



Synthesis and Properties of Sulphonated Polystyrene, Their New Composites and IPNs

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Abstract

Sulfonated polystyrene (SPS) is a polyelectrolyte solid that is widely used in many applications. In this study, the sulfonation reaction of food packages polystyrene (PS) waste was done using sulfuric acid as a sulfonation agent, with the variation of concentration and time. The degree of sulphonation was determined. Composites from SPS and two types of fillers (carbon black and nanosilica) were synthesized, and new IPNs between SPS and phenolic resin type novolac were prepared. The characterization of the sulfonated product and its IPNs has covered functional groups' analysis using FTIR spectrophotometers. On the other hand, tensile and modulus of the SPS composites were determined, and the results showed that the tensile strength and modulus increased with increasing filler content (0.48 Map for SPS alone to 1.051 Map when the percentage of Nano silica 7%) while the modulus increased from 0.527 Map for SPS alone to 1.272 Map when the percentage of Nano silica 7% , The same results were obtained in the case of carbon black as filler. The water absorption of the prepared thin films for SPS was greater than that of PS alone due to the presence of a polar sulphonic group in their polymer chains . The thermal behavior of SPS and their IPNs also studied and evaluated using TGA and DSC techniques; the results show that the decomposition temperature was more than 300 °C

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1. Introduction

Recycling of polymers is very interested methods used to rid the environment from pollution and accumulation of polymeric materials [1]. PS has many unique properties and can be used for any think from take-away food containers to multi-pack yogurts[2, 3] Like many other plastics. PS is non-biodegradable polymers[4, 5] PS comes in two forms: solid and foamed, and it is a very versatile material. Polystyrene (PS) foam has been widely used as an insulator and in packaging. More than a thousand tons of this plastic worldwide is being disposed of into the environment as municipal solid waste (MSWs). The amount is increasing every year. However, especially in developing countries, the successful development of electronic products has sharply increased Waste quantities from Electrical and Electronic Equipment (WEEE), amplifying their disposal problem, so most of the PS waste is discarded as a solid pollutant, so the solution can be found only through a modern Design for Environment (DFE) with big attention to recycling and dissemble [6-8]. PS, which is the subject of this work, has been recycled chemically in many. Ways, for instance, styrene and styrene dimer, trimer and oligomer were recovered from PS by thermochemical treatment [9], and by catalytic thermo- chemical treatment [10]. Functionalizing of the benzene ring which is a characteristic segment of the PS supports the formation of new polymer derivatives [11]. Many researches used sulphonated group in order to the sulphonated ion reaction of PS, and most of these studies have used sulphuric acid as sulphonating agent [12, 13] while other studies have used silica sulphuric acid [14], and the others used acetyl sulfate complexes as sulfonation reagent in a solution of di chloro ethane [15]. The sulfonation of polystyrene with sulfonic acid groups has been effective with various nitrogen-containing polymers (like, polyamides, conducting polymers: polyaniline). SPS has been growing in various industrial, domestic and medical applications. It also has been studied as a membrane for several electrochemical uses, such as fuel cells [16, 17]. On the other hand interpenetrating networks (IPNs) was the new process used to prepare polymeric material through mixing two types of monomers and there initiators and polymerized them to form new network structures. In this work high degree, water soluble sulphonated PS (SPS) was synthesis using packaging food waste PS and sulphuric acid as sulphonating agent, and new composites and IPNs were synthesis from the SPS.

2. Experimental Part

2.1 Materials

The PS plastic waste (food Jars) was collected and cut to obtain homogeneous grains of average size (10 x 5) mm. Sodium hydroxide, methanol, dichloroethane Hexamine, and novolac were supplied from Fluka comp. Sulphuric acid 96.11 %, hydrochloric acid 0.1 N Amplo (stander),



phenolphthalein indicator, styrene and Acrylonitrile monomers from BDH comp. Carbon black was supplied from the state company for Tine Industry (Iraq) with the following specification (Type N660). Iodine absorption ($36 \text{ g/Kg} \pm 5$), surface area ($36 \text{ m}^2/\text{g} \pm 5$), PH value (6-9), Heating less at $125 \text{ }^\circ\text{C}$ (max 1%), and mesh size (500- 700) nm. Nano silica was used as reinforcing material in the processes was supplied from Tokuyama (Japan) with the following specification, practical size Nanometer was (7-12 nm), surface property (hydrophilic), specific surface area was $220 (\text{m}^2 / \text{g})$, and bulk density was 50 g/L .

2.2 Instruments

The viscosity of the prepared SPS in water was measured using Brookfield viscometer series, Model RVDV-II with spindle. The FTIR spectra of the prepared samples was done using KBr disc. The spectrum was recorded in Shimadzu type 8400S in the frequency range of $400\text{-}4000 \text{ cm}^{-1}$. The stress-strain curves (Tensile strength and elongation) of SPS composites were done a Zwick Z010 (Zwick, Germany) universal testing machine, using dumbbell shaped specimens. Absorption was determined according to the standard ASTM DS 70-98 test methods. The samples films of PS and SPS (50mm X 0.5 mm) were prepared by dissolving SPS with low degree of swelling in 1,2 -dichloromethane solvent and casting in petri-dish, evaporate the solvent at room temperature, after this the samples were evacuated in oven at $40 \text{ }^\circ\text{C}$ for 6 hours. Then they were immersed in the vessel in order to determine the percentage of the water absorption using equation 1

$$\text{Water absorption \%} = (W_t - W_0 / W_0) \times 100 \dots\dots\dots (1).$$

Thermal measurements was recorded on TGAQ50V20, and the measured temperature range was $25\text{-}800 \text{ }^\circ\text{C}$, using nitrogen as inert gas to prevent oxidation of the samples, at heating rate of $50 \text{ }^\circ\text{C}/\text{min}$. Deferential Scanning Calorimetry (DSC) was recorded in a temperature range of $25\text{-}350 \text{ }^\circ\text{C}$ using Shimadzu DSC-60. at heating rate of $10 \text{ }^\circ\text{C}/\text{min}$, under a nitrogen atmosphere.

3.2 Synthesis

3.2.1 Synthesis of sulphonated PS (SPS)

SPS was prepared by dissolving waste PS (10 g) in 200 ml chlorinated solvent (1,2dichloromethane), then several quantities of sulphuric acid (5,10,15 and 20) ml was added dropwise with good stirring at $40 \text{ }^\circ\text{C}$ for several hours (2,4,6 and 8) hours. Hours. After that, the mixture was poured into ice distilled water to precipitate white product (SPS), then the product was washed with distilled water, then with sodium bicarbonate solution until the product was neutralized, filtered and dried under vacuum oven at $40 \text{ }^\circ\text{C}$ for about 6 hours.

3.2.2 Synthesis of composite materials.

SPS composite was synthesized using two types of fillers, carbon black type N660, and Nano silica, so 5g of high degree SPS dissolve in distilled water until the mixture's viscosity was 112 cp at 34.5 °C. This was taken about 20 ml of water. Several percentages of carbon black and Nano Silica (2.5,5 and 7.5) % related to SPS were added to the polymer solution with continuous stirring to get a homogenous solution. The mixture was then casting uniformly on polyethylene mold and kept for (3-5) days to full drying.

3.2.3 Synthesis of New IPNs (SPSN)

New semi –IPNs was synthesis from SPS and phenolic resin type (novolac).3g of SPS was mixed with 3g of novolac containing 15% hexamine as hardener, in a mortar in order to get homogenous mixture, then press the product to get disc sample. The disk was molded in a hot press at a temperature of 135 °C±5 using 50 ton for pressing the sample at about 30-35 min in order to cure the sample. The sample was ready to TGA analysis.

3.2.4 Synthesis of SPS- co polyurethane (PUS).

Polyurethane type sps were synthesized using methylene diphenyl diisocyanate (MDI) as a curing agent. 5g of SPS was dissolved in 30 ml of 1,2-dichloromethane, then different amount of MDI was added (5,10 and15) g to the solution with a good stirrer at room temperature for about 1 hour, after that the mixture was cast in a Petri dish and kept about 48 hours to complete curing. A samples sheet was taken to the tensile strength study. Also, the product was characterized by FTIR and TGA.

4.Result and Discussion

4.1 Sulphonation Process

Using deferent quantity of acid and times to sulphonation of PS order to determined degree of sulphonation. The reaction involved homogeneous sulphonation in the para position of benzene ring of ps as shown in Fig. 1.



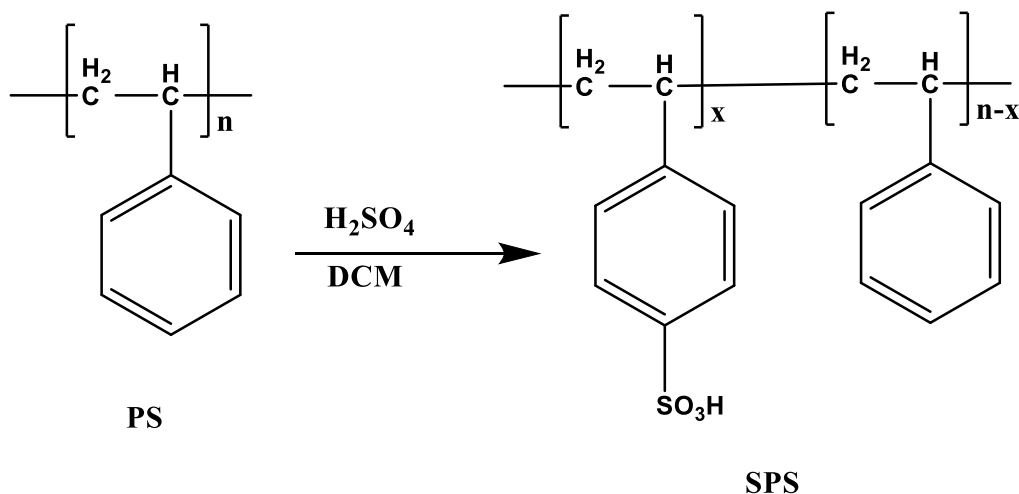


Figure 1: Synthesis of SPS

In all the different quantity of sulphuric acid the product (SPS) was white powder. Degree of sulphonation was measured [18], by titration using 0.1 N solution of sodium hydroxide. 1 g of SPS was dissolve in 20 ml mixed solvent toluene: ethanol (1/9 v/v) toluene (1/9 v/v) and the degree of sulphonation was calculated using following Eq. 2.

$$\text{Degree of sulphonation \%} = \frac{104 \, n \, v}{m - 81 \, n \, v} \times 100 \quad \dots\dots\dots (2).$$

n was the concentration of titrant, while **v** was the volume of sodium hydroxide used in titration, **m** was the weight of SPS sample. The number 104 was the molecular weight of styrene unite, while 81 was the molecular weight of sulphonic group. Figure 2. show the relationship between the degree of sulphonation and the Times with different concentration of sulphuric acid. The results show that the sulphonation process increase with increasing acid concentration, this may be due to the increasing the reactivity of sulphuric acid. Also, the result indicates the increasing in conversion with increasing reaction times. The optimum value of degree of sulphonation at 20 ml sulphuric acid and 10 hours times. The SPS with high degree of sulphonation was very soluble in water , while the others dose not , on the other hand low degree of SPS was soluble only in chlorinated solvents. This behavior due to the increasing in hydrophilicity of the polymer with increasing sulphonated groups

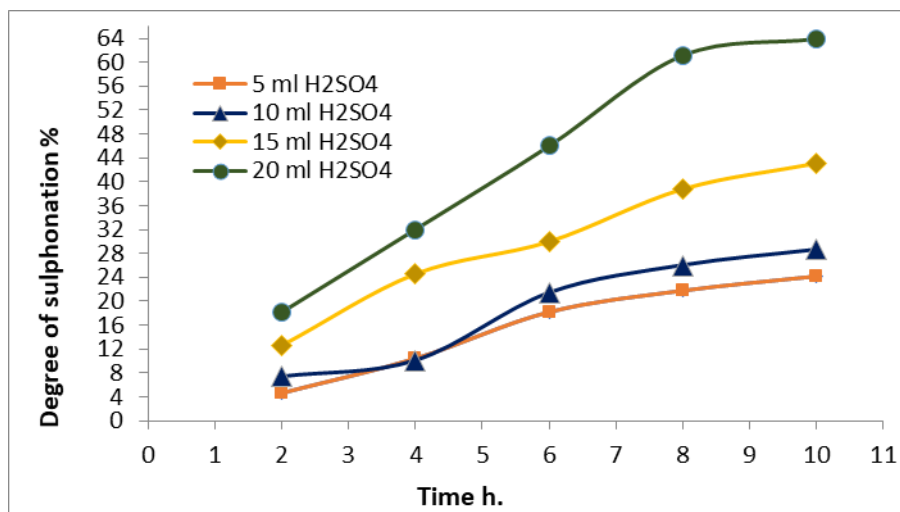


Figure 2: Degree of sulphonation products against times

On the other hand, polyurethane was prepared from SPS and MDI, through the reaction between sulphonic group and isocyanate group leading to produce polyurethane linkage according to the following reaction as shown in Fig. 3.

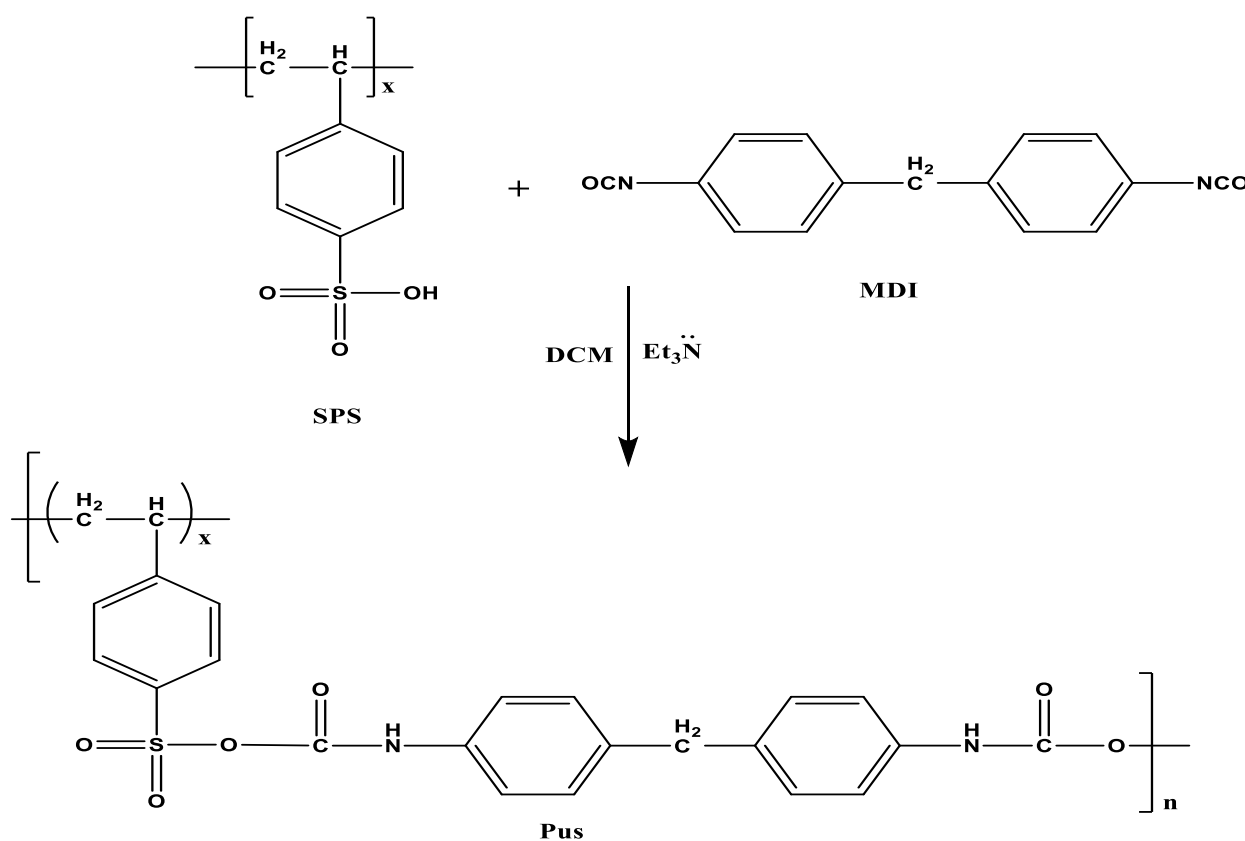


Figure 3. Synthesis of PUS

While the semi IPNs of SPS and novolac resin was prepared mixing SPS with (novolac containing 15% hexamethylene tetra amine as hardener) and then mold the mixture under pressure and heat , the scheme of the synthesis was shown in Figure 4.



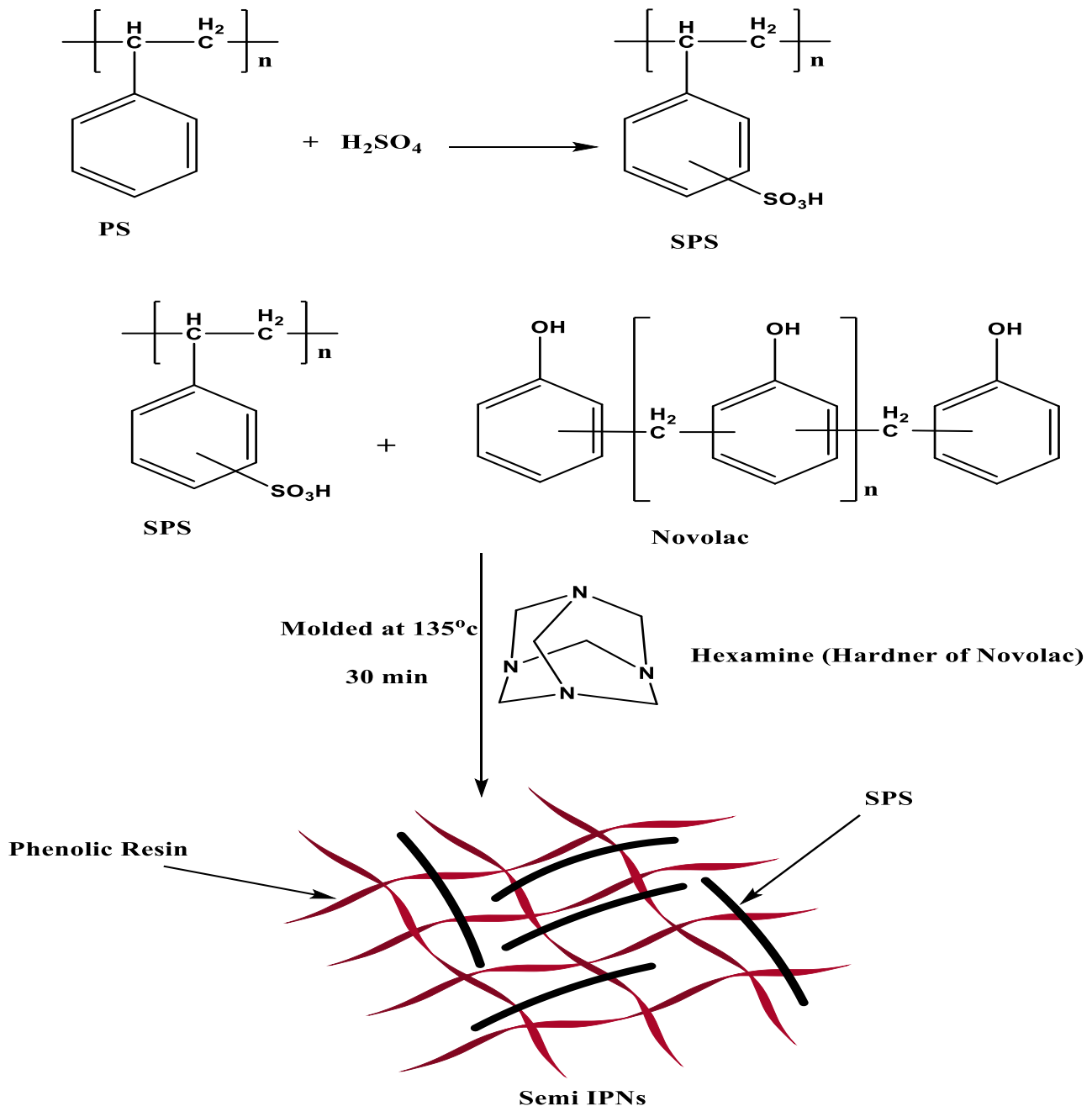


Figure 4: Semi IPNs between SPS and phenolic resin

4.2 FTIR Study

FTIR spectroscopy was used to study the sulphonation product. The spectrum in Figure 5. shows the characteristic peak at 1180 cm^{-1} , related to stretching vibration of SO_2 which is the good indication in the sulphonation reactions [19, 20]. Also, new absorption bands at $3417, 1068\text{ cm}^{-1}$ due to vibration of the hydroxyl groups and stretching of $\text{O}=\text{S}=\text{O}$ group, respectively. In the case of PUS, the spectrum shown in Figure 6. Bands at $1712, 2272\text{ cm}^{-1}$ was a good evidence to polyurethane linkage groups and stretching of isocyanate groups respectively, also the stretching vibration of $\text{N}-\text{H}$ was appear at 3410 cm^{-1} [21]. The spectrum of new IPNs between SPS was shown in Figure 7. the

strong band appears at 2920, 2850 cm^{-1} donating to methylene group of phenolic resin in the network structure, also band at 3228 cm^{-1} due to hydroxyl group and the double bond in benzene rings was appear at 1600 cm^{-1} . The band at 1330 cm^{-1} was due to the sulphone group in the IPNs structure.

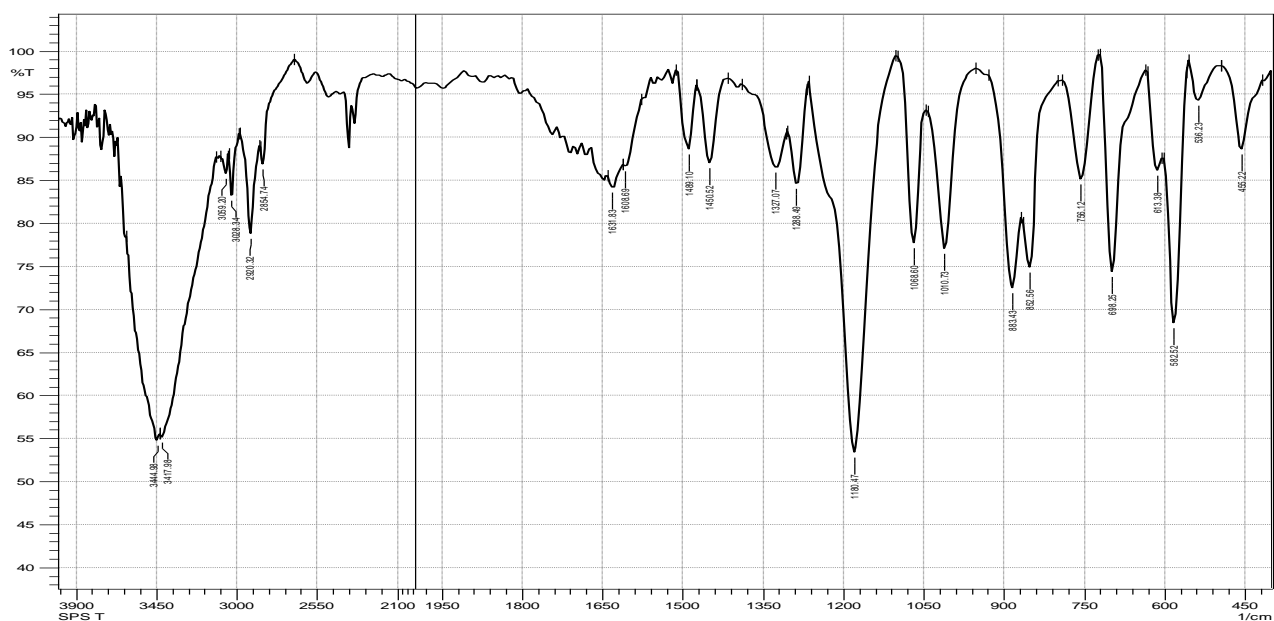


Figure 5: FTIR spectrum of SPS

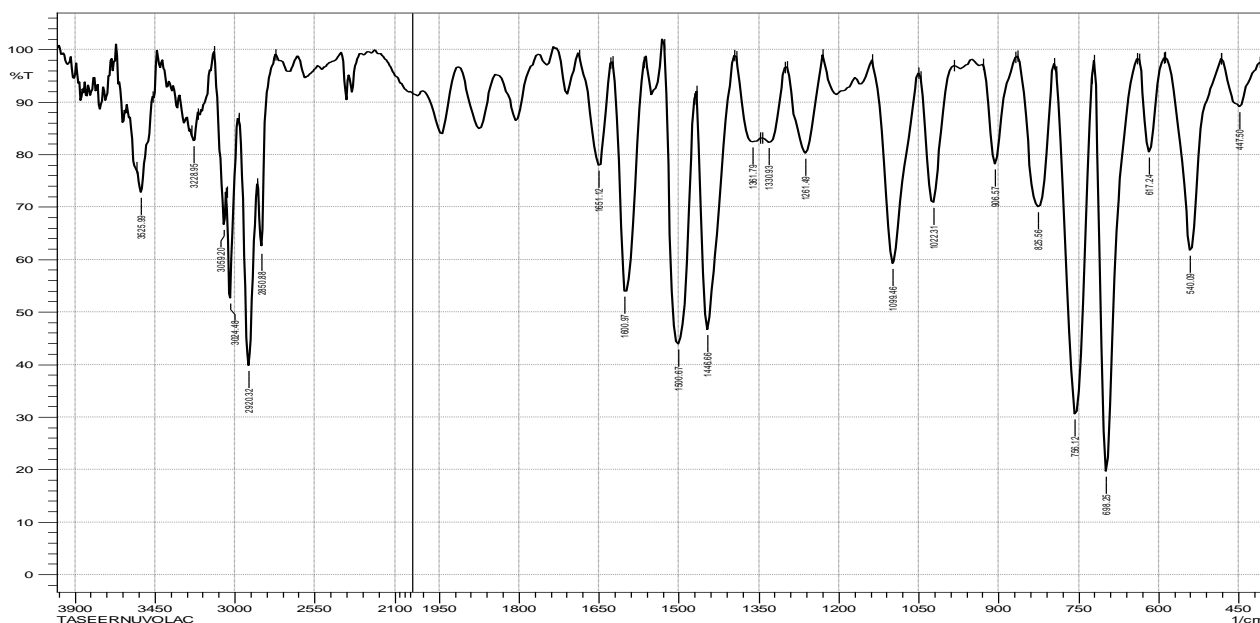
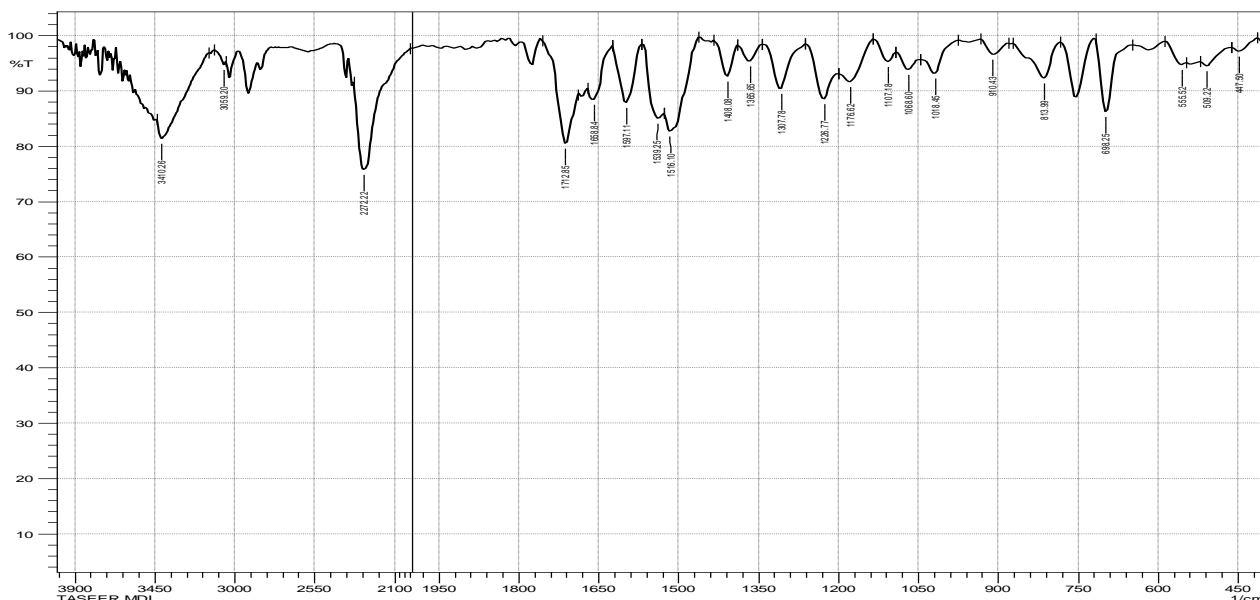


Figure 7: FTIR spectrum of SPSN

4.3 Water absorption test

Water absorption tests of PS and SPS was measured during 30 hours, and the results of this study as shown in Figure 8. indicates that the SPS absorb water than PS, this due to the presence of polar sulphonic group in the SPS which leads to somewhat highly absorbed water than PS alone. On the

other hand, the maximum value of water absorption of SPS after 25 hours while for PS after 18 hours and the curves remain nearly constant.

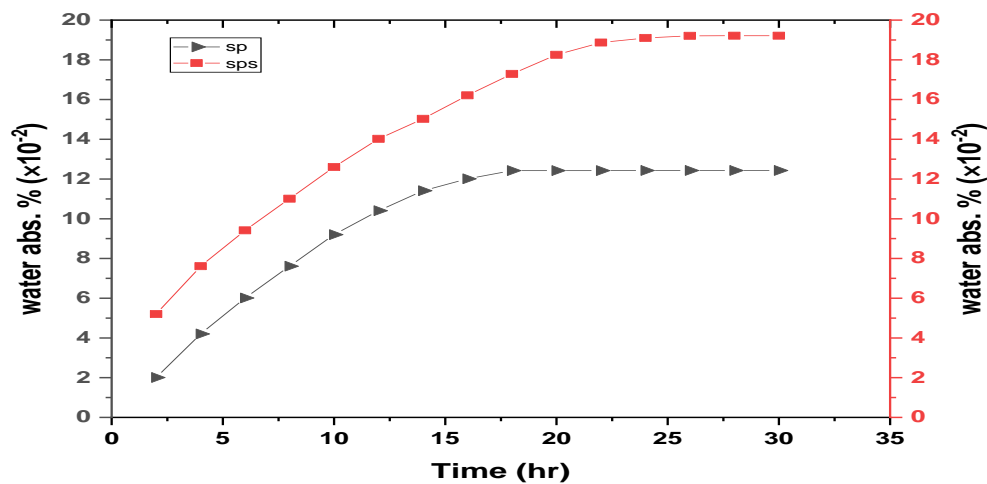


Figure 8: Water absorption of PS (black line) and SPS (red line)

4.4 Tensile strength test

The effect of untreated and treated sps with (carbon black and Nano silica) on the stress-strain curves were shown in Figures (9,10), while the results indicates that the tensile strength and modulus was increase with increasing fillers while the elongation was decrease due to decrease the flexibility of the composites, this result was agreement with many research's [22, 23]. On the other hand, the value of elongation of SPS-reinforced with carbon black was higher than SPS-reinforced with Nano-silica, as shown in Table.1 this may be due to sliding of carbon black which make somewhat flexibility of composite but still the value is less than the untreated SPS.

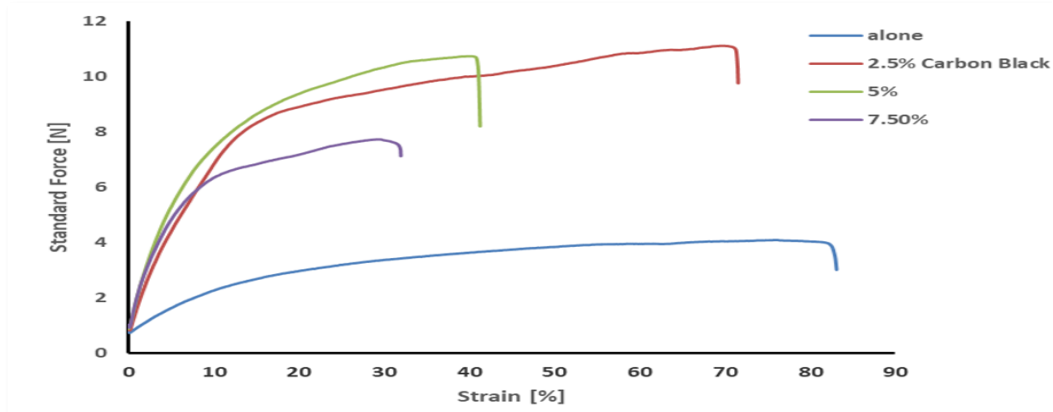


Figure 9: Stress-strain curve of SPS -carbon black composite

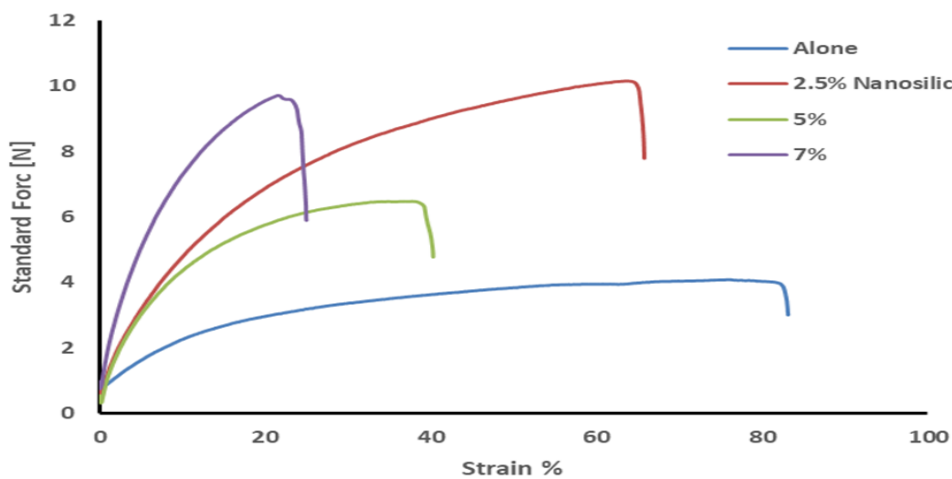


Figure 10: stress-strain curve of SPS-Nano silica composite

Table 1: Some mechanical properties of SPS Composites.

| Test | Results | | | | | | |
|------------------------|-----------|-------------------|-------|-------|--------------------|-------|-------|
| | SPS alone | SPS-Nano silica % | | | SPS-Carbon black % | | |
| | | 2.5 | 5 | 7.5 | 2.5 | 5 | 7.5 |
| Tensile strength, Map | 0.480 | 0.536 | 0.985 | 1.051 | 0.511 | 0.991 | 1.041 |
| Modulus, Map | 0.527 | 1.171 | 1.210 | 1.272 | 1.132 | 1.174 | 1.311 |
| Elongation at Yield, % | 83.90 | 63.51 | 36.92 | 21.63 | 69.80 | 40.10 | 29.65 |
| Elongation at Break, % | 97.11 | 65.82 | 40.34 | 25.01 | 71.62 | 41.31 | 32.01 |

4.5 TGA Study

The TGA techniques was used to evaluated thermal stability of the prepared materials , and this techniques is very important and widely used to determined decomposition temperature of polymers and other materials, [24]. Thermal stability of SPS, IPNS of SPSN and PUS was done at heating rate of 20 °C / min. Figures (11-13) show TGA and differential thermogravimetric (DTG) of these polymers respectively. According to the obtained results as shown in tables (2,3) ,the degradation process of SPS and SPSN starts at temperature higher than 400 °C ,while for PUS was higher than 350 °C ,on the other hand the optimum decomposition temperature (Top) of the SPS was 424°C ,while for the SPSN the TGA thermogram show two stage of decomposition , the first with Top at 420 °C which refer to degrade of SPS ,while the other at Top at 543 °C refer to novolac resin .In the case of PUS , the TGA thermogram indicate three stages of decomposition at Top 235 , 366 and 648 °C respectively .As a conclusion the rate of decomposition and the Temperature of 50% weight loss of SPS was less than SPSN and PUS which indicate that the thermal stability of the latter two polymers was higher than SPS alone.

Table 2: Some thermal parameters of the prepared polymers obtained from TGA and DTG thermograms

| Polymer Type | Results | | | |
|--------------|---------------|-----------------------------|--------------------------------|--------------------------|
| | Top°C | Temp.of 50% weight loss, °C | Rate of decomposition % / min. | Char residue at 800 °C % |
| SPS | 424 | 432 | 0.41 | 18 |
| SPSN | 420 , 543 | 439 | 0.32 | 2 |
| PUS | 235, 366, 648 | 579 | 0.37 | 1 |

Table 3: Weight loss of the polymers at different temperatures

| Temperatures °C | Weight Loss | | |
|-----------------|-------------|------|------|
| | SPS | SPSN | PUS |
| 50 | 1.50 | ---- | ---- |
| 100 | 4.24 | 0.23 | 0.25 |
| 150 | 5.46 | 0.98 | 1.01 |
| 200 | 5.66 | 1.91 | 2.03 |

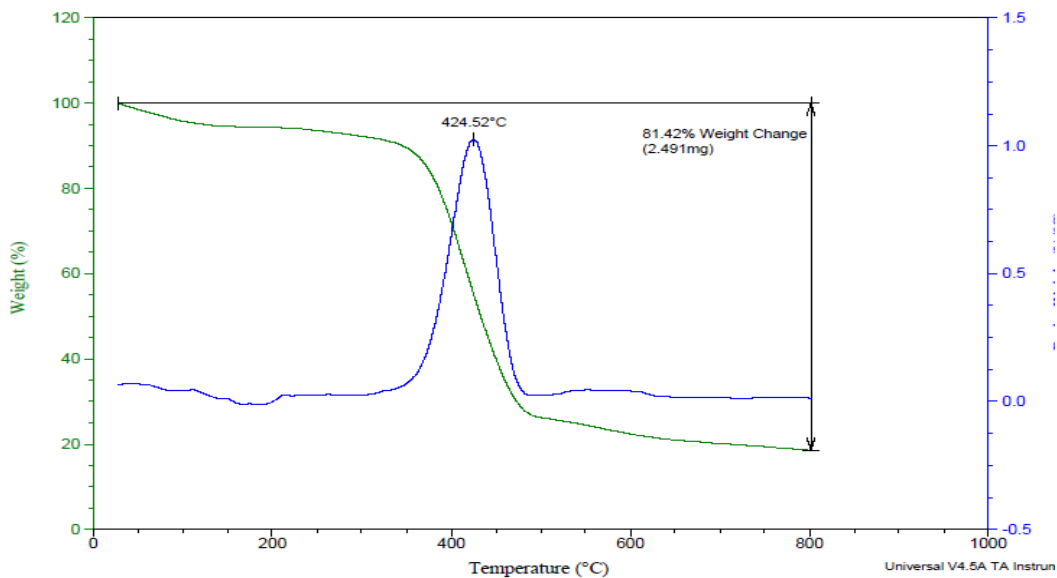


Figure 11: TGA and DTG thermograms of SPS

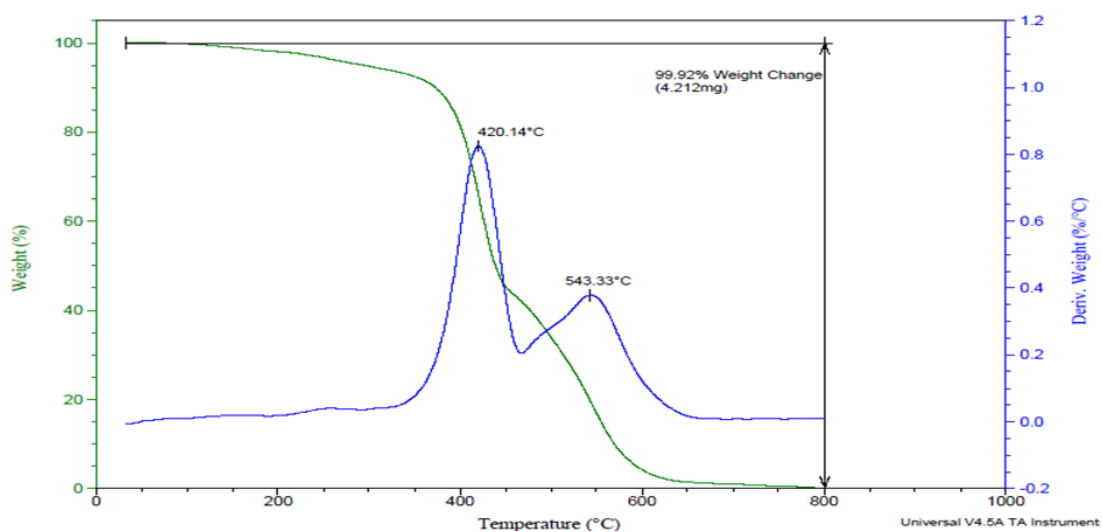


Figure 12: TGA and DTG thermograms of SPSN

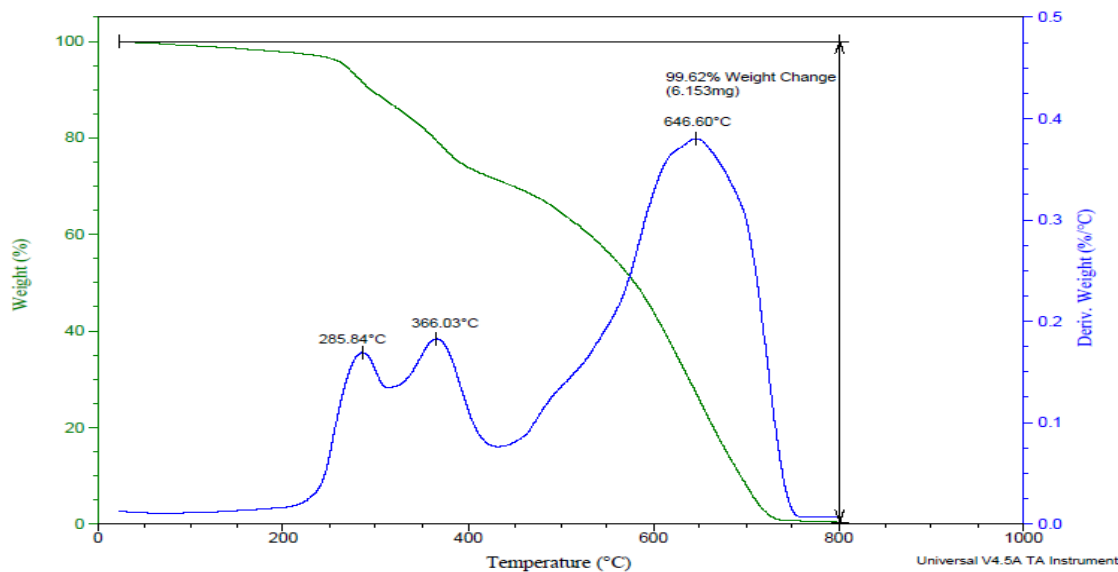


Figure 13: TGA and DTG thermograms of PUS

4.6 DSC study

DSC techniques is very important techniques used to evaluate the thermal transition of polymers [25] , and used to determined several thermal parameters of polymers like heat capacity and crystalline melting temperature as well as Tg. The DSC curves for SPS as shown in Figure (14) Indicate the presence of an endothermic peak at 290 °C due to the presence of sulphonic groups, while the Tg was nearly at 165 °C which was higher than polystyrene alone (Tg = 100°C), because of the presence this groups which leads to more steric effect thane PS alone [26] .In the case of DSC thermogram of the SPSN as shown in Fig. 15 a shoulder peak at 287 °C indicate the Tg of the IPNs, the higher value indicates the more cross-linking formation in the network structure of the IPNs compare to PS and SPS. While the DSC thermogram of PUS as shown in Fig. 16 indicate the presence of a shoulder peak due to the Tg at 225 and then the copolymer began to degraded nearly at 300 °C.

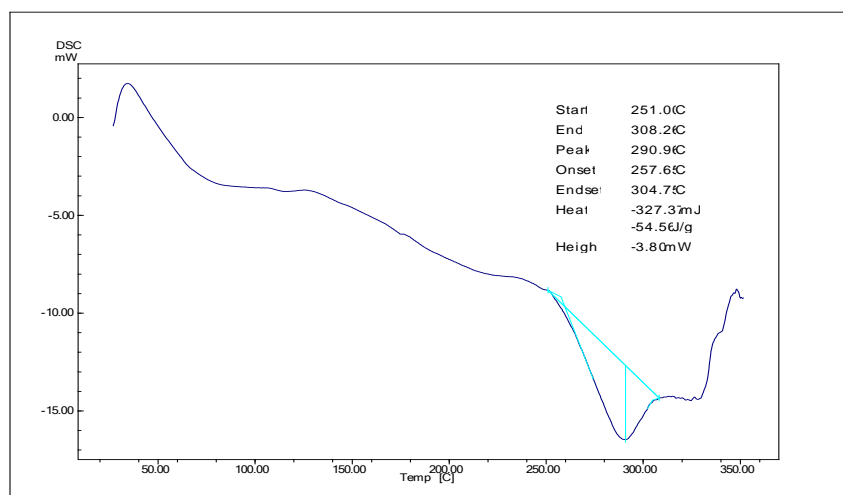


Figure 14: DSC thermogram of SPS

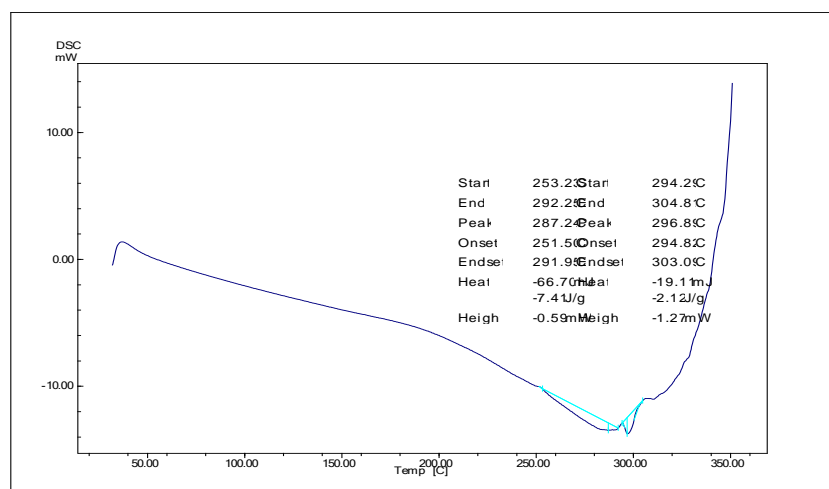


Figure 15: DSC thermogram of SPSN

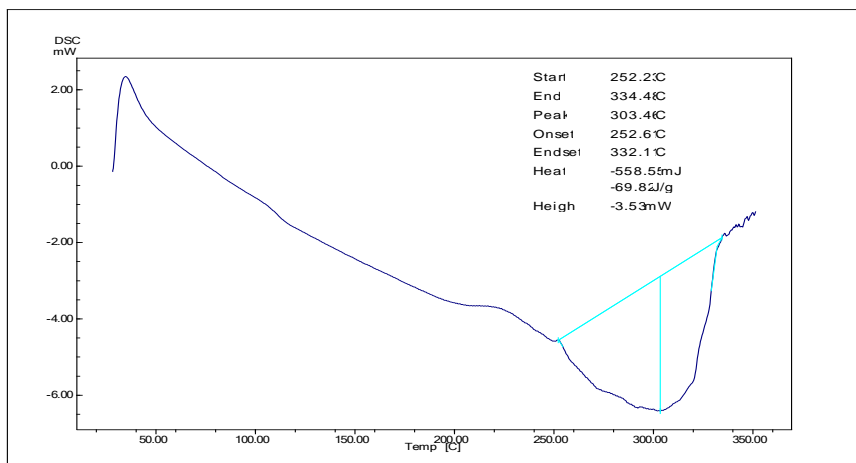


Figure 16: DSC thermogram of PUS

Conclusions

Sulphuric acid was used as a sulphonation agent to convert PS to SPS, and the optimum condition such as reaction time and sulphuric acid concentration were evaluated. The percentage of sulphonation was determined and was increase with increasing time and concentration. Elongation and ultimate strength of the prepared SPS composites were increased with increasing fillers content, and the results of using carbon black were better than using Nano silica. Thermal studies of SPS and their composites indicates high decomposition temperature.

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قسم الكيمياء- كلية العلوم- جامعة البصرة-العراق

المستخلص

يعتبر البولي ستايرين المسلفن (SPS) من البوليمرات الالكتروليتية التي لها استخدام واسع في المجال الصناعي . في هذه الدراسة تم اجراء عملية السلفنة لاحدى مخلفات البولي ستايرين باستخدام حامض الكبريتيك كعامل سلفنة وتم حساب درجة السلفنة , ومن ناحية اخرى تم تحضير مواد مركبة من البوليمر المحضر باستخدام نوعين من المائتات (النانوسليكا واسود الكربون) وحضرت نماذج لدراسة الخواص الميكانيكية والحرارية .ايضا تم تحضير بوليمر شبكي التداخل جديد من (SPS) مع راتنج الفينولي (نوع نوفولاك) , وشخصت البوليمرات باستخدام تقنية ال (FTIR) .اشارت نتائج الخواص الميكانيكية الى ان قيم قوة الشد ومعامل يونك تزداد بزيادة نسبة المائتات المضافة , وكذلك لوحظ ان قابلية امتصاص البوليمر المسلفن للماء اعلى منه للبولي ستايرين بسبب وجود مجاميع السلفونيك .تم تقييم الخواص الحرارية للمواد المحضرة باستخدام تقنيتي التحليل الحراري الوزني (TGA) والمسح التفاضلي المسعري (DSC) واوضحت النتائج بان درجة التفكك الحراري لتلك المواد هي اعلى من 300 درجة مئوية .

