Preparation and Characterization of PMMA Magnetic Nanocomposite

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Abstract - This work studies the effect of magnetic nanoparticles iron oxide (Fe3O4) on the rheological, physical, magnetic and thermal behavior of Poly(methyl methacrylate) PMMA magnetic nano solution and PMMA magnetic nano composite. Nanoparticles iron oxide (Fe3O4) where mixed with the PMMA solventm by ultrasonic device to produce MNPs polymer solution and the same MNPsblended with PMMA billets to prepare magnetic polymer composite by using twin screw extruder.Structure properties of Fe3O4 NPs , PMMA magnetic nano solution and PMMA magnetic nano composite were examined by using Atomic force microscopy (AFM). Magnetic properties for these solution and composite were measured using magnetic hysteresis circle. Electrical andthermal properties for MNPs polymer solution and MNPs PMMA composite were measured usingelectrical resistance device and KD2 respectively .In addation surface tension, density and pH for solutions was tested .While rheological properties was measured byCone and plate viscometer device. Additionally thermal behavior for MNPs PMMA composite was study by Deferential Scanning Calarometery (DSC). Whilerheological properties for this composite was tested by the capillary rheometer device. Result of structure test for MNPs PMMA solution and MNPs PMMA composite show that surface roughness fluctuating between the increaseing and decreaseing and the range of nanoparticle distribution increasing and shifting the center of NPs concentration towared the left with the increasing of Fe3O4 NPs.Magnetic properties showed that the hysteresis ring increases with the increasing of Fe3O4 NPs for MNPs polymer solution ,and reach maximum value at 0.5 wt%,then decreases in opposite direction. In the other side the ring hysteresis of MNPs polymer composite decreases with the increasing of Fe3O4 NPs.

Also, the results of thermal and electrical conductivity of the MNPs PMMA solutions increase from(0.25 to 0.39w/m.k) and from (0.1 to 0.53 µS/cm) with the increasing ofMNPs respectively . The density , surface tension and pH value increased with the ratio of MNPs for the solution .On the other hand ,the dynamic viscosity of MNPs PMMA solution increases with the 0.003wt % ,0.005 wt % ratios and decreases with 0.001wt % ratio .All the samples indicate decreasing in viscosity with the increasing of temperature and the shear rate.

The thermal behavior of MNPs PMMA composite showed a good agreement with the previous studies. The rheological test of this composite shows that the viscosity decreases and the shear stress increasing with the increase for all ratios.

keywords:- MNPs,polymer ,nanosolution and composite,structure, flow and thermal behavior,magnetic

Introduction

Duringthe last decade, the development of magnetic nanocomposite materials has been the source of discovery of spectacular new phenomena, with potential applications in the multidimensional fields. Among the broad spectrum of nanoscalematerials being investigatedforvarious environmental and biomedicalapplicationsl,magnetic jnanoparticlesk (MNPs) have gainedsignificantattention due totheir intrinsicj magnetic properties, which makes the successfull as magnetically recoverable catalysts, drug delivery agents, anticancer materials, magnetic resonance imaging devices, etc. [1].

nanofluids may be prepared either by one-stepp or two-stepo methodk. Inl the onel-stepl methodj nanoparticlesd aree synthesized in base fluid mainly by means of chemical methods or laser ablasion technique. In the case of two-step method nanoparticles are firsty prepared in a form ofpowders by physicalk orr chemicall methodsf, e.gp. grindingl, , ksol-gell processingk, etcc. andn thenn suspendedd inn basee fluidk[2, 18].

Aferrofluidj isaj stable colloidal suspensionofl sub-domain magnetic particles in a liquid carrier.The particles, which have an averagesizeof about 1008A °, are coated with a stabiliising dispersing agentwhichj prevents particle agglomeration even when a strongmagneticc field gradient isj applied to theferrofluid [3].

Magnetic polymer-basedh spheresj haveu been consideredd ass anm importantk materiall forjuthe biotechnologyo industryy, suchh asj, forr instancej, inkcelll separationand DNAextractionn . Inm particularr, mesoporoush polymerick templatesj couldj bee producedd ass micronsizedg beadsftthat allowk inn situy

chemicalj synthesisf of nanosized ferritee articlesu withj adjustable magneticm propertiesi andd masss densityy [4].

Polymethyll methacrylate(PMMAl) has beenthe primarychoicer for thepreparation of polymericc nanocompositesf duer tou its superior propertiessuchas high strength, compatibility with ceramics, dimensionalstability and opticaljclarity [5].

Ironoxidesf areone ofj thee mostimportant transition metal oxidesof technologicall importancek. Sixteen puree phasesu ofg ironn oxidesk, i.ek., oxides, hydroxidess ory oxy-hydroxidesare known too datee.Theseare $Fe(OH)_3$p, $Fe(OH)_2$9, $Fe_5HO_8.4H_2O$k, $Fe_3O_4$8, FeO,five polymorphs of FeOOHand four of Fe_2O_3o. Characteristics of these oxide compoundsincluded low solubility andk brilliant colorsj. All they iron oxides are crystalline exceptl Schwertmannitel andk ferrihydritel which are poorly crystallinel. These oxide can besynthesizedl by alll known wetlchemicalg. Some ofthe synthesis techniques include chemicalmprecipitation, sol-ge, hydrothermal, surfactant mediated-precipitation, and micro wave assisted hydrothermal technique.. [6].

Capillaryu rheometry is extensively used inboth industryand academiato assess them rheologicall behavior of polymer meltsathigh shear rates before testing their processability in full industriall scale . First, capillary flow involves flow through a contraction ofa certain angle or Bagle correction This pressure is required in order to calculate the trueshear stress, and also, frequently, the apparentextensional rheology of molten polymers, am method well practiced in industry therefore, itis important to understand the origin of this excess pressure and consequently to be able to predict it [7, 13].

Polycarbonate(PC), and PP, etc. Eachpolymer could present differentl critical pressure coefficientl toshearviscosityj,and then shows different geometrica dependences ofshear viscosity.The pressure sensitivity is mainly duel to the decrease of the action of distancebetween moleculesl. For the capillary flow of polymer melts in practicalprocessing, theshear ratecould be high companied by a high pressure, especiallyin micro-extrusion and micro-injection [8, 14, 17].

The magnetization measurements provide strong evidence of surfaces effects to magnetization, which explains the non-saturations of magnetizations at high fields.There magnetizationd versuse applied field behaviors ofas thethree ferrite systemsas showa similar jumps in the initial part of the magnetization curve in all the cases which implies the existence off a core-shell likes morphology of the particles over larges temperaturea ranged and its dominance overs thes interparticled interaction effects between the particles.[9].

the Fe3O4/PMMA composite particles were fabricated by a simple one-pot hydrothermal method. The magnetic measurement showed that the composite particles displayed a higher saturated magnetization and superparamagnetic property. The rheological properties of magneto rheological fluids (MRFs) based on Fe3O4/PMMA particles were measured on a rotational rheometer with a magnetic field enerator[10].

the ferrofluid consist of superfine particle having magnetic characteristics whose magnetic properties changes upon changing their temperature up to Curie temperature underh theh influencek of externall magnetich fieldr. The nanoparticles are coated with surfactant so as to prevent sedimentation and agglomeration of magnetic particles[11, 15].

The aim of this work is to prepare and investigate the different properties of Fe3O4 as magnetic nanoparticles (MNPs) dispersed in polymer solution and mixed in polymer melt .Study the structural ,magnetic, flow, thermal, electrical and physical behavior due to the Fe3O4 addition.Also to show the relation between these properties and state the method of controlling on the final product for different application.

Materials and method:

The materials used in this work are a PMMA as biopolymer provided from china.Iron Oxide (Fe3O4) Nanoparticlesis supplied as a powder from (Sigma-Aldrich power Technology Co., Ltd, China). The density, surface tension, dynamic viscosity, kinematic viscosity, electrical properties and thermal properties, of the PMMA solution and composite with and without NPs are tested .The properties of PMMA and Iron Oxide (Fe3O4) are shown in the tables (1and 2).

Table (1): The properties of PMMA[5]

Propriety	**Data**
Color	White
Melt Point	130 Cº
Glass Temperture	100-105 ºC

Table (2): The properties of Fe3O4

Propriety	**Data**
Color	Black
Molecular weight (g/mol)	536.39(g/mol)
Radius	20 Nanometer
Shape	spherical

Preparation of MNPs polymers solution:

The method of the preparation of the MNPs polymers solution includes the following steps :

1- Using 8g ofPMMAtoprepare solutionUsingsolvent.
2- Mixing of (0.001 ,0.003 and 0.005 wt%) of MNPs (Fe3O4) with each of(8 g) of PMMA solution in glove box consist of balance and vacume conditional to mix the PMMAsolution with theFe3O4NPs.
3- Improve the dispersion of the resulting mixture by using the ultrasonic device at the room temperature for 60 min. at 50 ^{0}C and 440w.

Preparation of MNPs PMMA Composite:-

The method of the preparation of the MNPs polymers solution includes the following steps:

1- Weighing (0.001, 0.003, 0.005wt. %) of Fe3O4 nanoparticles in vacuum glove box and mixing with acetone solvent by ultrasonic device at 440w for 30min and 50ºC.
2- The PMMA pellets and Fe3O4 MNPs acetone are mixed together at room temperature
3- The resulting mixture is melted in twin screw extruder at 180°C and the screw speed is (10 and 15 rpm) to prepare MNPs polymer composite sheet at thickness of 2mm.
4- The resulting sheet is ready for different testsusing.

Characterizations

1.Magnetic Properties:

The magnetic hysteresis loops of MNPs PMMA solution can be measured by magnetic hysteresis loopsconsist of:-

Digital gauss meter to measure magnetic flux density (B).

1- Electrical magnetic part consits of coil (n=1000,max current=1.25A)
2- Ammeter to measure electrical current
3- Power supply (0-20V).

The samples of MNPs polymer solution and MNPs PMMA composite was placed inside the coil and measure the flux density and current[10]

2. Density:

The measuring of density was performed at room temperature by using (matsu haku high precision density tester gp-12os), and the all experiment tested according to Archimedes law.

3. Surface tension :

Surface tension of the samples is measured by using JZYW-2008 automatic interface Tensiometer supply by being united test co., ltd.

4. Rheology Test:

4.1 Dynamic viscosity (cone-plate viscometer) :

The dynamic viscosity of the samples are examined using cone – plate viscometer (DV- III ultra programmable rheometer) as shown in the Fig.2with the cone diameter of 4.8 cm and cone angle of 3^0 degree.

4.2Capillary Rheometer:

The rheological measurements are performed for MNPs PMMA composite capillary rheometer model SR20 (single bore 20KN). Capillary diameter 1mm , capillary length 20mm and barrel diameter 15mm .

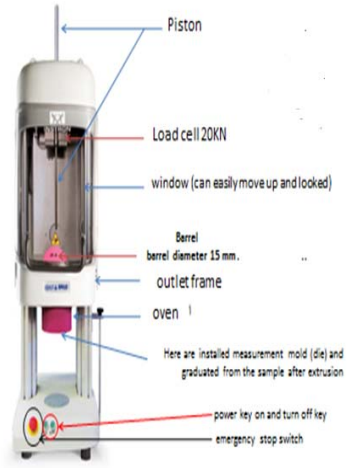

Fig.1.Capillary Rheometer Device[8]

5.Thermal conductivity :-

There are many method to find out the thermal conductivity of nanofluid and the transient hot-wire method .KD2 is the best suited method for that. The thermal conductivity is measured by using a KD2 Pro thermal properties analyzer (Decagon Devices, Inc., USA).

6.Optical measurement (UV-Visible Spectrophotometer):-

Absorbance spectra of MNPs PMMA solution and were measured by UV-Vis double beam spectrophotometers, (shimadzu, UV-1800, Japan).

7.Morphology:-

The morphological studies of MNPs PMMA solution and MNPs PMMA composite are conducted by tapping mode AFM (AA3000) in the Ministry of Science and Technology.

8. Electrical conductivity test:-

The measuring of electrical conductivity for MNPs PMMA composite performed by resistance measurement device (Applent EasiOhm ATS12 precision Ohmmeter).The MNPs PMMA solutionis measured using electrical conductivity for solution (inolab code 720).

9.Deferenttial Scanning Calarometery (DSC) test:-

This test is used to measure to the thermo history of MNPs PMMA composite such as (Tm), (Tg) and Tc using (DSC-60,shimadzu) device

Results and Discussion.

1. Hysterieses loop

Fig,2 show the hesterises loop of MNPs PMMA solutions .The realationship between the flux density (B) and current (I) is linaer . The curve starts from the point of origin , with I=0,B=0. The B increases to reach the saturation then go down gradually, but it's not return to the same track, and taking several paths. During this session the full heat generated inside the material arises from the friction of atoms.

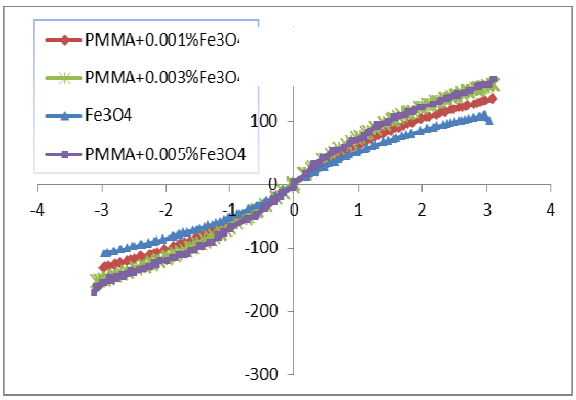

Fig.2 : Heysterises loop of MNPs PMMA solution without and with different concentration of MNPs

2.Density

Generally, Fe3O4 NPs increases the density of MNPs PMMA solution. The polymer density increases with the addition of Fe3O4 NPs concentration due to exchange reaction ensure the chemical bonding of the methacylate units to the surface iron atoms which in turn , undergo the wanted polymerization with vinyl groups of the PMMA to produce procerdureis expected to lead grafting of nanoparticles to the polymer chains shown in Fig 3[1 and 16]..

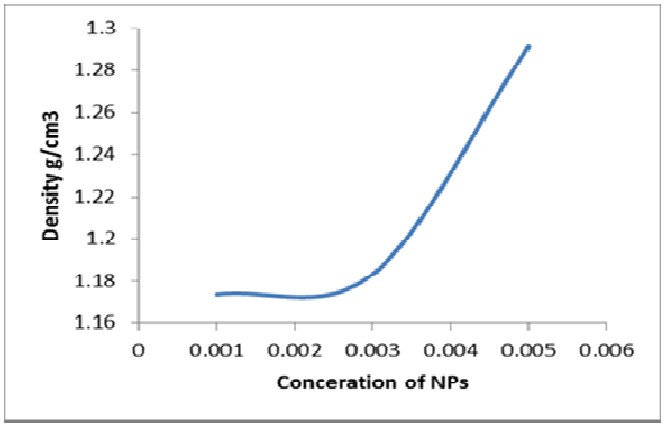

Fig.3 Density behavior of MNPs PMMA solution with the NPs concentration

3.Surface Tension

The surface tension of polymers increases with the addition of Fe3O4 NPs concentration due the Van der Waals forces between the nano particles increases the surface free energy and the attraction between surface molecules . Fig.4 shows the increasing of surface tension of MNPs PMMA solution.

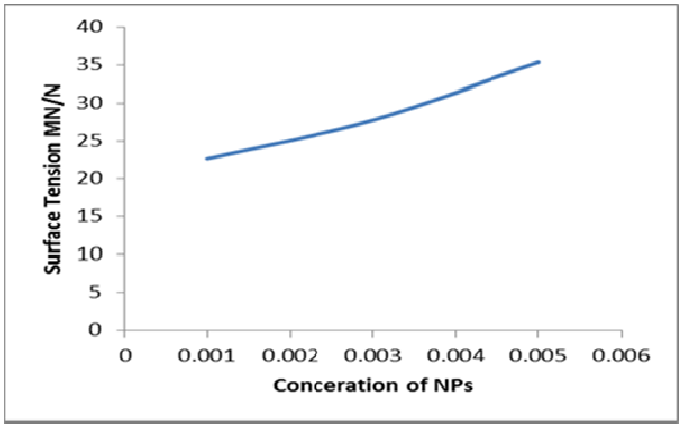

Fig.4: surface tension behavior of MNPs PMMA solution

4. Rheology Test

4.1 Dynamic viscosity (cone-plate viscometer)

4.1.1 Temperature Effect

Fig.5 shows that the dynamic viscosity behavior of PMMA solution with and without MNPs solution at different temperatures. The viscosity decreases for all solutions with the temperature increasing . The PMMA solution decreases from 3.3 cp to 2.8 cp with temperature rang (25-45)Cº .The MNPs PMMA solutions gradually and slowly decrease with the temperature increasing .The resistance to the temperature and the stability of viscosity of MNPs PMMA solutions increase with the MNPs concentration increasing. the 0.005 ratio indicates higher viscosity resistance.

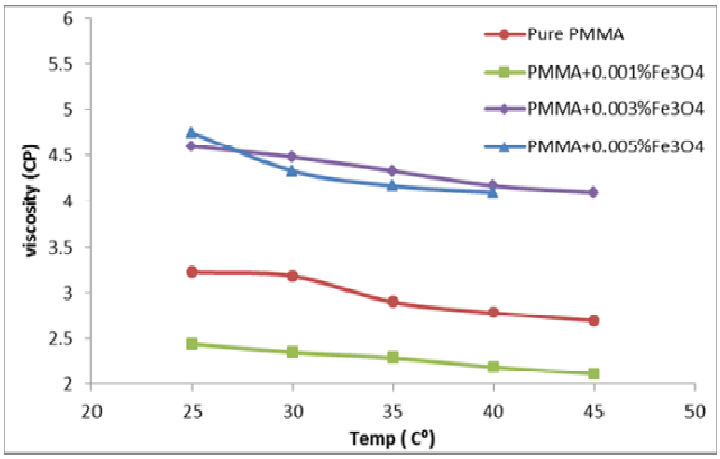

Fig.5: Dynamic viscosity behavior of MNPs PMMA solution without and with different MNPs concentration and with temperatures increasing

4.1.2 Shear Rate Effect

Fig.6shows that the dynamic viscosity behavior of MNPs PMMA solution without and with different MNPs concentration as a function of shear rate .PMMA solution shows Newtonian behavior where the viscosity keeps constant with the shear rate increasing . The other MNPs PMMA solutions indicate shear thickening,where the viscosity increasing with the shear rate increasing,the shear rate range shifted to the high magnitude with the MNPs 0.001concentration ,while the 0.003and 0.005 at low shear rate range.

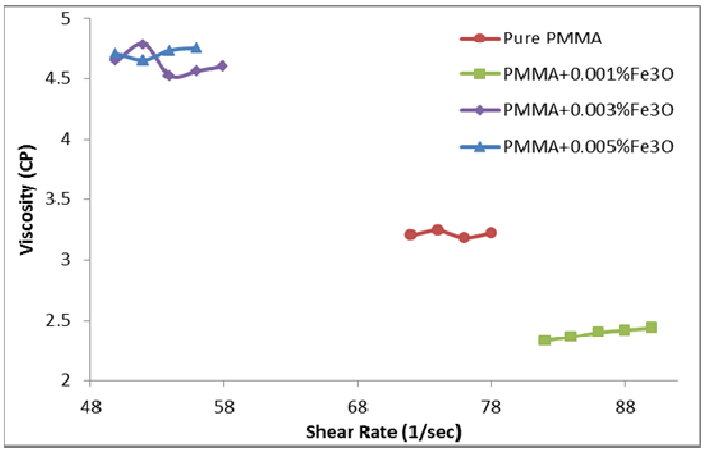

Fig.6: Dynamic viscosity behavior of MNPs PMMA solution without and with different MNPs concentration as a function of shear rate.

4.2 Capillary rheometer

4.2. 1Shear rate effects

Fig.7,8,9,10,11,12,13and 14,show the behavior of viscosity of MNPs PMMA composite as a function of shear rate. The viscosity decreases for all composite with the shear rate increasing . The PMMA composite decreases rabidly from 1589 cp to 1364 cp with the shear rate increases from (100-1000)1/sec. The results show the viscosity decreases for MNPs PMMA melt and shear thinning was dominated for all ratios. The PMMA solution decreases with the shear rate increases . Entanglements of the PMMA chains result in resistance to flow at low shear rates. At higher shear rates, the molecules are more aligned, there is less chain entanglement, and therefore the viscosity decreases.

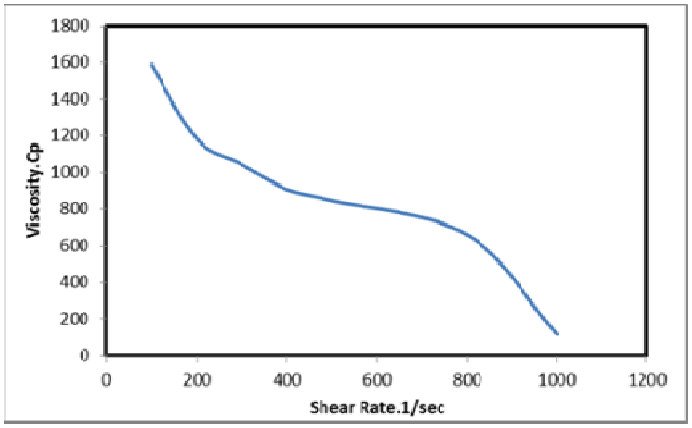

Fig.7: viscosity behavior of 0.001% MNPs PMMA composite melt as a function of shear rate.

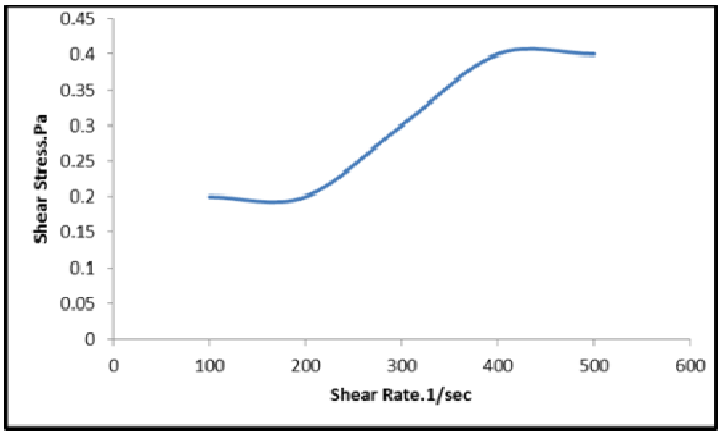

Fig.8 : viscosity and shear stress behavior of 0.001% MNPs PMMA composite melt as a function of shear stress

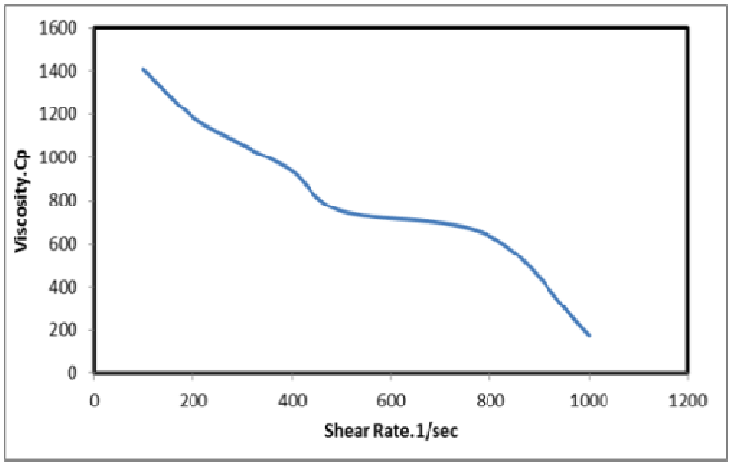

Fig.9 : viscosity behavior of 0.003% MNPs PMMA composite melt as a function of shear rate .

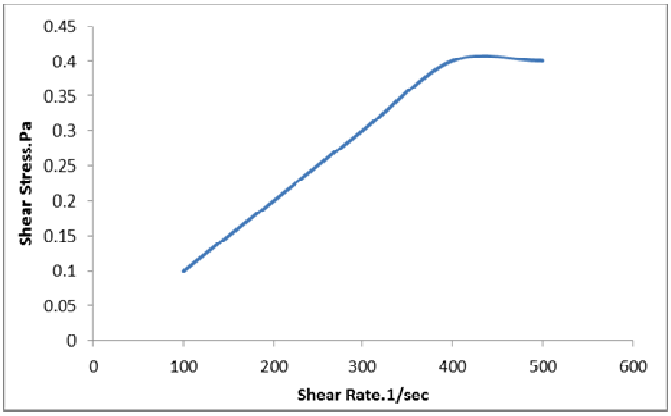

Fig.10: shear rate behavior of MNPs PMMA+0.003%Fe3O4 composite as a function of shear stress

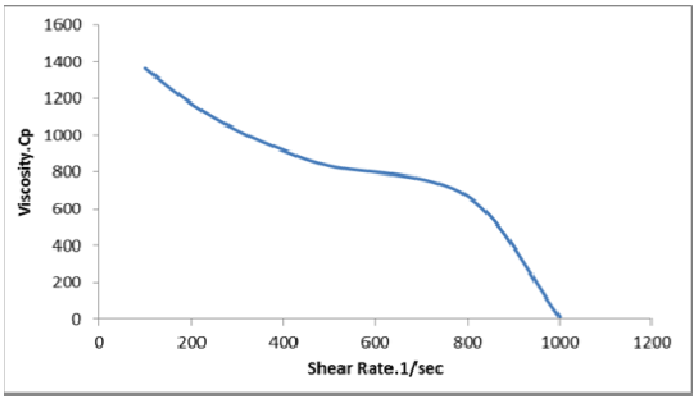

Fig.11 : viscosity behavior of MNPs PMMA+0.001%Fe_3O_4 composite as a function of shear rate.

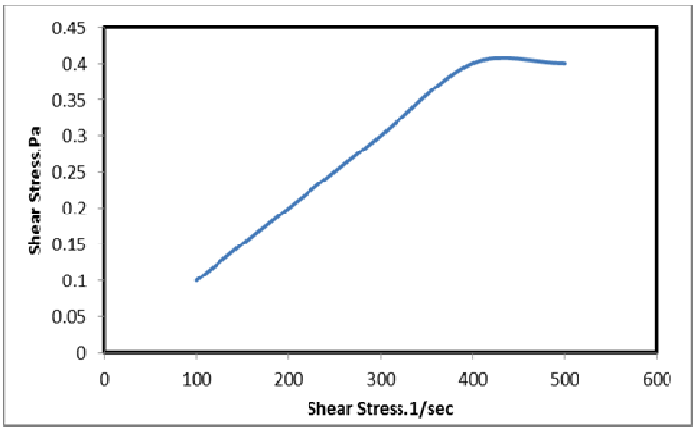

Fig.12: shear stress behavior of MNPs PMMA+0.005%Fe3O4 composite melt as a function of shear rate.

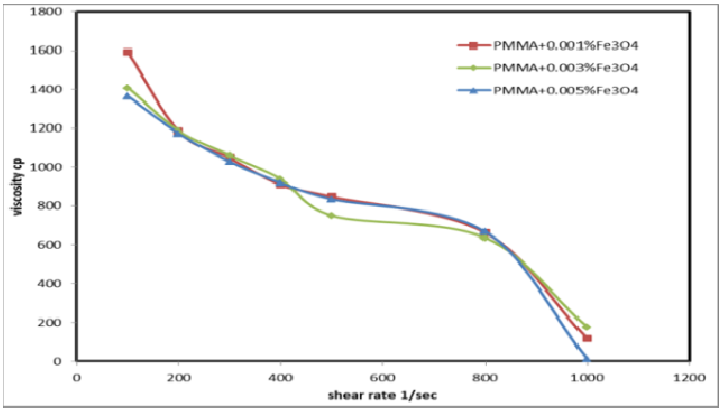

Fig.13: viscosity behavior of MNPs PMMA composite at different concentration as a function of shear rate

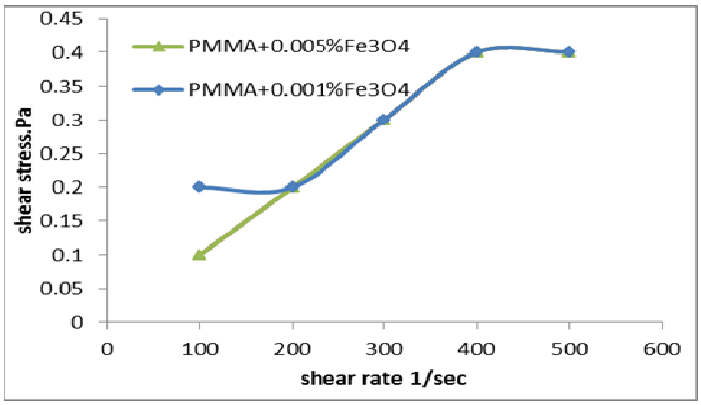

Fig.14: shear stress behavior of MNPs PMMA composite at different concentration as a function of shear rate

5. Thermal conductivity

Fig.15, shows the thermal conductivity increases of PMMA solution with the MNPs concentration and temperature increasing ,due to the high thermal conductivity of this NPs.Also thermal conductivity of 0.005% is higher than other ratios due to the PMMA structure and high dispersion of MNPs.

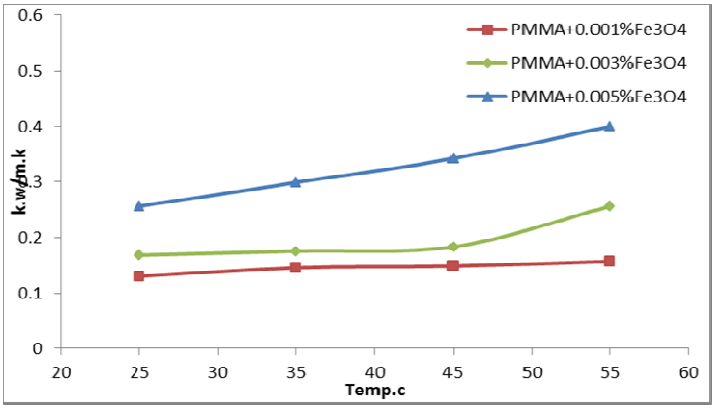

Fig.15 : Thermal conductivity behavior of MNPs PMMA solution with different temperture.

5.Optical measurement (UV-Visible Spectrophotometer)

Fig. 16 shows the UV- m Vis spectra of PMMA solution with and without MNPs .The measuring of a sensible increase in the absorption intensity, accompanied by a slight change in band width and maximum wavelength, when increasing the concentration of Fe3O4 NPs its band width are affected by the size, shape, and composition of the colloidal NPs. Absorption peaks are shifted towards higher wavelength side with the increasing of Fe3O4. Polymer chains of PMMA wrapped around the Fe3O4 particles, which keeps MNPs stable and prevent the agglomertion.

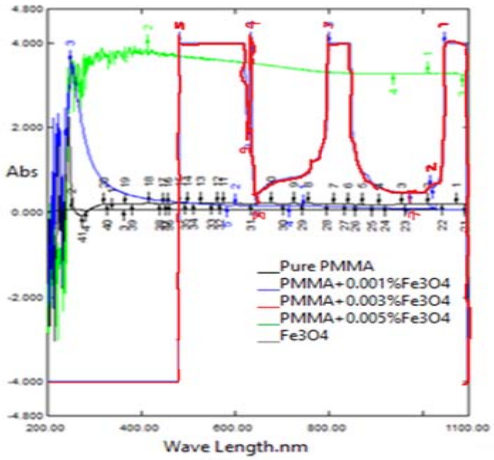

Fig.16: UV- m Vis spectrophotometer of PMMA solution with and withoutdifferent conentration of MNPs

7. Morphology results (AFM):-

7.1 Morphology results of MNPs PMMA solution:-

Fig. 17,18,19 and 20: show the morphography of the PMMA solution with and without different MNPs concentration.. The roughness and the average particle size distribution increasing with the MNPs concentration increasing, due to the different size and shape of NPs dispersed in PMMA solution .The shifting of center of particle size range to the left means that the magnetic particles in the nano scale. The agglomeration of MNPs in 0.001% is more than other ratio and 2D image and particle size distribution of this ratio supporting that, this result agreement with M.A.Ramazanov[12]

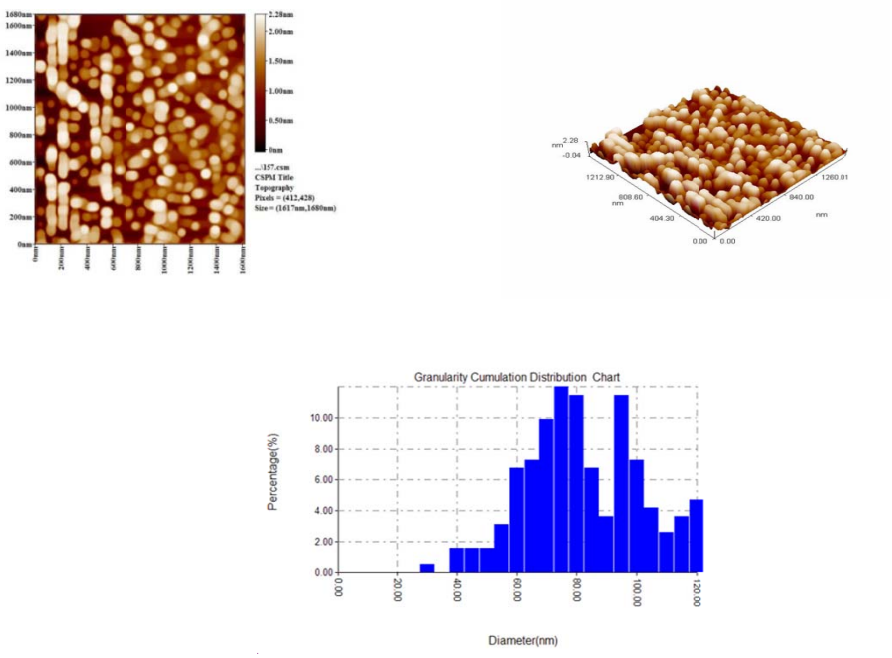

Fig. 17: Topography of the PMMA solution (A) 2-D (B) 3-D (C) Particle size distribution

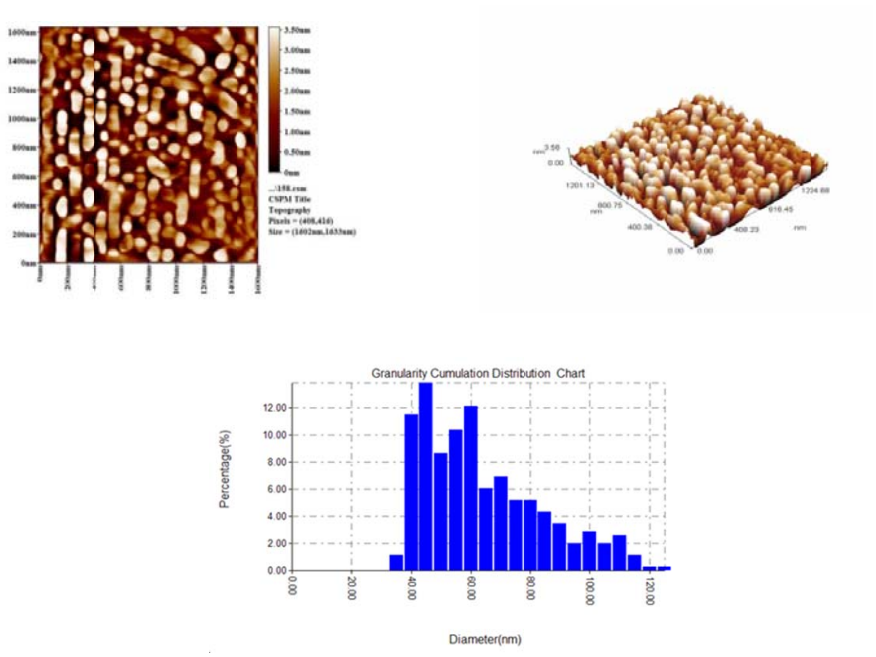

Fig. 18: Topography of the PMMA+0.001%Fe3O4 solution (A) 2-D particles size (B) 3-D particle size (C) Particle size distribution

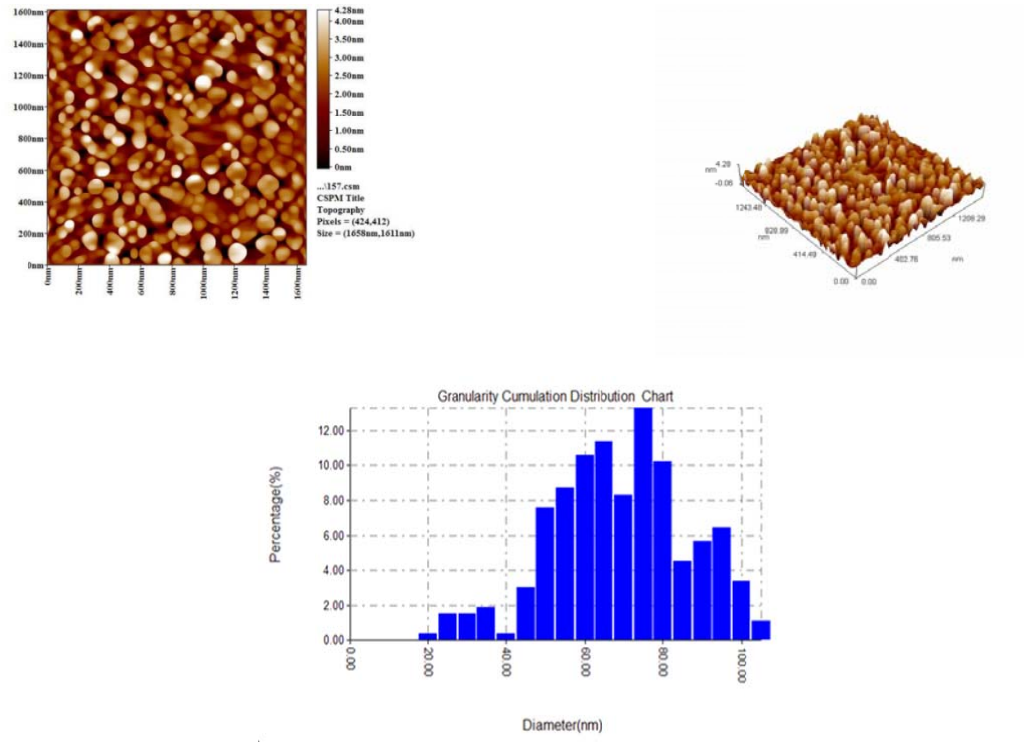

Fig. 19: Topography of the PMMA+0.003%Fe3O4 solution (A) 2-D particles size (B) 3-D particle size (C) Particle size distribution

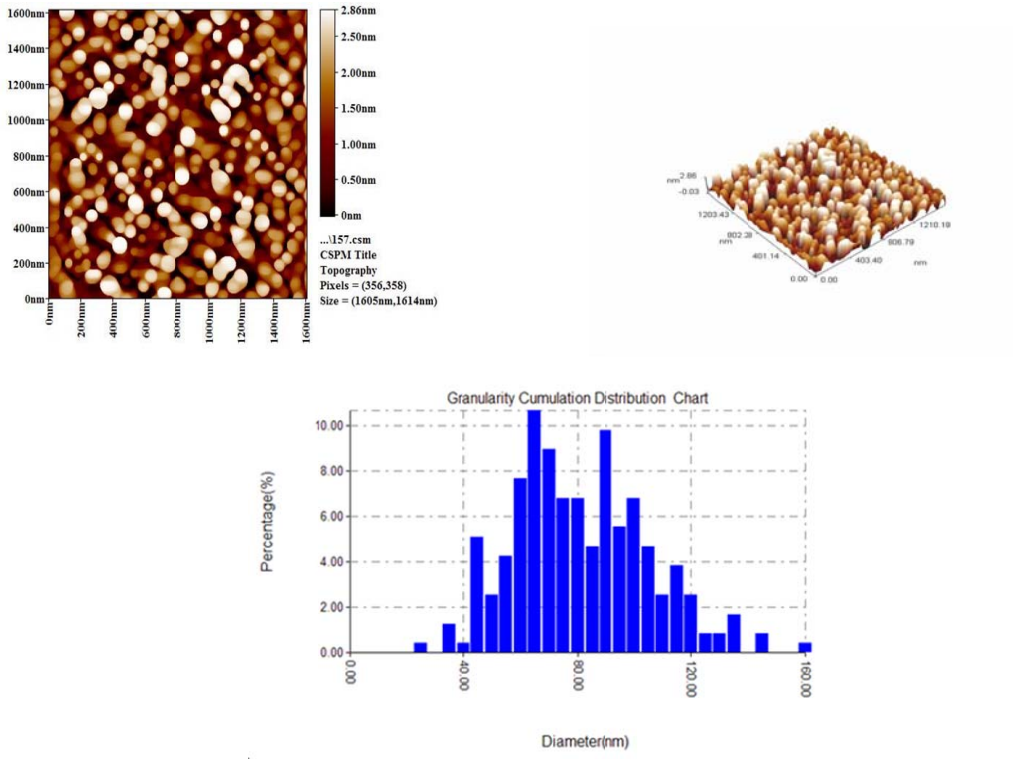

Fig. 20: Topography of the PMMA+0.005%Fe3O4 solution (A) 2-D (B) 3-D (C) Particle size distribution

7.2 Morphology results MNPs PMMA composite:-

Fig. 21,22,23 and 24,: show the morphography in 2-D and 3-D of MNPs PMMA solid. It can be seen that the nanoparticles have approximately spherical shape which show the accuracy of ultrasonic device to produce good dispersion and reduce the agglomeration of nanoparticles. The particles size distribution as shown in Fig.4.60.C can produce accurate information about the particles size, the average particles size obtained is from PMMA+0.005%Fe3O4 (20-120 nm) which confirmed that the size of the Fe3O4in the nano-scale range., this result agreement with M.A.Ramazanov[12]

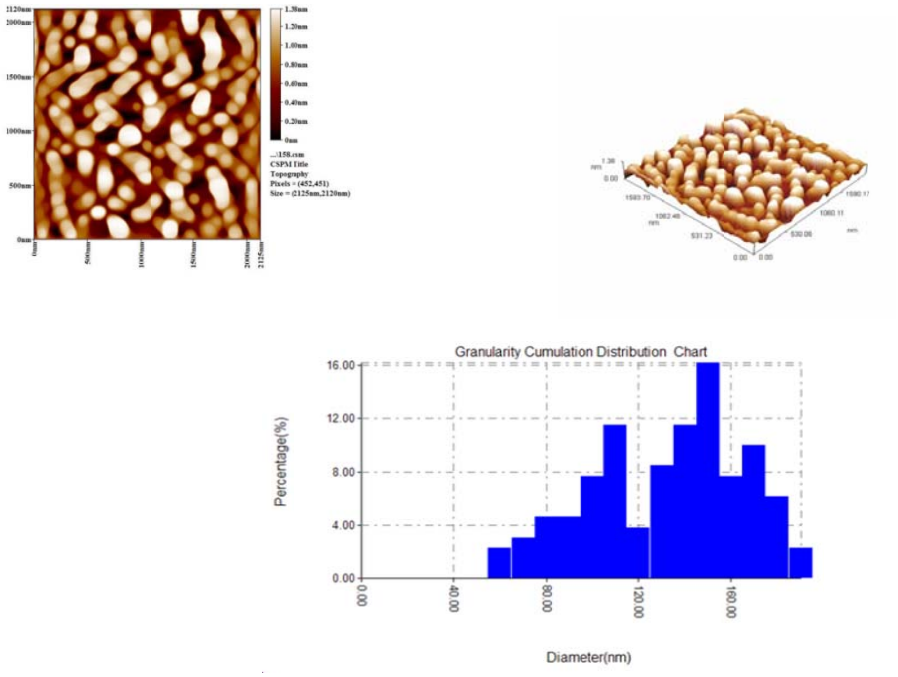

Fig. 21: Topography of the PMMA solid (A) 2-D particles size (B) 3-D particle size (C) Particle size distribution.

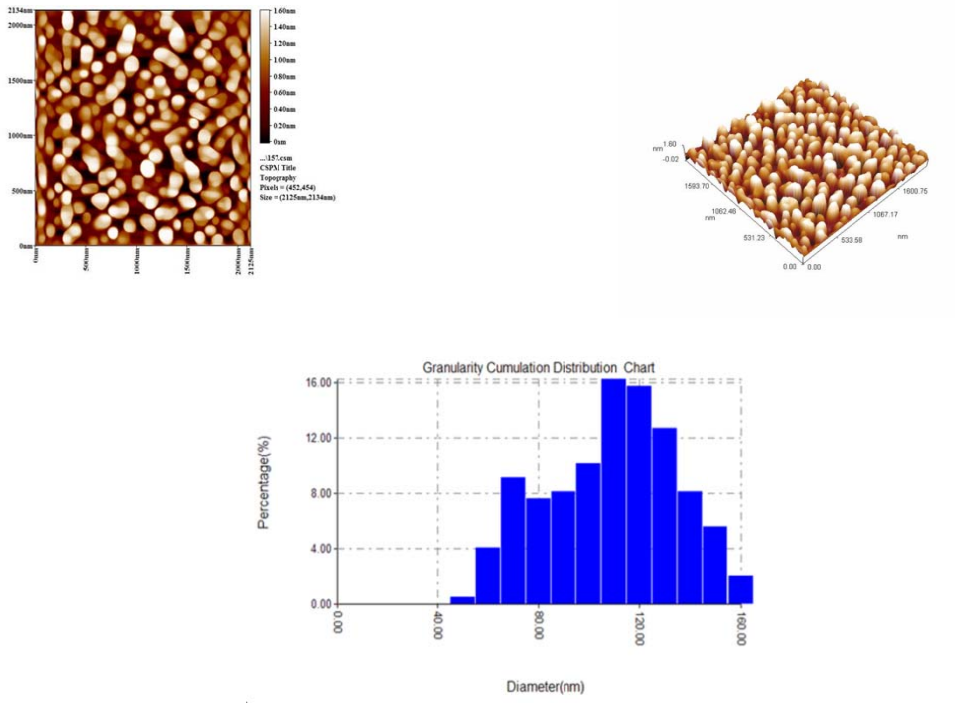

Fig. 22: Topography of the PMMA+0.001%Fe3O4 soild (A) 2-D particles size (B) 3-D particle size (C) Particle size distribution

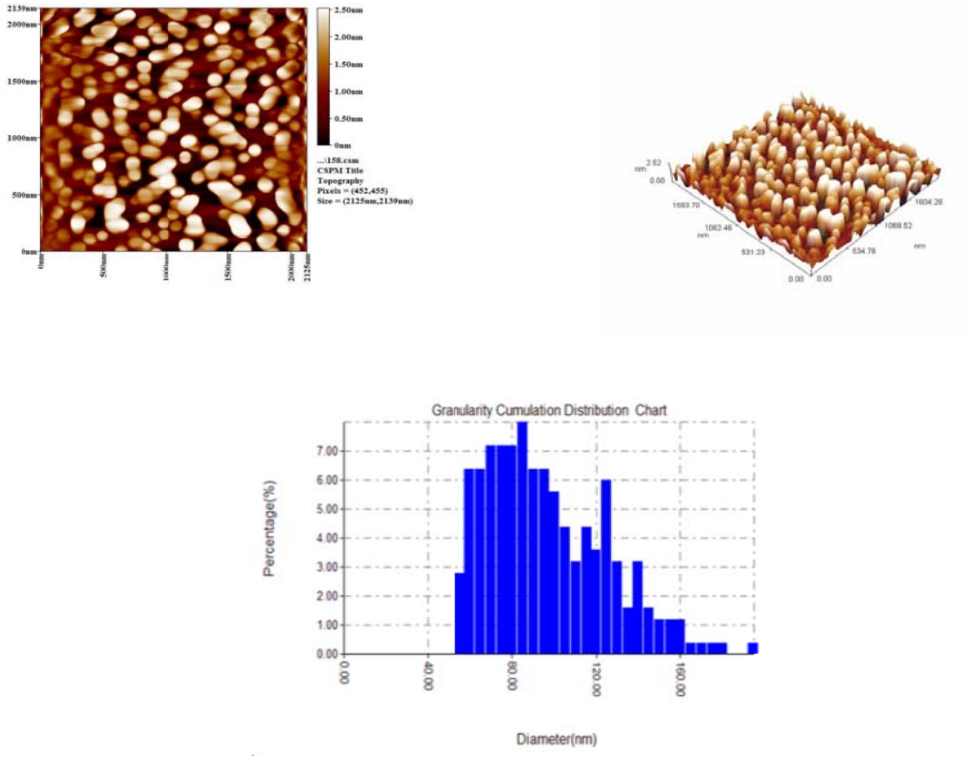

Fig. 23: Topography of the PMMA+0.003%Fe3O4 soild (A) 2-D particles size (B) 3-D particle size (C) Particle size distribution

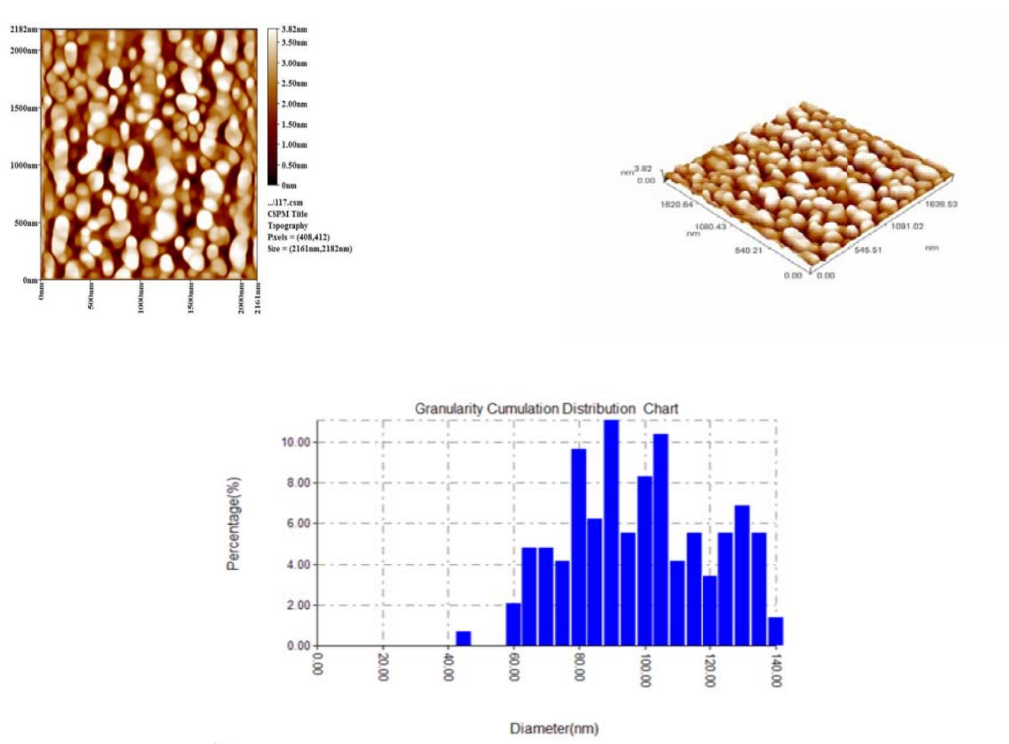

Fig. 24: Topography of the PMMA+0.005%Fe3O4 solution (A) 2-D particles size (B) 3-D particle size (C) Particle size distribution

8. Deferenttial Scanning Calarometery (DSC) test

This test is used to measure the thermophysical properties including: (Tm)and , (Tg).The Tg and Tm increasing with the addition of Fe3O4 NPS shown in table 1,becacuse Fe3O4 is polar oxide which interact with polar molecules PMMA through diple-diple interaction as secondary bond which increasing the crystallization ,and Tg and Tm.As the critical temperature is approached absorbed heat is increases by te Fe3O4 PMMA polymer in

order to annihilate the neigh boring dipole moment parallelism.At crtical temperture this behavior is stopped and the heat capacity curve indicates an infinite slope ,implying a latent heat of transformation .

The endothermic trend approaching the curie temp is from randomization of the magnetic dipoles .Then afast exothermic shift is indicates, when no more heat absorbed by the sample as shown in Fig. (25).

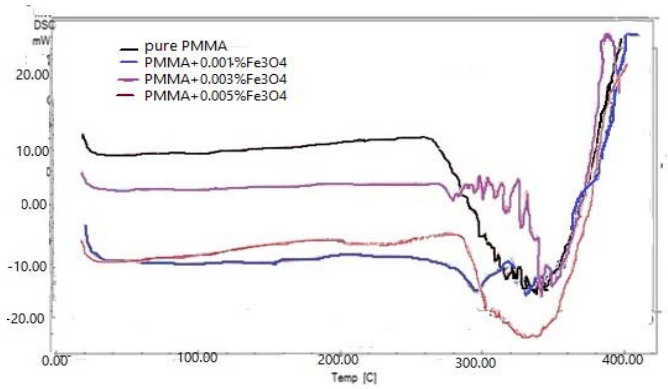

Fig.25 : Thermal histories of MNPs/PMMA composite

Conclusions:

1- Magnetic nanoparticles coating with organics species includes polymers can stabilize the magnetic nanoparticles and avoid aggregation.
2- Strong relation occurred between morphological, rheological, and optical properties of MNPs PMMA solution and MNPs PMMA composite.
3- The using of ultrasonic and twin screw extruder device produce a better distribution of nanoparticles in polymer matrix and less agglomeration. The uniformity of nanoparticles distribution strongly effect on rheological, physical and mechanical properties of melt nanocomposite.
4- The optimum ratio of the MNPs which produces the best improvement in properties of MNPs PMMA solution and MNPs PMMA composite found at 0.5% wt.
5- There is a relationship between dispersion of nanoparticles, the crystallinity and rheological properties for the PMMA solution. The higher levels of nanoparticles dispersion and less agglomeration giving higher magnetic ,thermal,electrical properties.
6- Using of capillary rheometer is very usefull to study the rheological behavior of PMMA polymer.
7- MNPs PMMA composite exhibited high shear-thining during flow in capillary die compared with the pure PMMA.

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