Study of Some Polypropylene Nanocomposite Properties

Abou El-Fettoh A. Abd El-Hakim^{1*}, Abdallah A. El-Sawey², A.M. Motawie³, Alaa I. Eid⁴, N.S. Mahruos⁵

¹Polymer Chemistry and Technology, National Research Center NRC, El dokki.
 ²Organic Chemistry, Faculty of Science, Benha University, Egypt.
 ³Petrochemical Department, Egyptian Petroleum ResearchInstitute EPRI, Nasr City, Cairo.Egypt.
 ⁴Metallurgical& Development Institute.
 ⁵El Taqwa Pure Company.

THE PREPARATION of both polymer composites and nanocomposites by melt compounding of polypropylene (PP) with micro $CaCO_3$ content 1, 3, 5, 7, 10, 15 and 20 wt.% and nano $CaCO_3$ content 1, 3, 5, 7 and 10 wt.% using fatty carboxylic acid as compatibilizer has been done. The presence of agglomerations of $CaCO_3$ with increasing the content in the composites must be taken into account. The prepared composites were characterized by using Fourier transform infrared spectroscopy (FTIR), X-Ray Diffraction (XRD), Transmission Electron Microscopy (TEM). Mechanical, Electrical, Thermal, and Physical properties of PP composites was obtained and compared to virgin PP.

Keywords: Polypropylene, Calcium carbonate, Nanocomposite, Physical, Mechanical, Electrical, Thermal properties.

Introduction

Polypropylene (PP) has various advantages such as easy conversion, good (hardness, environmental stress, cracking resistance, fatigue resistance) as well as resistance to temperatures when not subjected to mechanical stress, higher stiffness at lower density and the most distinctive advantage is the addition of low-cost materials such as calcium carbonate, talc, clay and fibre... etc. These materials with low cost play very important role in polypropylene properties.

CaCO₂ is widely used as filler with PP due to the low cost, abundance on nature and could improve mechanical, physical properties and increase the thermal stability. But, there's a main problem of using inorganic minerals with polyolefin is the poor interfacial bonding between them due to hydrophilic nature of the inorganic minerals inherently incompatible with hydrophobic polyolefins [1,2]. To overcome this problem the inorganic minerals usually treated with Carboxylic acids (fatty acids) which used as a coupling agent (Compatibilizer). In this study the mechanical and Thermal properties of PP composites and nanocomposites were investigated. The physical and electrical properties were determined and compared to virgin PP.

Experimental

Materials

PP (Hostalen PP H5416, LYONDELL BASELL) provided by Basell company, its density was 0.897 g/cm3. Melt flow index was 0.3 g/10 min (2.16 kg at 230 °C), 0.5 g/10 min (5 kg at 190 °C) and 1.3 g/10 min (5 kg at 230 °C) and the melting temperatures were 147 °C. Calcium Carbonate (micron size) filler is purchased from the ASCOM 20T is produced from highly white limestone and milled to a medium particle size with CaCO, content 99.6 %, main particle size was 24 - 26 microns, and brightness (95.8%) treated with Stearic Acid 7 - 8 KG/Ton. Calcium Carbonate (nano size) filler is purchased from Solvay, France. (Socal 312, Solvay, France). Ultrafine, white and odorless coated precipitated calcium carbonate with CaCO₂ content 99.6 % and main particle diameter of 70 nm, specific surface area $(17 \text{ m}^2/\text{g})$ and brightness (97%) used as filler phase. PP-g-MAH compatibilizer (Priex 20097, Solvay, France) with maleic anhydride content of 0.05 wt %.

Preparation of PP composites and nanocomposites

All samples were prepared in a Barbender Carl Wihelm (C.W Barbender instruments inc) for plastic-corder inter faceco-rotating twinscrew extruder (D= 20 mm, L = 440 mm and L/D 1/22) Barrel and Screw NO 18252 OB2. Internal mixer at 200-210 °C. Other conditions where 70 r.p.m and 10 min homogenization time. Then, the extruder put into the roll mill for extremely mixing and good uniform distribution then, comprised by the press that for test preparing sample for hardness, melt flow rate, tensile, electrical and thermal analysis. The weight content of Calcium Carbonate (micron size) were 1, 3, 5, 7, 10, 15 and 20 % wt and Calcium Carbonate (nano size) were 1, 3, 5, 7 and 10 % wt.

Characterization of the prepared composites. FTIR spectroscopy

The term Fourier transform (FT) refers to a modern development in the method in 66 which the data are collected and converted from an interference pattern to an infrared absorption spectrum that is like a molecular "fingerprint"[3].

The analysis was carried out by using FTIR 6300. Fourier Transform Infrared spectrometer in the wave number range (400-4000 cm⁻¹).

Morphological Analysis

X-ray Diffraction (XRD)

The samples of the wide-angle X-ray diffraction (WAXD) examinations were cut from the mechanical testing specimens. The WAXD experiments were conducted with PW 710 BASED /PC X-ray diffractometer. Cu K α (λ = 1.54Å) radiation, generated at a voltage of 40 kv and current of 55 mA was used as the X-Ray source. The diffraction angle 2 θ was scanned from 3° to 45° at a scanning rate of 1°/min and a step size of 0.02°. The molded specimens were used as the X-ray diffraction samples.

Transmission Electron Microscopy (TEM) Analysis

TEM is a very effective technique for the study of polymer morphology. Both 84 XRD and TEM are essential tools for evaluating the nanocomposite structure. However, TEM gives qualitative information on the sample such as internal structure, distribution of the various phases, whereas peaks in XRD allow quantification of changes in layer spacing.

The photographs were taken by using(Model: JEM-1230-Made in Japan-JEOL Co.-energy 120KV on steps-Line Resolution: 0.2nm-Max. Magn.:600Kx).

Mechanical Tests

Tensile Test

Tensile tests were performed on five samples

Egypt. J. Chem. 61, No.5 (2018)

for each composition according to the standards of EN-ISO 527-1 [4] and EN-ISO 527-2 [5] (international standards and organization) by using standardized TestometricM500-50ATUniversal testing machine.

Impact Test

The impact strength test shall be carried out according to DIN EN ISO 179-1 (Plastics – Determination of Charpy Impact Strength) with pendulum Ceast Resill mpactoron 10 test pieces using a 15 J pendulum impact testing machine with the blow being applied to the external sample surface or the machined side at velocity = 3.8 m/sec. The test shall be carried out at (23 $\pm 2^{\circ}$ C) by using SANS ZBC- 50A manufacture by SHENZHEN SANS TESTING MACHINE CO., LTD.

Hardness

the test was carried out by using the Shore "D" Durometer with scales from 0 to 100.

Electrical properties

Electrical and dielectric measurements were carried out at room temperature using PM 6304 programmable automatic RCL (Philips) meter, over frequency range from 100 HZ to 100 KHZ.

Thermal Analysis

Thermal Analysis test (Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA)) were carried out at 23 °C (beginning of test) to 500 °C By heating rate 10°C per minute using Universal V4.7A TA Instruments (SDT Q600 V20.9 Build 20).

Physical properties

Density

Density test was carried out at 23 °C, Using Sartorius mecatronics instrument within convex filter blow this because of the density of pp, pp/CaCO₃ composite and nanocomposite <H₂O density.

Water Absorption Test

The test was carried out according to ASTM D 570[6] in temperature 23 °C and humidity 50%.

Flow Characteristics

Melt Flow Index (MFI) Test

The melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) were determined according to iso 1133-1:2011(E) [7] by using IPT 1267-2036308212 Melt flow rate instrument.

Heat reversion

The longitudinal reversion was determined

according to DIN EN ISO 2505[8] using method B, which depend upon determination longitudinal reversion on air. The test was carried out using thermo electron corporation manufacture by heraeus.

Vicat Softening Temperature and Heat Deflection Temperature

Vicat Softening Temperature test was carried out according to ASTM D-2515-00 and Heat Deflection Temperature test was carried out according to ASTM D648 [9] or ISO 75 using (VST)/B/50/K/h(50N)).

Results

Physicochemical characterization FTIR spectroscopy

There are new peaks in Fig.1 appears nearly at 1735, 2018 and 2125 cm⁻¹ represented CH₃ groups of PP. the peak at 2912 cm⁻¹ represented CH₃ groups. Increasing loads of CaCO₃ in PP matrix didn't affect in the intensity and position of the peaks. This indicates that there is no interaction between PP and CaCO₃ particles. The CaCO₃ particles are existed inside the PP particles by physical interaction[3].

Morphological Analysis

X-Ray Diffraction (XRD) Analysis

For virgin PP presents peaks characteristics of α phase in 2 θ values of 14.365, 16.905, 18.86, 21.375 and 22.295° corresponding to (110), (040), (130), (111) and (131) lattice planes of the most common α -monoclinic crystal[10,11]. For both PP/CaCO₃ composite and nanocomposite represented in Fig. 2, there are new peaks at 2 θ of around 16.0° was observed. It is assigned to (300) lattice plane of β -crystal, indicating the existence of β -crystal in PP composites and nanocomposites [13], due to presence of CaCO₃.

Transmission Electron Microscopy (TEM) Analysis

For PP/CaCO₃ composite (Fig.3), it was showed that there's a good dispersion of CaCO₃ in PP matrix within low contents 3 and 7 %, but for higher contents 15 % CaCO₃ begin to agglomerate.

For PP/ CaCO₃ nanocomposite (Fig.4), due to strong tendency of nanoparticles to agglomerate than micro, the percent of nano CaCO₃ add to PP forming the nanocomposite is less than that of the composite. It was showed that there's a good dispersion of CaCO₃ in PP matrix within low contents 3 and 5 %, for high contents 7 % CaCO₃ begin to agglomerate.







Fig. 1. IR spectrum for PP/CaCO₃ composite and PP/CaCO₃ nanocomposite.

PP/CaCO₃ composite



PP/CaCO3 nanocomposite



Fig. 2. XRD for PP/CaCO₃ composite and PP/CaCO₃ nanocomposite.

Egypt. J. Chem. 61, No.5 (2018)



Fig. 3. TEM micrographs for PP/CaCO₃ composite.



Fig. 4. TEM micrographs for PP/CaCO₃ nanocomposite.

						Ū.			
CaCO ₃ % contents	Total test sample	pass sample	failure sample	Results	CaCO ₃ % contents	Total test sample	pass sample	failure sample	Results
0%	10	10	0	pass	0%	10	10	0	pass
1%	10	10	0	pass	1%	10	10	0	pass
3%	10	10	0	pass	3%	10	10	0	pass
5%	10	10	0	pass	5%	10	10	0	pass
7%	10	10	0	pass	7%	10	10	0	pass
10%	10	10	0	pass	10%	10	10	0	pass
15%	30	28	2	pass					
20%	30	26	4	fail					

Mechanical properties study

a-PP/CaCO₃ composite.

TABLE2. Hardness results for PP/CaCO₃ composite and PP/CaCO₃ nanocomposite.

TABLE 1. Impact results for PP/CaCO₃ composite and PP/CaCO₃ nanocomposite.

	USILC.	D-11/CaCO ₃ nanocomposite.				
CaCO ₃ % contents	Hardness(shore D)	CaCO ₃ % contents	Hardness(shore D)			
0%	65	0%	65			
1%	66	1%	66			
3%	67	3%	67			
5%	67	5%	68			
7%	66	7%	69			
10%	68	10%	70			
15%	69					
20%	70					

At these percent which give the best mechanical properties and flow rate of the composite, within increase of $CaCO_3$ addition the mechanical properties and flow rate of the prepared composite decreased given lower results for the composite due to $CaCO_3$ agglomeration.

Agglomeration and nano $CaCO_3$ diameters play important roles in properties level of samples. The agglomeration extent increases by addition of nano $CaCO_3$ content and reduction of nano $CaCO_3$ size. Moreover, the agglomeration decreases the effectiveness of nano $CaCO_3$ in polypropylene matrix, which finally results in the poor properties of samples [12].

Tensile Test

Increasing the amount of $CaCO_3$ in PP composite from 1 % up to 20 %, enough to decrease the tensile strength from 28.803 to 21.544 Mpa and the elongation increase from 123.47 % to about 200 %, recording it's maximum

value at 15 % (229 %) referring to collapse in the mechanical properties, where the young's modulus changed from 830.296 up to 805.225 mm recording it's maximum value at 5 % (1022.267 mm). For PP/ CaCO₃ nanocomposite, increasing the amount of nano CaCO₃ in PP from 1 % up to 10 %, enough to decreases the tensile strength from 28.803 to 25.404Mpa and the elongation increase from 123.47 % to about 235 %, recording it's maximum value at 7 % (246 %) referring to collapse in the mechanical properties, where the young's modulus changed from 830.296 up to 343.443 mm recording it's maximum value at 7 % (888.683mm) (Fig. 5).

b-PP/CaCO₃ nanocomposite.

In general at the appropriate manufacture conditions the addition of $CaCO_3$ to PP improved the tensile properties, due to good dispersion, good coherent, better physical adhesion and strong interactions between them [13].

PP/CaCO₃ composite



PP/CaCO₃ nanocomposite



Fig. 5. Stress & Strain curve for PP/CaCO₃ composite and PP/CaCO₃ nanocomposite.

Egypt. J. Chem. 61, No.5 (2018)

Impact Test

The results represented in Table 1 showed that, for PP/ CaCO₃ composites the last sample within CaCO₃ contents 20 % was failed, this's because there's no homogenous distribution and agglomeration occurs, the addition of CaCO₃ had a negative effect on the impact strength of virgin PP [14]. But for PP/ CaCO₃ nanocomposites, all the samples were passed the test, this means that there's a homogenous distribution of nano CaCO₃ inside PP matrix [14].

Hardness

It was shown that for both PP/ $CaCO_3$ composites and nanocomposites (Fig.6) hardness increase with increasing $CaCO_3\%$ contents, depending upon the distribution of filler into the matrix[15].

Electrical properties

AC conductivity.

The frequency dependence of $\delta_{tot}(w)$ for PP/ CaCO₃ composite and nanocomposite (Fig 7), the general behavior describe mild increase of $\delta_{tot}(w)$ with increasing frequency within relatively low frequency range followed by strong reliance beyond certain frequency relative to the following universal law:

where,

 $\delta_{ac}(w)$ is the direct current conductivity.

 $\delta_{dc}(w)$ is the frequency dependence conductivity.

DC conductivity

The dependence of (δ_{dc}) of PP/ CaCO₃ composite and nanocomposite on temperature (Fig. 8) can be separated into three regions:-

- a- Region (I) from 303333- K, in this region the increasing in (δ_{dc}) could be due to the hydroscopic nature of the samples ease to absorbed water, which provide H⁺ and OH⁻ ions that cause sharp increase in conductivity.
- b- Region (II) from 333393- K, conductivity decreased due to desorption process which reduced H⁺ and OH ions[16].
- c- Region (III) from 393433- K, the conductivity begin to increase due to the thermal activation of charge carriers from the shallow traps (ΔE =0.13-0.48 *ev*).

Dielectric properties

The variation of dielectric constant (ε) as function of frequency at room temperature for PP/ CaCO₃ composite and nanocomposite (Fig.

9), respectively it was proved that ε independent upon frequency. This indicates that PP is non-polar polymer[17].

It was showed that the dielectric constant (ε) initially increase with increasing CaCO₃ contents due to increase in polarizability group with the incorporation of CaCO₃. Then, after 10 wt. % of CaCO₃ for PP/CaCO₃ composite and 5 wt. % for PP/ CaCO₃ nanocomposite, respectively, the value of the dielectric constant (ε) decreased due to CaCO₃ agglomerations which sufferance the dipoles to be oriented with the oscillatory applied electric field.

Thermal Analysis

For TGA results:-

It was found that virgin PP showed the lowest thermal stability, based upon T_{d} , 419.03°C than PP/CaCO₃ composite and nanocomposite (Fig. 10).

For DSC results

It is clear that both Tonset and T_m are nearly unchanged with all CaCO₃ contents where ΔH_m recording its highest degree at 5 wt. % for composite and 1 wt. % for nanocomposite recording highest degree of crystallinity fraction, Can be attributed to the increase of composite viscosity during processing which resists the PP structure rearrangement (Fig.11).

The degree of crystallinity fraction (DOC (%)) can be calculated from the following equation:-

$$DOC (\%) = \frac{\Delta H m}{(1 - Wf) \cdot \Delta H^{\circ} m} \times 100$$

Where,

 w_f is the weight fraction of PP in the composite H_m is the experimental melting enthalpy

 H_{m}° is the melting enthalpy for 100% crystalline PP, (207.1 J/g).

Physical properties

Density

Density results for both PP composite and nanocomposite (Fig.12) gradually increases with increasing content of CaCO3, this is due to higher density of CaCO3.

Water Absorption Test

According to hydrophilic nature of $CaCO_3$, Water absorption increase with increasing $CaCO_3$ contents in PP matrix especially in $CaCO_3$ within micron size, due to increasing the exposed surface of water absorption compared with $CaCO_3$ within nano size (Fig.13), enabling to compatible with PP.





Fig. 6. Hardness(shore D) results for PP/ CaCO₃ com¬posites and PP/ CaCO₃ nanocom¬posites.





PP/CaCO₃ nanocomposite

Fig. 7. Frequency dependence of the AC Conductivity at room temperature for PP/CaCO₃ composite and PP/CaCO₃ nanocomposite.





Fig. 8. DC conductivity versus (1000/T) for PP/CaCO₃ composite and PP/CaCO₃ nanocomposite.





Fig .9. The relation between dielectric constant (ε,) and frequency at room temperature for PP/CaCO₃ composite and PP/CaCO₃ nanocomposite.







Fig. 11. DSC for PP/CaCO₃ composite and PP/CaCO₃ nanocomposite.



Fig. 12. Density for PP/CaCO₃ composite and PP/CaCO₃ nanocomposite.

838



Fig. 13. Water absorption for PP/CaCO₃ composite and PP/CaCO₃ nanocomposite.

Flow Characteristics

Melt Flow Index (MFI) Test

the results shown in Fig.14 that Melt Flow Rate increases with increasing $CaCO_3$ contents, due to decreasing flow resistance then decrease:-

- For the PP/ CaCO₃ composites, Melt Flow rate (MFR) increases till CaCO₃ contents 15% then decreases.
- 2- For the PP/ CaCO₃ nanocomposites, Melt Flow rate (MFR) increase till CaCO₃ contents 7% then decrease.

Decreasing in Melt Flow rate after increasing due to torque increasing caused by adhesion take

place between the material and (external surface of screw and internal surface of barrel).

Heat reversion

All results in Fig.15 showed that heat reversion affected with increasing $CaCO_3$ contents, Longitudinal contraction decreases with increasing $CaCO_3$ wt. %, which Interfaces in PP matrix.

Vicat Softening Temperature and Heat Deflection Temperature

In accordance with hardness results, it was found that both VST in Fig. 16 and HDT in Fig.17 increase with increasing CaCO₃ contents for both PP/CaCO₃ composite and nanocomposite.



Fig. 14. MFR for PP/CaCO, composite and PP/CaCO, nanocomposite at 230 °C/2.16 kg.



Fig. 15. Heat reversion for PP/CaCO₃ composite and PP/CaCO₃ nanocomposite.



Fig. 16. VST for PP/CaCO₃ composite and PP/CaCO₃ nanocomposite.



Fig. 17. HDT for PP/CaCO₃ composite and PP/CaCO₃ nanocomposite.

Egypt. J. Chem. 61, No.5 (2018)

<u>Conclusion</u>

FTIR results showed that there is no chemical change in the prepared PP composite. XRD results indicated that there is no effect on the crystallinity of PP by adding CaCO₂. TEM results indicated good dispersion of CaCO₃ especially nano size till certain value then, agglomeration occurs. Mechanical properties indicated that strain at break and elongation at break improved and reach its maximum value at 15 wt. % micro CaCO, and 7 wt. % nano CaCO₂. Impact Test showed failing of micro CaCO₂ composite with 20 wt. %. Electrical properties, mild increase of $\delta_{tot}(w)$ with increasing frequency within relatively low frequency range followed by strong reseliance beyond certain frequency. Dielectric constant remains constant with increasing frequency for each sample, but increase till the concentration of 10 wt. % micro CaCO₂ and 5 wt. % nano CaCO₂ then decreases. Thermal Analysis, slight improvement in thermal stability of PP by adding CaCO₂ but, both Tonset and T_m are nearly unchanged. MFR improved and reach its maximum value at 15 wt. % micro CaCO₃ and 7 wt. % nano CaCO₃ then decrease. Longitudinal contraction decrease with increasing CaCO₂ wt. %. Finally, it was detected that density, Water absorption, Hardness, VST and HDT increases with increasing CaCO₃ wt. %.

References

- Toro P., Quijad R., Peralta R., Yazdani-Pedram M., Influence of grafted polypropylene on the mechanical properties of mineral-filled polypropylene composites. *J Appl Polym Sci.*; 103(4), 2343–50. (2007).
- Zhidan L., et al. Crystallization and melting behavior of polypropylene in b-PP/polyamide 6 blends containing PP-g-MA. J Ind Eng Chem.; 19(2), 692–7 (2013).
- Griffiths P.R., DeHaseth J. A., Fourier Transform Infrared Spectroscopy, John Wiley and Sons Inc., New York (1986).
- Czichos, Horst HYPERLINK "https://books. google.com/books?id=8lANaR-Pqi4C"Springer Handbook of Materials Measurement Methods. Berlin: Springer. pp. 303–304. HYPERLINK "https://en.wikipedia.org/wiki/International_ Standard_Book_Number" \o "International Standard Book Number"ISBN HYPERLINK "https://en.wikipedia.org/wiki/Special:BookSo urces/978-3-540-20785-6" \o "Special:BookSo

urces/978-3-540-20785-6"978-3-540-20785-6. (2006).

- DIN EN ISO 527-1, Plastics Determination of tensile properties — Part 1:General principles (2012).
- ASTM D 570,"Standard Test Method for water absorption (1995).
- HYPERLINK "https://www.iso.org/obp/ui/" \l "iso:std:iso:1133:-2:en"ISO 1133, Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-rate (MVR) of thermoplastics, part 1:(2011)(E).
- ISO 2505:, Thermoplastics pipes -- Longitudinal reversion -- Test method and parameters (2005)
- 9. ASTM D 648-6 Test method for deflection temperature (2007)
- Yuan Y., Chen B. and Zhang X. Study on the formation of β-crystal during the crystallization process of polypropylene reactor granule. *Polymer.*; 47(15), 5480-5483 (2006).
- Hou W.M., Liu G., Zou J.J., Gao X., Li Y., Li L. et al. The influence of crystal structures of nucleating agents on the crystallization behaviors of isotactic polypropylene. *Colloid and Polymer Science.*; 285(1), 11-17 (2006).
- 12. Yasser Zare "Study of nanoparticles aggregation/ agglomeration in polymer particulate nanocomposites by mechanical properties. composites Part *Applied Science and Manufacturing* May (2016)
- 13.Cai H. Z., Yang K. Y., Yi W. M., Effect of Calcium Carbonate on Preparation and Mechanical Properties of Wood/Platic Composite. *Int. J. Agric. & Biol. Eng.* **10** (1), 184-190 (2017).
- 14. ASTM D 3029 or D 256-93a Impact Strength.
- 15. ASTM D 2240 Hardness measurements .
- Blythe T., Bloor D., "Electrical Properties of Polymers" 2th Ed., Cambridge University Press (2005)
- Myers H. P., " Introductory Solid State Physics," Taylor & Francis, London (1990)

(Received 22/5/2018; accepted 26/6/2018)

دراسة بعض خواص المتراكبات النانويه للبولى بروبلين

أبوالفتوح عبدالمنعم عبدالحكيم', عبدالله عبدالمنعم الصاوى', أحمد مجدى حسين مطاوع'',علاء ابراهيم أحمد عيد', نائل شكرى محروس سعيد' 'قسم مواد التعبئة والتغليف بالمركز القومى للبحوث كلية العلوم - جامعة بنها معهد بحوث البترول 'مركز بحوث و تطوير الفلزات ' شركة التقوى والنقاء

تم اختيار بولى البروبلين ليكون موضوع در استنا حيث انة يمتلك خصائص تقع بين بولى الايثلين منخفض الكثافة ((HDPE) ع) بولى الايثلين عالى الكثافة , و لقد سعينا فى هذة الدراسة الى تحسين الخواص الفيزيائية & الحرارية و الميكانيكية للبولى بروبلين من أضافة كربونات الكالسيوم العادية و النانوية لما تتميز بة من (رخيصة الثمن & متوفرة بكثرة) علاوة على الكثير من الخصائص التى تجعلها من أفضل الأضافات الشائعة التى تستخدم لعمل الosite . بتحضير عينات الأتية:-

PP/CaCO₃ composites بالتركيزات الأتية (1, 3, 5, 7, 10, 15 20 %)

* PP/CaCO₃ nanocompositesبالتركيزات الأتية (1, 3, 5, 10 7 and 7 %) بأستخدام طريقة العجن فى العجانة تُنائى الفتيلة عند درجة حرارة تتراوح ما بين 200-210 درجة سيليزية و عدد لفات 70 لفة فى الدقيقة و زمن خلط 10 دقائق لضمان تجانس الخامة.

تم توصيف العينات المحضرة بأستخدام التقنيات الأتية:-

(aتم در اسة طيف الأشعة تحت الحمراء (FT-IR Transmission Spectra) في المدى من (4000- 4000) سم⁻¹ لكلا من PP/ CaCO₃ composites and nanocomposite. حيث أوضحت النتائج ظهور بعض القمم تشير الى امتصاص بعض الروابط مثل C=O, C=C and C-H

b) تم دراسة حيود الأشعة السينية ((X-ray Diffraction XRD) لكلا من

PP/ CaCO₃ composites and nanocomposite. حيث أوضحت النتائج وجود قمم للشدة عند بعض زوايا الحيود تشير أحداهما الى بولى البروبلين و قمة واحدة تشير الى الكالسيوم كربونات دليل على عدم تأثر الشكل البلورى.

c) تم دراسة تضاريس السطح لكلا من PP/ CaCO₃ composites and nanocomposite الميكروسكوب الالكترونى النافذ ((TEM حيث أظهرت النتائج أن بزيادة تركيز الكالسيوم كربونات يؤدى الى ظهور تجمعات غير متجانسة مع بولى البروبلين.

تم در اسة الخواص الميكانيكية (الشد-الصدمات-الصلادة) و أوضحت النتائج ما يلى:-

- مزيادة خواص الشد الى أقصى درجة عند 15% في PP/CaCO3 composites و 7% في

PP/ CaCO3 nanocomposites ثم التراجع نتيجة ظهور تجمعات غير متجانسة مع بولي البروبلين. -b عدم أجتياز العينة التي تحتوى على %PP/CaCO3 composites لأختبار الصدمات مع أجتياز جميع العينات في

ال PP/ CaCO₃ nanocomposites نتيجة الأنتشار الجيد ل الكالسيوم كربونات (نانوية) في بولى البروبلين. - ريادة الصلادة بزيادة تركيز الكالسيوم كربونات في بولى البروبلين في نطاق التركيزات المستخدمة.

٣- دراسة الخواص العزل الكهربي عند درجة حرارة الغرفة و ترددات مختلفة و التي أظهرت ثبات (3) عند تلك الترددات.
٢- دراسة العلاقة بين ال (3) و التركيز ات المختلفة ل الكالسيوم كربونات و التي أوضحت زيادة ال (3) عند ٢٠٪ في composites PP/CaCO.

nanocomposites PP/ CaCO, ثم التراجع نتيجة ظهور تجمعات غير متجانسة مع بولى البروبلين.

٤ - تم دراسة الخواص الحرارية لكلا من composites and nanocomposite PP/ CaCO, باستخدام كلا من ال (TGA) و (DSC) حيث أظهرت النتائج عدم وجود تغيير في درجة حرارة الأنصهار للبولي بروبلين مع زيادة تركيز الكالسيوم كربونات. زيادة معدل الثبات الحراري للبولي بروبلين بأضافة الكالسيوم كربونات.

- كما تم در اسة الخواص الفيزيانية (الكثافة-الأنتفاش بالماء-معدل تدفق الخامة المنصهرة) وأوضحت النتائج ما يلى: - ه زيادة الكثافة مع زيادة تركيز الكالسيوم كربونات.

-b زيادة معدل الأنتفاش بالماء خصوصا في composites PP/ CaCO, نظر الزيادة المساحة المعرضة للماء عن /PP . . nanocomposite CaCO,

-c زيادة تدفق الخامة المنصبهرة (MFR) الى أقصى درجة عند ١٥٪ في

composites PP/CaCO, و ۷٪ في nanocomposites PP/CaCO ثم التراجع نتيجة زيادة حمل الماكينة نظرا لحدوث التصاق بين الخامة المستخدمة مع كلا من (سطح الأسطوانة الداخلي و سطح الفتيل الخارجي). ٦- ايضا تم در اسة معدل الأنكماش الحراري لكلا من

,composites and nanocomposite PP/ CaCO و أوضحت النتائح نقص معدل الأنكماش الحر ارى مع زيادة تركيز الكالسيوم كربونات.

٧- وأخيرا تم دراسة كلا من (VST) و (HDT) والذين يزيدان مع زيادة تركيز الكالسيوم كربونات.