Electrical Insulation Breakdown Strength and Thermal Conductivity of Different Blended Nanocomposites of New Epoxy Resins

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Abstract

This research studies the development and synthesis of blended nanocomposites filled with Titanium dioxide (TiO_2). Blended nanocomposites based on unsaturated polyester resin (UPR) and epoxy resins were synthesized by reactive blending. The optimum quantity from nano partical of titanium dioxide was selected and different weight proportions 1%, 3%, 5%, and 7% ratios of new epoxy are blended with UPR resin. The dielectric breakdown strength and thermal conductivity properties of the blended nanocomposites were compared with those of the basis material (UPR and 3% TiO_2).The results show good compatibility epoxy resins with the UPR resin on blending, dielectric breakdown strength values are higher while thermal conductivity values of blends nanocomposites are significantly lower compared to that of the(UPR and 3% TiO_2), semi-interpenetrating UPR/Epoxy blends (semi-IPNs) for one type of new epoxy [P_2]was prepared and noticed the blend nanocomposites show higher dielectric breakdown strength than the semi- IPNs (UPR/Epoxy) at low loading of new epoxies but the thermal conductivity is a higher than the semi- IPNs UPR/Epoxy at all loading. Thermogravimetric analysis (TGA) was employed to study the thermal properties of the blended nanocomposites.

Key word: blened nanocomposite: unsaturated polyester resin: epoxy resin: dielectric breakdown strength.

Introduction

With the rapid development of the electronic information industry, better properties are required for substrate and packaging materials such as low coefficient of thermal expansion (CTE), low dielectric constant, high electric insulation, and high thermal stability. Therefore, many studies have been focused on developing new materials with superior strength and attributes[1]..

In the nano studies with notable researches advances that revealed new physical phenomena and improved material properties in the nano-sized since ,the introduction of nanofillers demonstrate several advantages in their properties when compared with the similar properties obtained for epoxy systems with micrometer sized fillers [2-5] polymer nanocomposites have attracted considerable attention from both fundamental research and application points of view, because it exhibited much better mechanical, thermal, and multifunctional properties in comparison with the polymer matrices and their conventional microcomposite and macrocomposite counterparts.[1].

Epoxy resins are well-known materials in the field of insulation systems for heavy electric equipment such as mold-type transformers, potential transformers (PT), current transformers (CT), and metering out-fit (MOF) because the epoxy resin provides essential electrical, thermal, and mechanical properties. In the last two decades, rapid development of epoxy-based composites in electric applications has been achieved to give the epoxy resins much higher electrical performance with lower cost [6,7]. Unsaturated polyester resins (UPR) are comparatively hard and brittle materials and one of the important thermosetting materials used for the manufacture of glass-reinforced plastics and polymeric composites. These resins have wide spread use which is due to their relatively low cost, comfortable of processing, high wetting properties with reinforcements[8] .There are many reports that the technique of polymer blending is a useful method to obtain a new insulating material[9].

For the design of these insulating materials the most important parameter is dielectric voltage breakdown strength. This is used to measure the failure strength of the insulation against the applied electric field. It is to be noted that, when electrical breakdown occurs due to high applied voltage it results in a fracture due to breaking of insulation barrier as well generation of electrical sparks. Li et al[10] observed that hybrid nano/micro Al_2O_3 epoxy composite exhibits higher partial discharge resistance as compared to micro Al_2O_3/epoxy composite. In addition,epoxy/nano-TiO_2 composite show improved alternating current AC voltage endurance, and dielectric strengths compared to its micro size TiO_2 filled epoxy composite[11]

With respect to the electrical breakdown characteristics in polymer nanocomposites, several reports are available on different polymeric systems. In epoxy nanocomposites filled with TiO_2 fillers, the voltage endurance (AC), short-term direct current and alternating (DC , AC) dielectric strengths and impulse breakdown strengths are found to be higher as compared to microcomposites [5]

Thermal conductivity of polymers is an important thermal property for both polymer applications and processing. Polymers typically have intrinsic thermal conductivity much lower than those for metals or ceramic materials, and therefore are good thermal insulators. In other applications which require higher thermal conductivity, such as in electronic packaging and encapsulations, satellite devices, and in areas where good heat dissipation, low thermal expansion and light weight are needed, polymers reinforced with fillers, organic or inorganic, are becoming more and more common in producing advanced polymer composites for these applications[12]. In a solid material, there are two main mechanisms of heat conduction: through elastic waves, which are called phonons, and through free electrons. Polymer materials generally do not have freely moving electrons. So, heat transfer has effected only via elastic waves. The electron conductivity is dominant in metals and absent in insulators [13].

Thermal conductivities of polymer materials depend on many factors, such as chemical constituents, strength of bonding, structure type, molecular weight of side groups, molecular density distribution, type and strength of defects or structural faults, size of intermediate range order, processing conditions and temperature, etc [14]. Commonly used polymer materials are materials of low thermal conductivity. The typical range of polymers is from 0.17 to 0.35 W/mK [15]. Epoxy resin is one of the common materials that is used for electrical insulation In spite of the excellent performance of epoxy, its relatively low thermal conductivity cannot satisfy the increased demands of new electronic devices. Improved thermal conductivity in polymers may be achieved either by changing the molecular orientation, by the addition of conductive fillers [16] or by synthesizing the polymer materials with enhanced thermal conductivity [17].

In this study, an epoxy/ UPR /TiO_2 composites were prepared, with particle sizes of 10-15nm; an electrical insulation breakdown test and thermal conductivity were then carried out.

2. Experimental

Materials

All the raw materials were supplied from Merck,GCC and Aldrich Chemicals Co. and used as received.

Testing

The FTIR spectrum of UPR/TiO_2 and blended nanocomposites were taken using bromide discs on a Shimadzo(Ir prestige-21) FTIRspectrophotometer .^{1}HNMR spectra were carried out by company:Bruker,model:ultra-shield 300MHz origin:Switzerland,and Bruker model: ER-av-400MHZ,origin: Switzerland are reported in ppm(δ),DMSO was used as solvents. The dielectric voltage breakdown strength and thermal conductivity testing were taken for UPR/TiO_2 and blended nanocomposites. The dielectric breakdown strength properties were tested using the device (Rang HV-50Hz 300 KV). The time rate to increase voltages in electrical breackdown (0.5KeV/ s) and thermal conductivity properties was tested by using A Lee's disc instrument manufactured by the Griffen and George Company, England.

Methods

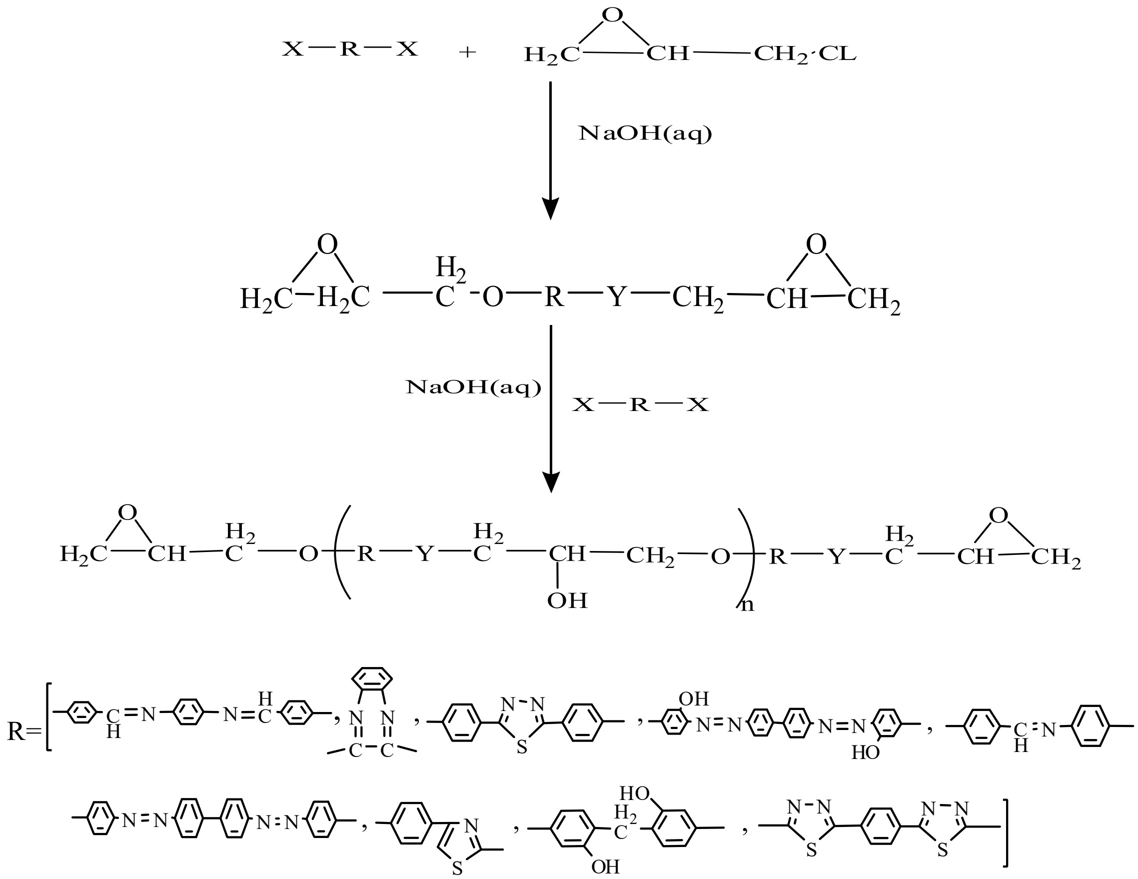
Preparation of monomers [M_1-M_{10}]

The monomer;Bis-[4,4'hydroxybenzylideneamino)phenol[M_1],quinoxaline-2,3-diol[M_2] ,[2,5-di-(p-hydroxylphenyl)-1,3,4-thiadiazole][M_3],4,4–diphenylenebis[(azo)-2,4-dihydroxy phen -yl][M_4],4-(4$^-$-hydroxybenzyliodeneamino)phenol[M_5],4,4-diphen-ylenebis[(azo)phenol l] [M_6] were prepared according to reference [18],2-amino-4-(4-aminophenyl)-1,3-thiazole[M_7] and 2-amino-4-(4–hydroxyphenyl)-1,3thiazole [M_8] were prepared according to reference [19],methylene-bis-(2,4-dihydroxyphenl) [M_9] was prepared according to the reference[20],and the monomer 1,4-bis(5,5-2-amino-1,3,4-thiadiazol)phenol[M_{10}] was prepared according to the reference [21].

Preperation of polymers [P_1-P_{10}]

A suitable monomer [M_1-M_{10}](0.01 mol) was dissolved in 10 % aq. NaOH solution (0.0188 mol) in a three neck round bottom flask which is balanced in an oil bath set at 50^{o}C. A homogeneous solution was obtained after vigorous stirring under reflux followed by addition of freshly distilled epichlorohydrin (0.0157 mol) in one batch. The temperature was raised to 90 ºC and the reaction proceed for another 2 hrs. Then the reaction mixture was allowed to settle and the aqueous layer decanted to give a big gel like mass product. After washing the product for many times with distilled water and neutralized with 38 % HCl, vacuum distillation was performed to remove any unreacted epichlorohydrin. [22].The structure and melting point of polymer are listed in Table(1).

The reaction pathway for the synthesis of epoxy prepolymer was illustrated in the following scheme.



X= OH, NH_2
Y= O, NH

Scheme

Preparation of semi-interpenetrating blend (S-IPNs)

For one type of new epoxy P_2 preparation semi-interpenetrating blends (UPR/Epoxy).Testing dielectric breakdown and thermal conductivity properties have been carried out for (semi-IPNs) the data are listed in Table(2).

The optimum quantity from Titanium dioxide

The optimum quantity from titanium dioxide (TiO_2) was determined by means of measurement properties when mixing different percentage (1,3,5,7%) from nano material with unsaturated polyester were mixed together by ultrasonic for 2 hour at room temperature, then add methyl ethyl ketone peroxide 2% to the mixture and mixed mechanical stirrer for 5 min., its found that 3% is optimum quantity. Table 3 illustrated the results of dielectric breakdown and thermal conductivity on respectively for UPR/TiO_2.

Preparation of polymer blend and composite

Unsaturated poly ester, epoxy resin in varying amounts (1,3,5,7%) and optimum quantity from titanium dioxide (TiO_2) (3%) were mixed together in a suitable beaker, and a mixture was placed in a high intensity ultrasonic bath for 2 hrs at ambient temperature conditions ,used ice bath to avoid temperature rise during the sonication process .

Then curing agent methyl ethyl ketone peroxide (2%) part by weight was added to mixture, the mixture was stirred well with mechanical stirring to give a homogeneous mixture, the mixture poured into mold of iron with dimension (15×5×0.4) cm and left overnight. The first curing was done at room temperature followed by a second curing step at 120º C for 2 hrs. When the solidification process is finished the sample was cut down into standard dimensions.

3. Results & discussion

Spectroscopic identification

Polycondensation was successfully conducted between epichlorohydrin and M_1,M_3,M_4,M_5,M_6,M_7,M_8,M_9 and M_{10} using aq. NaOH incentive as shown in Scheme1. The pure polymer and blended nanocomposite after post curing were studied by FTIR spectrum. The FTIR spectra of the purified epoxy prepolymer of polymer [P1-P10] showed the characteristic C-O band in oxirane ring and ether linkages formed which can be seen in Table 4 presents the principal FTIR stretching vibrations for the epoxy resins. Comparing the FTIR spectrum of UPR/TiO2, blended nanocomposites [B1] and polymer P_1 can be seen in Figure 1 the FTIR of blend polymer [B1] showed the appearance of good peak for C=O stretching band at 1722 cm-1 besides to other characteristic bands of polymer[P1] .The ^{1}HMNR spectrum (in DMSO as a solvent) for polymer P_2, showed the following signals: signals in the region δ(7.23-7.60)ppm for aromatic protons and OH proton. Many signals in the range δ (3.91-5.35) ppm for aliphatic protons of CH_2 methylene groups. the ^{1}HMNR spectrum (in DMSO as a solvent) for P_3 showed: a singlet signal at δ 8.87 ppm for proton of OH group, double of doublet signals at δ (7.32-8.06)ppm that could be attributed to aromatic protons and a signal at δ5.76 ppm for one proton of CH group. Another signal at δ (4.312-4.44)ppm due to four protons of $2CH_2O$ groups. The ^{1}HMNR spectrum (in DMSO as a solvent) for polymer P_5, showed the following signals: a singlet signal at δ 9.96 ppm for one proton of OH, a singlet signal at δ 8.56 ppm that could be attributed to proton of imine group (CH=N).Many signals in the region δ(6.59-7.83) ppm are due to aromatic protons. Also, multiplet signal between δ (4.70-5.59) ppm for protons of CH_2CH in oxarine ring and CH-OH group. while the protons of ($2OCH_2$) group appeared in region δ (3.70-4.20) ppm, the ^{1}HNMR can be seen in Figure (2).

Dielectric breakdown properties

Dielectric breakdown properties of the composites are mostly affected by the insulating structures, defects and impurities, such as the external factors form, type and the nature of the polar used and outer surface and measurement conditions such as the pressure, temperature, moisture, frequency the source, the time period in the application of voltages, the type and ratio of reinforced used in it. Dielectric breakdown strength values of blends nanocomposites with different weight percentage are significantly higher compared to that of the UPR/TiO_2.Figure 2 shows that the strength decreases with further increase in weight percentages of epoxy in some structure. Found where the chain conjugation was interrupted by aliphatic moiety, which led to increase flexibility due to a large volume and increase the conductivity and that led to decrease dielectric breakdown, On the other hand the conductivity was decreased where the chain was interrupted by long aliphatic group which weaken the conjugation in the main chain, so the flexibility effect on conductivity is dominate and dielectric breakdown. Illustrated the results of dielectric breakdown tests of the blended nanocomposite at different weight percentages of epoxy resins, at room temperature were shown in Figure 3 and Table 5.

Thermal conductivity properties

The blend nanocomposites show higher thermal conductivity than the sime-IPNs UPR/Epoxy for P_2, the results showed that the thermal conductivity increases with presence of nanoparticles. When comparing the thermal conductivity for UPR/TiO_2, blended nanocomposites the results showed that the thermal conductivity decreases with the increase of wt% of epoxy resin , the thermal conductivity of TiO_2/ UPR/Epoxy nanocomposites attains an optimum value of 0.307933 W/m.K for B_2 at loading 1% wt Epoxy. But when increase %wt of Epoxy with existence percentage a fixed of the TiO_2 led B_2 show minimum value of thermal conductivity. The high density of functional end groups thermosetting hyperbranched epoxy resin is conducive to well dispersion of nanoparticles and improve the compatibilities and interaction between the particles and matrix, thus compromise the comprehensive properties of the thermally conductive. Illustrated the results of thermal conductivity tests of the composite at different weight percentages of epoxy resins, at room temperature were shown in Figure (4) and Table (6).

Thermal studies

TGA analyses for the blended nanocomposites after curing are shown in Figure (5), the blended nanocomposites has thermal stability as shown in Table (7). Illustrated the onset temperature(°C), temperature of maximum (°C) and Temperature of half loss (°C) .

Conclusion

Epoxy prepolymer was successfully prepared**,** epoxy resins are highly miscible with UPR. Dielectric breakdown property for blended nanocomposites show maximum improvement at about 3% by wt epoxy concentration, for B_9 was 13.57KV/mm. Thermal conductivity lower with the increase of the percentage of epoxy for all blended nanocomposites. The thermal conductivity was increased from K=0.2205 W/mK for S-INPs (UPR/Epoxy P_2) to 0.255970 by for the B_2 with 3 wt% of epoxy resin. High electrical insulation and thermal conductivity will make TiO_2/UPR/epoxy nanocomposites promising in the electrical package field. In blended nanocomposites show substantial improvement in thermal stability.

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Table (1): Summary of compound structure, melting point

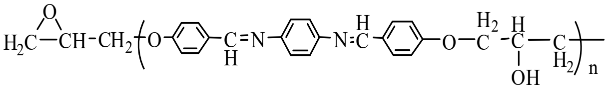
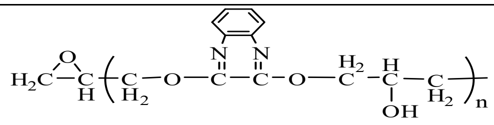
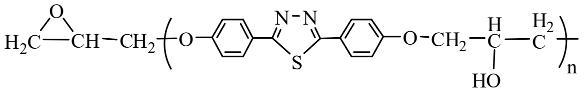
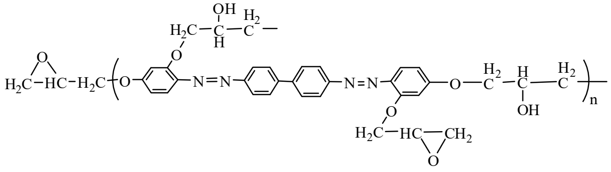
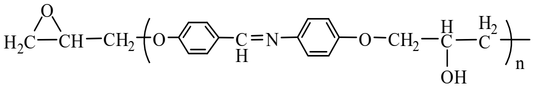
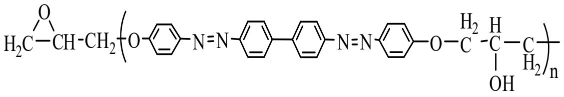
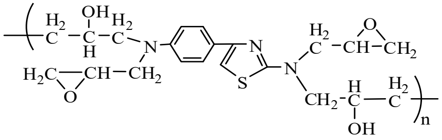
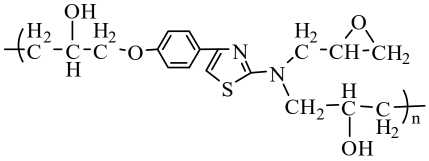
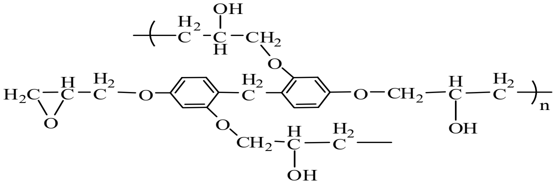
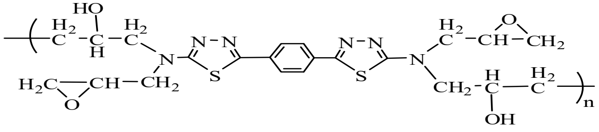
symbol of polymer	Structure of polymer	M.P.
P1		>300
P2		250
P3		>300
P4		>300
P5		128
P6		>300
P7		>300
P8		>300
P9		>300
P10		>300

Table (2): Dielectric breakdown and thermal conductivity properties of S-IPNs UPR with (1, 3,5,7) wt% epoxy resin [P_2].

Property	UPR/(1%)wt Epoxy	UPR/(3%)wt Epoxy	UPR/(5%)wt Epoxy	UPR/(7%)wt Epoxy
Dielectric breakdown	9.71	9.91	10.56	11.97
Thermal conductivity	0.235	0.2205	0.2192	0.2102

Table (3): Dielectric breakdown and Thermal conductivity of properties of UPR+ (1,3,5,7)%TiO_2 modified

Property	UPR/TiO_2(1%)	UPR/ TiO_2(3%)	UPR/TiO_2(5%)	UPR/TiO_2(7%)
Dielectric breakdown	5.87	9.24	8.27	7.28
Thermal conductivity	0.29287	0.38438	0.29585	0.2860

Table (4): Summary of identification of infrared bands for epoxy resins[P_1-P_{10}]

Polymer No.	OH cm-^{1}	C-Hcm^{-1} aromatic	C-H cm-^{1} aliphatic	C=N cm^{-1}	C=C cm^{-1}	C-O-C cm^{-1}	C-O ring cm^{-1}	others
P_1	3369.64	3060	2927.94	1676.14	1512.19	1217	914.26	—
P_2	3419.79	3070	2935.66	1649.14	1595.13	1269.16	918.12	—
P_3	3400.50	3060	2931.80	1602.85	1504.48	1238.30	952.84	—
P_4	3400.50	3060	2935.66	—	1597.06	1246.02	914.26	N=N1460.11
P_5	3388.93	3076.46	2931.80	1678.07	1575.84	1240	914.26	—
P_6	3409.33	3054.41	2930.96	—	1495.86	1234.18	943.23	N=N1460.18
P_7	3325.28	3030	2918.30	1606.70	1515.5	1257.59	929.69	—
P_8	3315.63	3010	2924.09	1604.77	1533.34	1240.23	939.33	—
P_9	3394.72	3000	2939.52	—	1510	1246.02	941.61	—
P_{10}	3377.36	3001	2945.30	1608.63	1552.70	1265.3	939.33	—

Table (5): Summary of dielectric breakdown properties of blended nanocomposites (B_1-B_{10})with (1,3,5,7) wt% epoxy resins in the present 3%TiO_2.

Compound No.	Dielectric breakdown Strength KV/mm			
	UPR+1%epoxy	UPR+3%epoxy	UPR+5%epoxy	UPR+7%epoxy
B_1	10.24	11.07	10.27	9.6
B_2	10.22	11.97	9.87	9.8
B_3	12.9	11.17	10.9	10.5
B_4	12.18	12.9	12.3	11.78
B_5	11.2	12.8	12.07	11.78
B_6	12.15	11.42	10.97	11.87
B_7	12.18	12.57	12.97	11.96
B_8	11.5	12.45	12.01	11.32
B_9	12.48	13.67	13.64	11.7
B_{10}	10.16	10.61	11.74	10.8

Table (6): Summary of thermal contuctivity properties of blended nanocompos -ites[B_1-B_{10}] with (1,3,5,7) wt% epoxy resins in the present 3%TiO_2.

Compound No.	Thermal conductivity Strength W/mK			
	UPR+1%epoxy	UPR+3%epoxy	UPR+5%epoxy	UPR+7%epoxy
B_1	0.414000	0.300665	0.239722	0.274821
B_2	0.307933	0.255970	0.234000	0.232596
B_3	0.309000	0.257000	0.244063	0.239500
B_4	0300007	0.327208	0.321062	0.272081
B_5	0.32162	0.301871	0.275231	0.231677
B_6	0.289663	0.271310	0.277302	0.225242
B_7	0.248258	0.234637	0.231171	0.215556
B_8	0.295200	0.280180	0.273146	0.268891
B_9	0.243371	0.267000	0.23783	0.27851
B_{10}	0.158733	0.196705	0.187540	0.18324

Table (7): Thermal properties-TGA blended nanocomposites in the present 3%TiO_2.

Blend NO.	Onset temperature (°C)	Temperature of maximum (°C)	Temperature of half loss (°C)	Temperature of melt(°C)
UPR/TiO_2	281.1	409.1	389.2	556.7
B_1	268.9	404	386.5	552
B_2	263.5	379.7	405.3	540.5
B_3	197	405.8	371.6	537.4
B_4	192	406.2	365	547.3
B_8	264.9	395.6	372	552.7

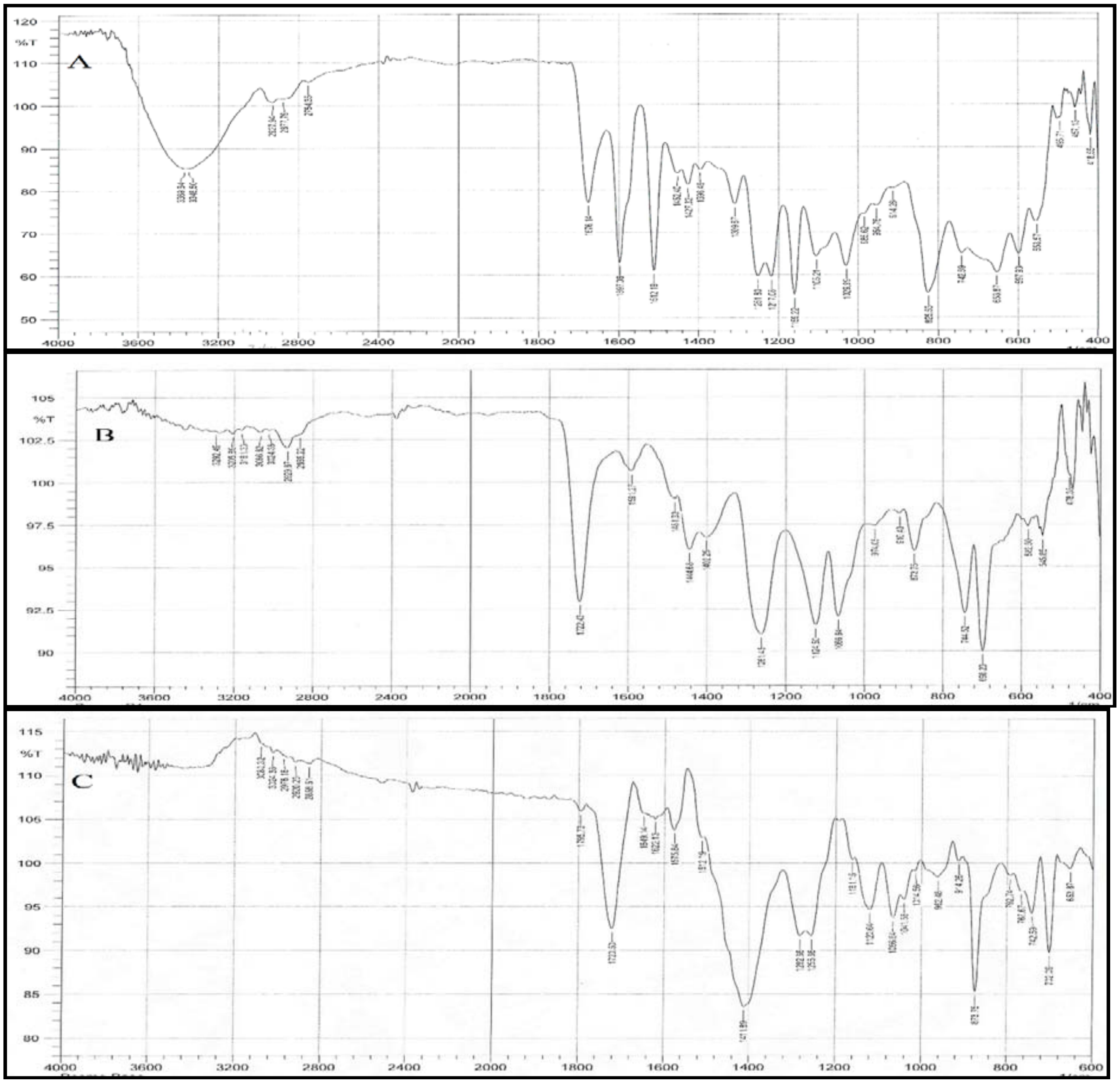


Figure (1) A: The FTIR –spectrum of [P_1] , B: The FTIR –spectrum of blended nanocomposite [B_1], C: The FTIR –spectrum of UPR/TiO_2.

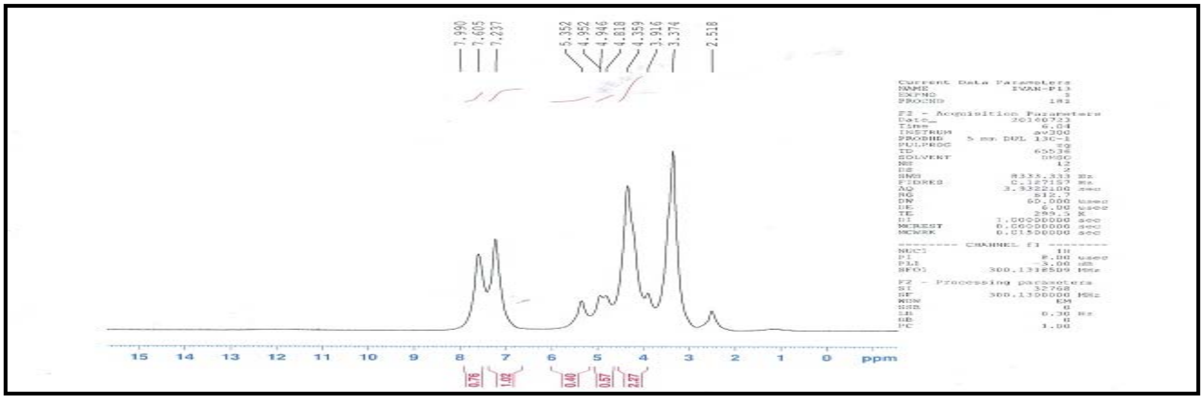


Figure (2) A: The ^{1}HNMR –spectrum of [P_2]

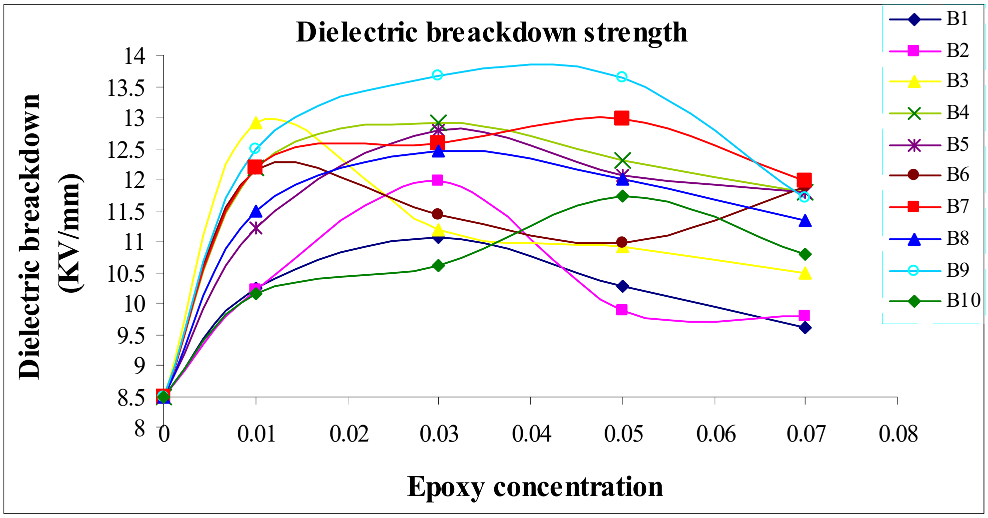


Figure (3): Dielectric breakdown properties strength of blended nanocomposites versus epoxy concentration

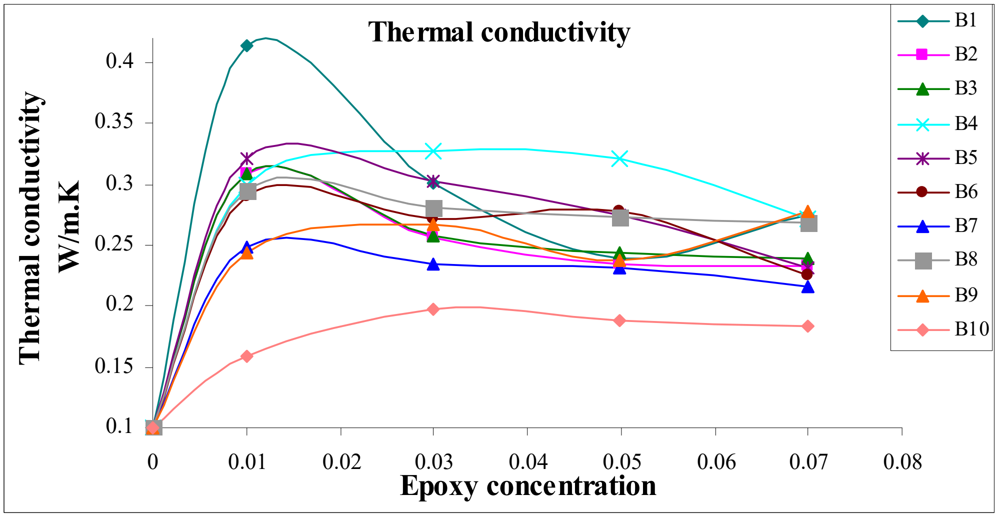


Figure (4): Thermal conductivity properties strength of blended nanocomposites versus epoxy concentration

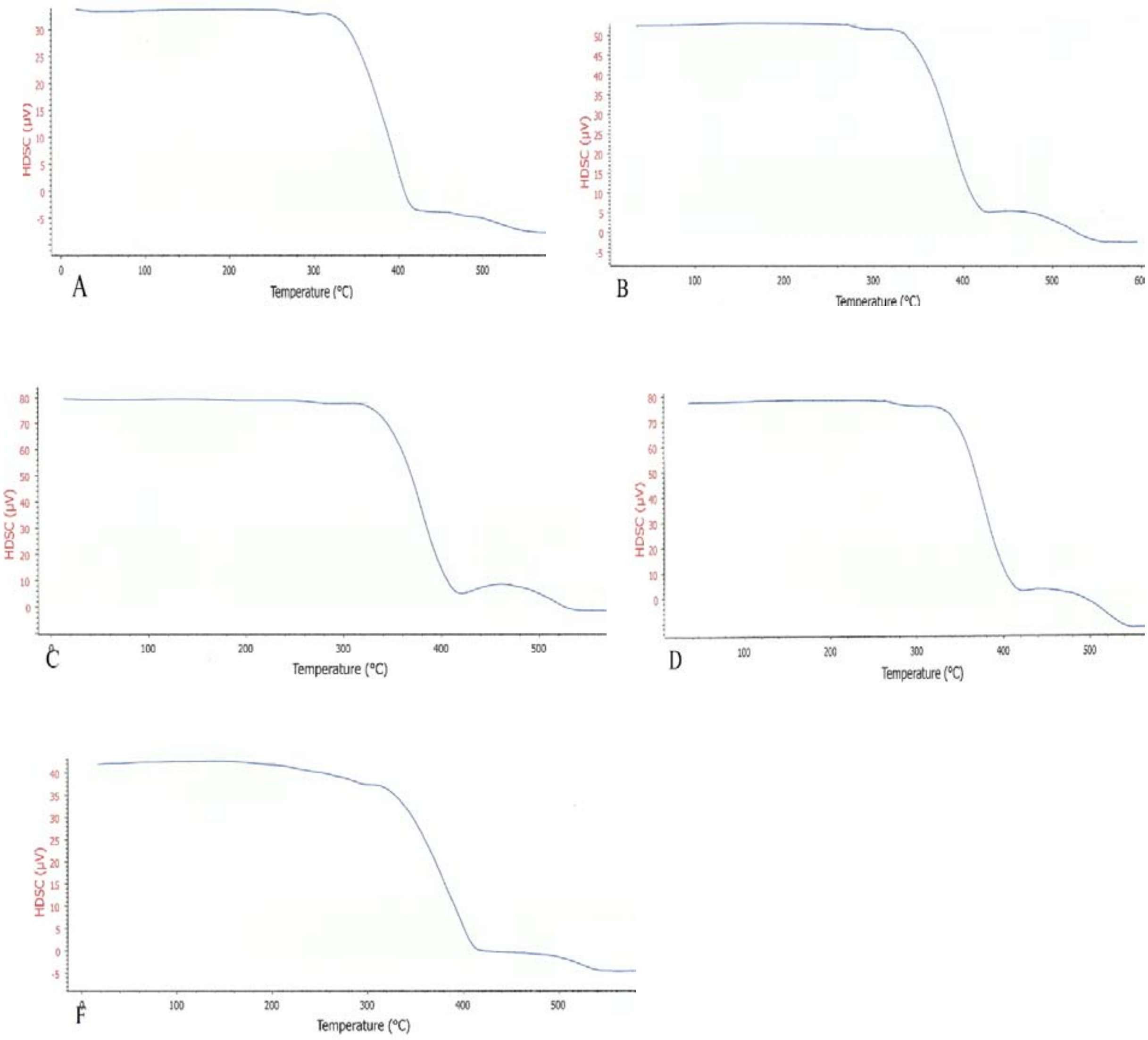


Figure (5): TGA of UPR and blended nanocomposites, A:UPR, B:blend $_1$,C: blend$_2$, D: blend $_3$, E:blend $_4$,F: blend $_8$.

قوة انـهيار العزل الكهربائي والتوصيل الحراري لمختلف السبائك النانوية المتراكبة لراتنج الايبوكسي الجديد

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الخلاصة

يكشف هذا البحث عن تحضيروتطوير عشرة مخاليط متراكبة ومليئة بجسيمات نانويه وهي ثاني أوكسيد التيتانيوم (TiO_2). تم تحضير شبكات البوليمر المتراكبة النانويه على أساس راتنج البوليستر غير المشبعة (UPR) وراتنجات ايبوكسي جديدة مختلفه عن طريق مزج فعال .أذ تم اختيار النسبه المثلى من الجسيمات النانويه لثاني أوكسيد التيتانيوم (3%) وهي نسبة مختارة من نتائج مزجها بنسب مختلفة مع راتنج البولي استر غير مشبع. ومن ثم مزجت النسبة المثلى (3%) من(TiO_2) مع بولي استرالغير مشبع مع نسب مختلفه من مركبات الايبوكسي الجديدة 1 ٪ , 3٪ , 5٪ و 7٪ من وزن راتنج البولي استر غير مشبع. وتمت مقارنة خصائص قوة انهيار العزل الكهربائي والتوصيل الحراري للمتراكبات النانويه المقساة مع تلك المواد الأساس (UPR و 3٪ TiO_2). تظهر الاولى قوة انهيار العزل الكهربائي اعلى في حين التوصيل الحراري هي اقل وكما تم تحضيرمخاليط لاحد تراكيب الايبوكسي رقم (2) وبالنسب المذكورة نفسها (UPR و Epoxy) و مقارنتها مع مخلوط المتراكبة النانويه لوحظه ان قوة انهيار العزل الكهربائي للمخلوط المتراكبة النانويه هو الاعلى عند نسب التحميل الواطئة للإيبوكسي رزن وكما اظهرت التوصيلية الحرارية عالية مقارنة بما اظهرته مخاليط من (Epoxy/ UPR) .اجري التحليل الحراري الوزني (TGA) لدراسة الخواص الحرارية للمخاليط المتراكبة النانويه .

الكلمات المفتاحية:السبائك النانويه المتراكبة,البولي استر الغير مشبع,قوة انهيار العزل الكهربائي,الايبوكسي رزن.