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Apparent Properties Characterization for PMMA/ Solid Lubricant, PMMA/ MWCNT Composites via in Situ Polymerization

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ABSTRACT

In situ bulk polymerization of methyl methacrylate was studied in the presence of multi-walled carbon nanotubes (MWCNT) and a lubricant (calcium stearate, polyethylene wax, and beeswax). Both the heat released over time and the isothermal temperature were calculated. The in situ bulk polymerization of methyl methacrylate was characterized using DSC and FTIR to evaluate the thermal behavior. It showed improved chemical miscibility, and it was observed that the large particle sizes of both beeswax and calcium stearate hindered the polymerization and delayed the reaction time. As for the very small nanoparticles on the microscopic scale, they lead to fast polymerization reactions. The glass transition temperature (Tg) and entropy were measured using DSC. A significant improvement in the polymerization rate, an increasing in Tg of nanocomposites and self-lubricating composites compared to pure PMMA and produces ductile and less rough structures.

Key Words: self-lubricating composites, Nano-composites, plasticizer, in situ polymerization, chemical miscibility.

INTRODUCTION

During the past almost 30 years, organic polymer nanocomposites have captured increasing attention from researchers around the world, as they often have unique hybrid physicochemical properties, dating back to their synergistic origins from the two components [1]. The concept of nanocomposites made of polymer and nanoparticles was introduced by researchers Prof. Pulikle M. Agyan. In 2000, they published a paper demonstrating the possibility of enhancing PMMA with MWCNTs to produce nanocomposites that would improve the physical, chemical, and tribological properties. Further research and development have been conducted in this field by many scientists and research groups, which has contributed to the understanding and application of MWCNT/PMMA Nano-composites [2]. This paper examines the possibility of advanced dispersion of nanoparticles in a methyl methacrylate matrix using mixing and the ultrasonic technique by adding plasticizing particles and studying the resulting reaction kinetics.. In particular, the process has shown extreme benefit when dealing with toxic compounds as is the case with nanoparticles. The literature focusing on in situ polymerization technology and reaction kinetics is briefly reviewed next. In 2003, Li et al. developed PMMA/MMT nanocomposites via in situ polymerization [3]. The study showed that the nanocomposites possessed a partially exfoliated and partially intercalated structure, while their thermal stability, glass transition temperature (Tg), and mechanical properties were significantly improved compared to pure PMMA. Moreover, the Tg and thermal decomposition temperature of nanocomposites Xie et al.[4]. Mengya Shang et al. used multi-walled carbon nanotubes (MWCNTs) as reinforcement through an in situ polymerization method to prepare PMMA/MWCNTs composites by changing the reaction conditions (reaction time, polymerization temperature, MWCNTs content). The study shows that when the content of MWCNTs is 3 wt. %, the thermal conductivity of the composite is 0.335 W/ (m-K), which increases by 138%, and the electrical conductivity is 3.94 S/m with the thermal stability of the composite being greatly enhanced. Therefore, they emphasized the widespread use of modified PMMA in medicine, communications, electronics, and other fields. Changchun Zeng et al recommended that through the synergistic combination of improved synthesis methodology and surface functionalization of CNTs. The presence of CO2 as a foaming agent profoundly affects the cell

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nucleation mechanism and cell morphology. However, both the synthesis and surface functionalization methods of carbon nanotubes affect the dispersion of MWCNTs in the polymer matrix, so MWCNTs can be effective heterogeneous nucleation agents that increase the density of Cells at the lowest concentration of particles. Therefore 1% MWCNTs indicates an increase in cell density by ~70-fold and a decrease in cell volume by ~80% in the composite Nano-foam [5]. Lanjuan Xu et al concluded that a dense and stable interconnected network structure (i.e. carbon layer) is formed for CNT/PMMA composites, which can effectively prevent the combustion of pyrolysis products, prevent the transfer of heat and combustible gas and finally stop the combustion of composite materials [6]. Ke Zhang et al confirmed that enhancing the adsorption of multiwalled carbon nanotubes (MWNT) onto the main chains of poly (methyl methacrylate) (PMMA) using thionyl chloride and hexamethylene diamine led to the amino-functionalized MWNT was covalently linked to PMMA [7]. Lachache et al. They adopted additions of ammonium polyphosphate (APP), Al2O3, and TiO2 in

PMMA, which enhanced thermal stability and reduced the heat release rate (HRR) of PMMA. They confirmed that the catalytic effects of the oxide surface were a reason for modifying the decomposition path of PMMA and the formation of a carbonized and ceramic structure [8]. Zheng J et al. in their paper proposed the increase or decrease of PMMA/SiO2 as an indicator of the average molecular weight of nanocomposites compared to neat PMMA, taking into account the silica modification [9]. Shih et al. Xie XL et al concluded that nanoparticles of Sb2O3 do not inhibit the polymerization of MMA during in situ MMA/Sb2O3 polymerization [10]. Roman F et al found the use of organically modified MMT to enhance the reaction kinetics of epoxy resins [11]. Slightly higher maximum degrees of conversion were obtained for radio-catalytic di-methacrylate-based nanocomposites, Peila R et al. [12]. With changing the reaction conditions (time, polymerization temperature, and the content of MWCNTs), the effects of different reaction conditions on the properties of the composites were studied. Shows that the mechanical properties, thermal/electrical conductivity, and thermal stability of the composites are improved compared with the PMMA matrix[13]. By cross-linking carbon nanotubes with poly methyl methacrylate, nylon 6, and polystyrene. Convergence in the characteristic spectroscopic behaviors of each compound with different loading ratios of carbon nanotubes was observed using FTIR and Raman spectroscopy to analyze the interactions between functional and non-functional nanomaterials and polymeric materials[14]. Indeed, after the incorporation of bulk CNT powders, their chemical modification into various commercial products (rechargeable batteries, automobile parts, sporting goods, etc.) and their purification enable the integration and development of carbon nanotubes in thin-film electronics and large-area coatings[15]. Using two different preparation techniques, the first approach was in situ polymerization with a reducing agent (HH), resulting in R-(GO-PMMA)(GO) compounds. In the second approach, polymerization with a free radical initiator produces RGO-(PMMA) compounds. The success of the first method was indicated in the presence of MWI, which has better shape and dispersion with enhanced thermal stability compared to compounds prepared without MWI[16].

MATERIALS AND METHODS

Methyl methacrylate monomer and benzoyl peroxide (BPO) polymerization initiator were purchased from the local market. The comparative effect of three types of lubricant solids used as fillers with proportions shown in table (1).

Table -1: Illustrate PMMA\solid lubricant, PMMA\MWCNT Compositions.

Sample	Composition
Pure PMMA	PMMA without additives
PMMA/CS	PMMA+ (0.05, 0.1, 0.2, 0.5 wt. %) Calcium stearate
PMMA/Bwax	PMMA+(0.05, 0.1, 0.2, 0.5 wt. %) Beeswax
PMMA/PE-wax	PMMA+(0.05, 0.1, 0.2, 0.5 wt. %) Polyethylene-wax

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PMMA/MWCNT	PMMA+(0.05, 0.1, 0.2, 0.5 wt. %) MWCNT

FRP process carried out through except the solid lubricant fillers were added to the solution (MMA (100g) + BPO (0.9%wt.)), Fig. (2). During mixing state with percentages for 15 min. PMMA /Solid lubricants composites (PMMA/PE-wax, PMMA/Bwax, and PMMA/CS) were prepared using a magnetic stirrer. The polymerization process (free radical method) was carried out in the laboratory using methyl methacrylate (MMA) and Benzoyl Peroxide (BPO) as initiators, The polymerization molds were brought into the oven for 6 hr. at a temperature of 80 0C. The comparative effect of three types of lubricant solids used as fillers was added to the solution (MMA (100g) + BPO (0.9%wt.)) for (15 min). PMMA /Solid lubricants composites (PMMA/PE-wax, PMMA / Bwax, and, PMMA / CS) were prepared using a magnetic stirrer, while the PMMA/MWCNT filler was prepared using ultra-sonication process, Fig. (2), for (20 min) at room temperature.

Fig -1: Illustrate polymeric composites.

While PMMA/MWCNT filler prepared using an ultra-sonication process for (20 min) at room temperature.), the mixture (H2SO4+HNO3 (3:1)) was magnetically stirred during the treatment to facilitate the reaction. When the reaction was completed, the mixture was cooled down slowly and washed with distilled water several times using a vacuum filter to react the PH (from 6.5 to 7). The resultant filtered cake was dried in a vacuum oven at 100°C for 8 hr., Fig. (2).

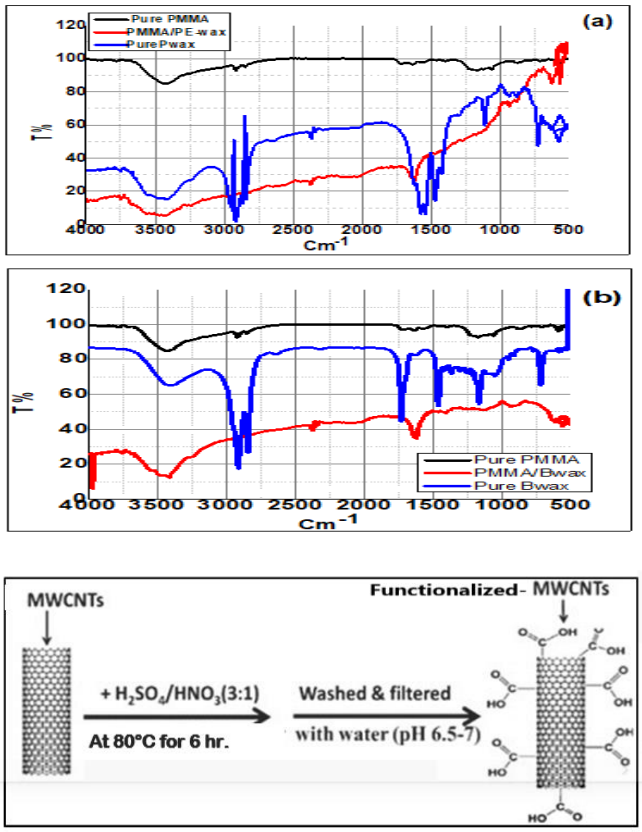

Fig. -2: Illustrate the functionalized MWCNT Preparation Method.

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RESULTS AND DISCUSSION

FTIR Measurements

Polymeric nanocomposites can be described as miscible or partially miscible. The specific form depends on the MWCNT, the plasticizer content, the chemical nature of the acids, and the method used. In general, the system matrix is more feasible with lower filler content (about 0.05-0.1 wt. %), while an agglomerated structure is frequently observed for nanocomposites with higher Nano-filler content and a more streamlined structure for ductile materials. FTIR results for pure PMMA and all PMMA nanocomposites with different Plasticizers are shown in Fig. (3). Fig.(3, a) shows that the intensity of the bands observed in the spectrum (O-H Stretch) starts increasing with the emergence of a doublet at (3757–3456 cm-1), indicating the structural transformation of the molecule, also, at (3903-3672 cm-1) for Pure PE-wax, but shows a wrinkled shape for Pure PMMA. The spectrum (C-H Stretch) shows this doublet at (2947–2846 cm-1) for Pure PE-wax and (2924 cm-1) for Pure PMMA, but less shifting for PMMA/PE-wax at (2846 cm-1). The (C-O stretching bands) observed at (1550-1660 cm−1) in Pure PMMA is less shifting in PMMA/PE-wax at 1635 cm-1, and the (C-C stretching bands) observed at (1110 cm−1)in PMMA/PE-wax. Fig. (3, b), (C–O Stretch) at (1550-1600 cm-1) respectively for PMMA is higher shifting in the PMMA/Bwax peak, which appears at (1643 cm-1). The (O-H Stretch) here less shifted with the emergence of a doublet at (3572 - 3433 cm-1) for PMMA\Bwax. The(C-H Stretch) here is less shifted at (2384cm-1) for PMMA \Bwax. Fig. (3, c), (O-H Stretch) for PMMA\CS show a decrease of this peak in the same position for Pure PMMA. The(C-H Stretch) is less shifting for PMMA/CS spectrum at (2769 cm-1), the same band appears for pure CS at doublet (2962-2769 cm-1), the (C-O Stretch) is higher shifting for PMMA/CS at (1635 cm-1), the(C=O Amide) observed at (1658 cm−1) in PMMA/CS. Fig.(3, d) , pure PMMA, and PMMA/MWCNT compounds are shown. When comparing the FTIR specification set for pure PMMA and PMMA /MWCNT composites, only small peak shifts and one significant change can be seen. Specifically, the(C-O stretch) observed at (1627 cm−1 & 1573 cm−1) in pure MWCNT and PMMA\MWCNT respectively, took a wrinkled shape in both PMMA/MWCNT. The decrease of these peaks in the range (1597-1490 cm-1) indicates the changes in the structure of the carbon nanotubes when carboxylate is used which agrees with [17][18]. It is also correct to refer to such a decrease in these peaks as the presence of hexagonal asymmetric carbon which agrees with [15]. The (O–H stretch) appeared in PMMA/ MWCNT at (3500 cm−1). Tables 2, 3, 4, 5 illustrates the absorption area of FTIR results.

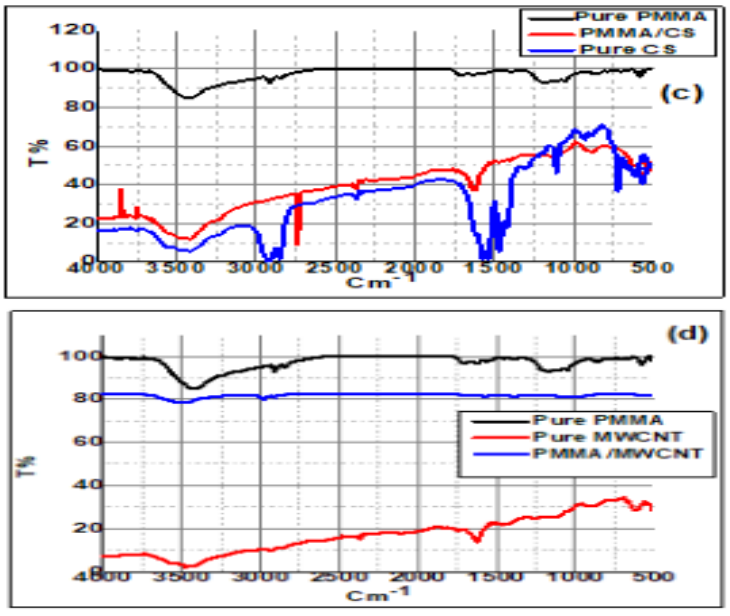

Fig -3: FTIR spectrum for Pure PMMA with (a),PMMA/PE-wax , Pure PE-wax ,(b)PMMA/Bwax , Pure Bwax , (c)PMMA/CS , Pure CS , and (d) Pure MWCNT,PMMA/MWCNT.

Type of bond	Pure PMMA	Pure Pwax	PMMA/Pwax

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O - H Stretch	3448 cm^{-1}	3903-3672 cm^{-1}	3757–3565 cm^{-1}
C - H Stretch	2924 cm^{-1}	2947 – 2846 cm^{-1}	2846 cm^{-1}
C – O Stretch	1550-1600 cm^{-1}	1519 cm^{-1}	1635 cm^{-1}

Table -2: Absorption areas of Fig. (3a)

Type of bond	**Pure PMMA**	**Pure Bwax**	**PMMA/Bwax**
O - H Stretch	3448 cm^{-1}	3417 cm^{-1}	3572-3433 cm^{-1}
C - O Stretch	1550-1600 cm^{-1}	1774 cm^{-1}	1643 cm^{-1}
C - H Stretch	2924 cm^{-1}	2916 cm^{-1}	2384 cm^{-1}

Table -3: Absorption areas of Fig. (3b)

Table -4: Absorption areas of Fig. (3 c)

Type of bond	**Pure PMMA**	**Pure CS**	**PMMA/CS**
O - H Stretch	3448 cm^{-1}	3919 - 3672 cm^{-1}	3919 - 3672 cm^{-1}
C - H Stretch	2924 cm^{-1}	2962 - 2769 cm^{-1}	2769 cm^{-1}
C - O Stretch	1550-1600 cm^{-1}	1550 cm^{-1}	1635 cm^{-1}

Table -5: Absorption areas of Fig. (3 d)

Type of bond	**Pure PMMA**	**Pure MWCNT**	**PMMA/MWCNT**	**Hybrid**
O - H Stretch	3448 cm-1	3433-3556 cm^{-1}	3500 cm^{-1}	3332 cm^{-1}
C - H Stretch	2924 cm-1	2862-2947 cm^{-1}	2870 – 2985 cm^{-1}	2916 cm^{-1}
C - O Stretch	1550-1600 cm^{-1}	1627 cm^{-1}	1573 cm^{-1}	
C ≡ C Stretch		2384 cm-1		

Kinetic of the reaction & DSC results

From second thermodynamic low [21]:

$\Delta S = \frac{\Delta Q}{T}$ … (1)

$\Delta H = Q = m\ cp\ \Delta T$ … (2)

$\Delta H = T\Delta S$ … (3)

Fig.(4) Shows radar graph representations of lubricants, orientations, and entropy values. Here it can be visualized more clearly that, regardless of the general trend, the polymeric materials filled with solid lubricants showed the amount of heat lost in the thermodynamic system through the movement of particles and their ability to move to the surface (migration). (PMMA/Bwax) showed the highest value of particles migrating to the surface.

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DSC-based investigations confirmed the good miscibility and uniform structure of PMMA with solid lubricants. Moreover, the main differences in specific heat capacity, crystallographic temperature, and melting point affect the crystal structure of the polymeric matrix, as all samples (PMMA/PE-wax, PMMA/Bwax, PMMA/CS) showed an increase in specific heat capacity with increasing the filling percentage is attributed to the role played by solid lubricants in migrating to the surface and lubricating the appropriate surface to increase the hydrophobic susceptibility of the polymer, as well as an increase in heat flow upon heating and passing through the glass transition phase, with a decrease in the crystalline temperature and melting point with an increase in the filling percentage. The heat capacity is greater upon heating above the glass transition temperature and when it reaches a certain temperature it gains enough energy to enter into a more organized structure known as "crystals", which release latent heat upon crystallization because it is an exothermic reaction. Table (5) Represented the results of NETZSCH / Proteus 70 software programs as Delta Specific Heat Capacity (J/ g K), Crystalline temperature (Tc), Area (J / g), and melting point (Tm) from Fig.(4) Crystalline temperature (Tc) and the glass transition temperature (Tg) of PMMA\MWCNT are varied with Nano content.

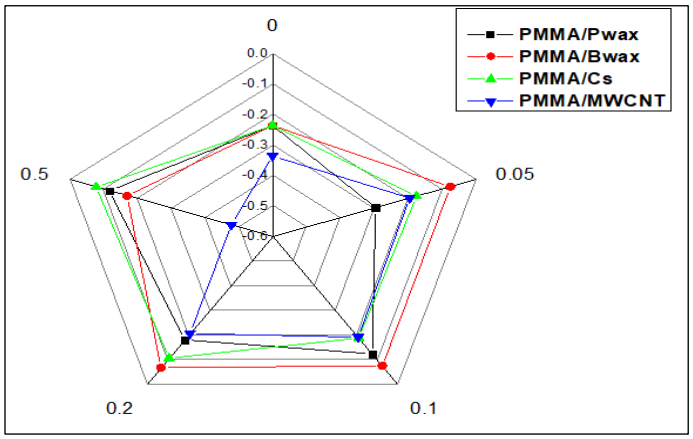

Fig -4: Radar chart of entropy for PMMA with solid lubricant and Filler wt. %.

Table -6: Illustrates DSC analysis results.

Samples	(Cp) (J/ g K˚)	(Tc) (˚C)	Area (J / g)	(Tm) (˚C)
Pure PMMA	0.001	211.3	-6.622	208
PMMA / PE-wax				
0.05 wt.%	0.000	206.4	-10.93	202
0.1 wt.%	0.021	190.4	-4.91	191
0.2 wt.%	0.018	200.6	-4.737	193.25
0.5 wt.%	0.104	175.3	-3.713	179.25
PMMA / Bwax				
0.05 wt.%	0.018	190.7	-3.828	189
0.1 wt.%	0.041	198.1	-3.682	188.75
0.2 wt.%	0.062	176.8	-3.521	188.125
0.5 wt.%	0.072	199.4	-8.054	186.375
PMMA / CS				

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0.05 wt.%	0.043	200.1	-7.344	190
0.1 wt.%	0.088	194.5	-6.187	187.5
0.2 wt.%	0.006	184.0	-5.720	190.875
0.5 wt.%	0.136	186.6	-3.531	187.5

Fig.(5, a) shows PMMA\PE-wax, the conventional DSC results (total heat flow) are shown for pure PMMA polymer and PMMA polymer with solid lubricants. Only one glass transition as received can be observed for pure PMMA polymer an overlapping group of transitions at 200 – 220 ˚C, a glass transition, an overlapping group of transitions at 205 –220˚C, and a crystalline melt. Cold crystallization transitions was observed for all lubricant ratios at 167 °C. Fig. (4, b) shows PMMA\CS, which two transitions were observed for PMMA\CS with lubricant ratio (0.5%), a glass transition at 170 °C, while for the remaining solid lubricant ratios, a glass transition was observed at 167 °C. Fig. (4, c) shows PMMA\ Bwax, the cold crystallization transitions for all ratios observed with a glass transition at 167 °C and a crystal melting point at 215 °C for the 0.1% lubricated polymer.

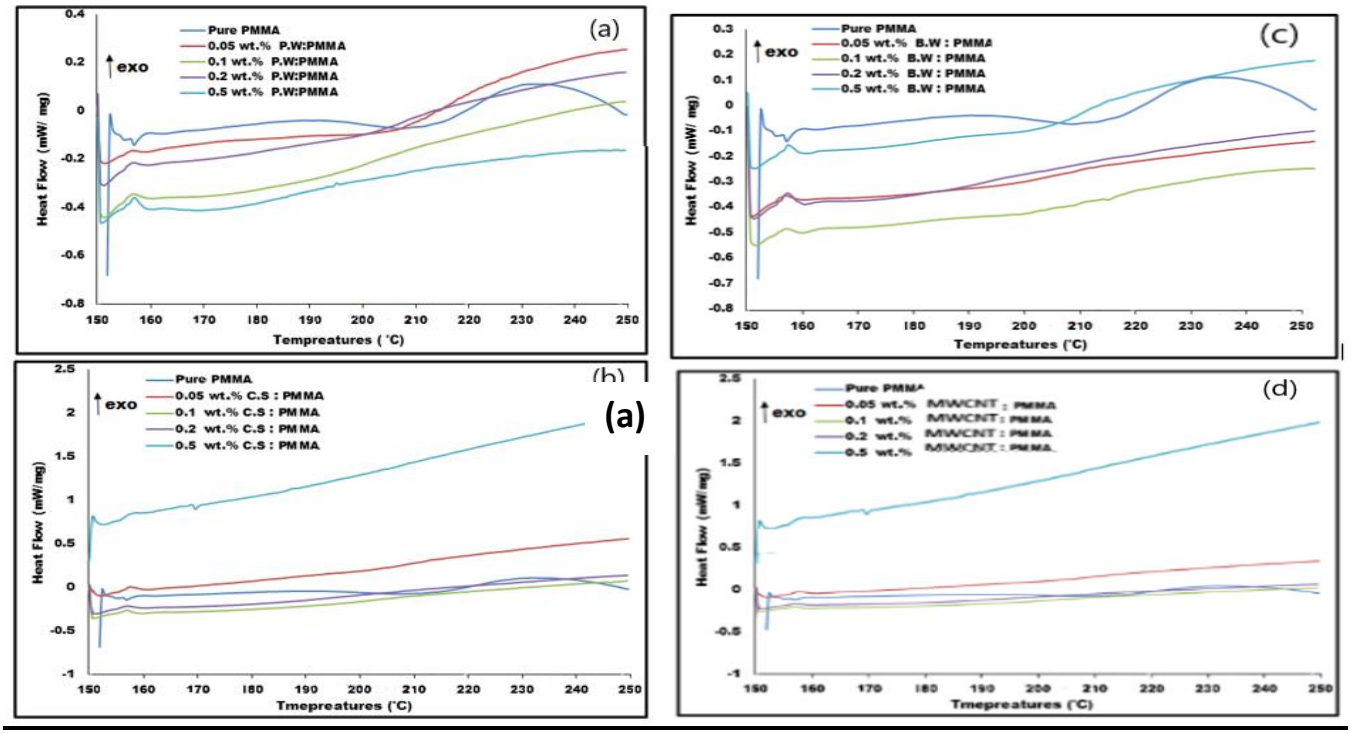

Fig -5: DSC result of Pure PMMA & PMMA/solid lubricant wt. % from 150 to 250˚C.

CONCLUSIONS

In-situ free radical polymerization is a quite acceptable technique for producing pure PMMA, PMMA loaded with solid lubricant, and nanoparticles. FTIR analysis revealed characteristic peaks corresponding to PMMA and PMMA\MWCNT solid lubricants. The incorporation of solid lubricants gave rise to additional peaks indicative of their functional groups, confirming successful integration into the PMMA matrix. For PMMA/MWCNT composites, the characteristic transitions and intensities of the FTIR spectra showed strong interfacial interactions between PMMA and MWCNTs. DSC analysis provided results that were very consistent with the thermal transformations of the compounds. It was observed that the glass transition temperature (Tg) of PMMA varies with the proportion of different fillers. The incorporation of plasticizers (calcium stearate, beeswax, and paraffin wax) resulted in slight modifications to the Tg values, attributed to the plasticizing or reinforcing effects of these additives, which work to move the polymeric chains apart and increase the entropy values accordingly. PMMA/MWCNT composites showed an increase in Tg, indicating enhanced thermal stability due to the solid and thermally conductive nature of MWCNTs. Finally, the addition

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of self-lubricants and fillers significantly influences the structural, electrical, chemical, and thermal properties of PMMA composites, making them excellent candidates for advanced material applications with tailored functions.

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Conflict Of Interest

The author declares that no conflict of interest.

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