of prepared samples. The results showed that the two properties improved with increasing nano calcium carbonate loadings to reach the maximum at blend sample with 6% but it decreased when the loading increased to 9%. The effect of LLDPE addition can be seen in the results of elongation percent values. The PP elasticity improved with the addition of LLDPE. Table 1 clarifies elongation percent results of samples. The maximum improvement was at PLL6 sample with 6% NCC.

Table 3 elongations results

| Sample code | PP0 | LLDPE | PLL | PLL1.5 | PLL3 | PLL6 | PLL9 |
|-------------|-----|-------|-----|--------|------|------|------|
| Max. Elongation at Beak % | 4.02 | 18.13 | 6.07 | 4.6 | 5.79 | 6.6 | 457. |
| Total Elongation % | 4.48 | 56.8 | 6.43 | 5. | 6.05 | 7 | 4.7 |

Figure 1 tensile strength results

Figure 2 E-modulus results
3.1.1 Flexural strength:
The results of Flexural strength are clarified in figure 3. The nano calcium carbonate loadings contributed to increasing bending strength for nanocomposites. The maximum improvement percent was recorded for 6% NCC sample. 

![Figure 3 Flexural strength results](image)

Role of nanoparticles in improving the mechanical properties is investigated in many works. The effect of nano size of fillers has the key role in improving properties where calcium carbonate particles with tiny size occupy the voids between the molecules of chains of the blend (PP/LLDPE) and working on reinforcing the backbone of PP against applied forces. Nano calcium carbonate particles work on restriction the mobilities of blend chains because of immobility of nanoparticles themselves. As a result, the mechanical properties of nanocomposites samples improve. The interfacial interaction between the blend with particles of calcium carbonate is responsible for the increasing the properties values. Besides, the modification particles surfaces with coupling agent have positive impact on the mechanical properties via increasing the compatibility of non-polar blend with polar nano CaCO₃ which are polar with high surface energy leading to increasing the strength of interaction between blend and the particles. With increasing the nanoparticles to 9% NCC, the mechanical properties deteriorated because of the increasing the particle-particle interfaces instead of the polymer-particle interfaces. This contributed to reduce the strength of bonding against the applied forces. These results agreed with results of Zaman & Beg [26] but the results of tensile strength differ from the obtained results of Nezhad and his team [27] because they used unmodified nanoparticles in their work.

Flexural strength is increased for the same reasons where the particles tend to align in the stretch direction leading to increasing the resistance against the applied force.

The effect of LLDPE is clear in increasing the elongation and elasticity of PP compared to the results of virgin PP elongation where the improvement percent is about 51% at break and 43.53 as a total elongation percent. Besides, the flexural strength of blend is higher than both virgin polymers.

3.2 Thermal analysis:
The results of the thermal analysis that were done by means of DSC & DTA are presented in table 4. The effect of NCC nanoparticles on the melting points of prepared samples was limited. It is noticed that the increase was about 5 degrees for polypropylene from 158 to 163 whereas the increase was about 6 degrees of LLDPE. The improvement of melting point indicates the improvement the crystallinity of composites.
The degradation temperature of composites, which is obtained by DTA test, is improved with the increasing NCC content comparing with pure PP and pure LLDPE. The maximum value was recorded for PPL9 sample and the increasing was about 43 degree. The results are similar to the results of Faud and his team [28].

### Table 4 thermal analysis results

| Sample code | PP0 | LLDP | PPLL | PPL1.5 | PP3 | PP6 | PP9 |
|-------------|-----|------|------|--------|-----|-----|-----|
| Melting point | 158.4 | 119.5 | 117.8 | 159.3 | 122.6 | 159.5 | 125.14 | 161 | 123.1 | 162 | 122. | 163 |
| Temp. of degradation | 400 | 391 | 402 | 406.63 | 416.46 | 417.23 | 443.13 |

#### 3.3 Thermomechanical behavior:

#### 3.3.1 Thermal expansion:

Figure 4 shows the results of the thermal expansion coefficient for the prepared samples. This coefficient has extraordinary importance because it is considered an indication of composite behavior when it is subjected to the thermal heating system. It is noticed that the thermal expansion coefficient of pure LLDPE is the highest compared with other samples but this coefficient decreased with adding nanoparticles and the maximum reduction was recorded for PPL6 sample (6% NCC) where the percent of reduction is about 31.43% compared with PPLL sample and it is about 8.55%, 37.12% for PP, LLDPE respectively.

![Figure 4 thermal expansion results](image)

#### 3.3.2 Thermomechanical E-modulus: the Thermomechanical test is aimed to investigate the response of samples when they are exposed to a heating rate and applied mechanical load simultaneously. Figure 5 clarifies this behavior of composites samples.
7

Figure 5 thermomechanical E-modulus results

Form the data in the above figure, it is noticed that PPL6 (contained 6% NCC) has the maximum value of E-modulus for full thermal range (20 °C -140 °C). The improvement percent is about 140%, 264% compared with pure PP at 20 °C & 140 °C respectively. In addition, the worst values were recorded for pure LLDPE and PP/LLDPE blend.

The thermal expansion and thermal E-modulus results show that the effect of NCC incorporation in PP/LLDPE blend. The nanoparticles addition enhances the immobility of the polymer chain and to support it against thermal and applied force. The enhancement mechanism is similar to improving mechanism of mechanical properties which is explained in above.

3.4 Fire retardancy assessment:
3.4.1 Limited oxygen index (L.O.I): the results of this test are listed in table 5. The maximum index was noticed for PPL9 sample (9% NCC) with 24.21% as the improvement percent. Whereas, the minimum index value was noticed for pure LLDPE which was 17.5.

| Sample code Test | L.O.I | PP0 | PPLL | PPL1.5 | PPL3 | PPL6 | PPL9 |
|------------------|------|-----|------|--------|------|------|------|
| L.O.I            | 17.5 | 19  | 19.2 | 20.7   | 21.8 | 22.4 | 23.6 |
| Improvement %    | -    | -   | 1%   | 8.95   | 14.74| 17.89| 24.21|

3.4.2 Rate of flame
The test was done according to ASTM- D 635- 03 D in which the rate of burning of samples was calculated according to Eq. 1

\[
RB = 60 \frac{L}{t} \quad -------- (1)
\]

Where:
\[
RB = \text{rate of burning (mm/min.)}
\]
\[
L = \text{length of specimen (mm)} \quad \text{and} \quad t = \text{time of burning (sec.)}
\]

The results of this test are clarified in figure 6. It is noticed that the incorporation NCC with blend samples contributed to reduce the burning rate for all addition ratios but the maximum reduction was
recorded for PLL9 (9% NCC). The burning of samples is classified as HB grade according to ASTM standard.

![Figure 6 rate of burning](image)

3.4.3 First drip time:
The results of this test are listed in table 6 below. The behavior of all samples was similar to the behavior of burning rate. The time of first drip falling increased with NPPC concentrations. Sample PLL9 (9% NCC) has the highest dripping time (76 s) with improvement percent was about 61.7% compared with virgin PP.

3.4.3 Ash content: the results are shown in table 6

| Sample code | LLDPE | PP0 | PLL | PPL1.5 | PPL3 | PPL6 | PPL9 |
|-------------|-------|-----|-----|--------|------|------|------|
| Time of 1st drip (sec.) | 23 | 47 | 46 | 51 | 69 | 71 | 76 |
| Improvement percent % | - | - | -2.13 | 8.51 | 46.81 | 51.1 | 61.7 |
| Ash content % | 0.99 | 1.5 | 3.5% | 5.1 | 6.7 | 7.6 | 8.2 |

The flame retardancy of nano calcium carbonate can be interpreted via barrier mechanism which is a suitable explanation of ceramic materials like nano-clay, nano alumina, nano silica etc. This mechanism is explained by [29, 30]. It suggests that nanoparticles are pushed to the interfacial boundaries by molten bubbles of the flaming polymer during degradation and pyrolysis processes in condensed phase and begin to agglomerate forming an insulation barrier working on reducing and preventing the flame from propagation and enhancing the thermal stability of polymer matrix. Depending on the results of L.O.I and rate burning values, it is noticed that NCC helped to reduce the rate of burning via forming a barrier in front of flame but this barrier was not sufficient enough to extinguish the flame because of the high flammability of polypropylene and linear low density polyethylene.
4. Conclusion:

Depending on the results of this work, nano calcium carbonate (NCC) has a positive role when it is used as nanofiller for PP/LLDPE blend. The mechanical, thermal, thermomechanical and fire retardancy properties were improved. The maximum improvement was at 6% sample for mechanical properties, thermomechanical and thermal expansion coefficient. While, the sample with 9% NCC loading has the maximum values for thermal and fire resistance properties. Despite, the improvement in fire resistance of polypropylene with the addition of nano calcium carbonate, self-extinguish action did not be seen for all samples. The results suggest that the possibility of using nano calcium carbonate to improve the properties of polypropylene and its blends.

5. Recommendation:

To increase the efficiency of fire resistance of PP/LLDPE blend with nano calcium carbonate, two solutions are proposed either via:

1. Increasing the nano calcium carbonate loading more than 9% and to assess the fire retardancy
2. Using a mixture of different nano and micro conventional retardants with nano calcium carbonate.

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