PERIÓDICO TCHÊ QUÍMICA
ARTIGO ORIGINAL

INVESTIGAÇÃO DAS CARACTERÍSTICAS DO POLIFURFURAL PREPARADO ATRAVÉS DO PROCESSO DE POLIMERIZAÇÃO

CHARACTERISTICS INVESTIGATION OF PREPARED PLOYFURFURAL THROUGH THE POLYMERIZATION PROCESS

[1]Hind Mahdi Saleh
[1]Department of Chemistry, College of Science, University of Misan, Misan, Iraq.

** Correspondence author*
e-mail: hind1980@uomisan.edu.iq

Received 12 November 2019; received in revised form 14 January 2020; accepted 20 January 2020

RESUMO

Recentemente, o furfural é o produto químico industrial mais comumente produzido, devido à sua produção ser muito flexível. Neste estudo, o furfural foi convertido em poli-furfural através do processo de polimerização em meio ácido. Algumas características mecânicas e térmicas foram investigadas para o polímero viscoso. O polímero foi caracterizado pelo espectro de FTIR da resina furana preparada neste estudo, mostrando várias bandas devido ao anel furano, que aparece em (3116,1508,1149 e 735) cm^{-1}. Além disso, o espectro mostrou uma banda a 3450 cm^{-1} devido ao grupo hidroxil e uma banda a 1714 cm^{-1} devido ao grupo carbonil que foram gerados por clivagem do anel furano sob polimerização no meio ácido. A análise de CHN, condição de cura do polímero, foi seguida pela técnica DSC, e a temperatura de transição vítrea da resina curada foi estimada (246 °C). A análise termogravimétrica (TGA) fornece uma boa abordagem para acelerar o teste de vida útil do polímero que monitora as alterações de peso nos materiais. O TGA e o DSC da resina furana são compostos após aquecimento a 250 °C, 300 °C, 350 °C e 400 °C; teve uma etapa de decomposição. Por outro lado, o tratamento térmico do polímero foi realizado em diferentes temperaturas e tempos, e vários parâmetros térmicos foram determinados

Palavras-chave: *condição de reação, Perda por ignição, síntese, viscosidade, teste de tração, cargas*

ABSTRACT

Recently, furfural is the most commonly produced industrial chemical because of its production is very flexible. In this study, furfural was converted into ploy furfural through polymerization process under acidic medium. Some mechanical and thermal characteristics were investigated for the viscous polymer. The polymer was characterized by the FTIR spectrum of the furan resin prepared in this study showed several bands due to the furan ring, which appears at (3116,1508,1149 and 735) cm^{-1}. In addition, the spectrum showed a band at 3450 cm^{-1} due to the hydroxyl group and band at 1714 cm^{-1} due to carbonyl group which was generated by cleavage the furan ring under polymerization at the acid medium.The CHN analysis, curing condition of the polymer, was followed by the DSC technique, and the glass transition temperature of cured resin was an estimate (246 °C). Thermo Gravimetric Analysis (TGA) provides a good approach to accelerate the polymer lifetime testing that monitors weight changes in materials. The TGA and DSC of the furan resin compounds after heating at 250 °C, 300 °C, 350 °C, and 400 °C; had one decomposition step. On the other hand, thermal treatment of polymer was done at different temperatures and times, and several thermal parameters were determined.

Keywords: *reaction condition, Loss on Ignition, synthesizing, viscosity, tensile testing*

الخلاصة

في الآونة الأخيرة، الفورفورال هو المادة الكيميائية الصناعية الأكثر شيوعًا التي يتم إنتاجها بسبب مرونة إنتاجها. في هذه الدراسة، تم تحويل الفورفورال إلى فورفورال متعدد من خلال عملية البلمرة في وسط حمضي. تم التحقيق في بعض الخصائص الميكانيكية والحرارية للبوليمر اللزج. تميز البوليمر بطيف FTIR لراتنج الفوران الذي تم تحضيره في هذه الدراسة والذي أظهر عدة نطاقات بسبب حلقة الفوران، والتي تظهر عند (3116،1508،1149 و735) سم-1. بالإضافة إلى ذلك، أظهر الطيف نطاقًا عند 3450 سم-1 بسبب مجموعة الهيدروكسيل والنطاق عند 1714 سم-1 بسبب مجموعة الكربونيل والتي تم إنشاؤها عن طريق انشطار حلقة الفوران تحت البلمرة في الوسط الحمضي. تم تتبع تحليل CHN، وحالة المعالجة للبوليمر، بتقنية DSC، ودرجة حرارة التحول الزجاجي للراتنج المعالج كانت تقديرية (246 درجة مئوية). يوفر التحليل الوزني الحراري (TGA) نهجًا جيدًا لتسريع اختبار عمر البوليمر الذي يراقب التغيرات في الوزن في المواد. TGA و DSC لمركبات راتنج الفوران بعد التسخين عند 250 درجة مئوية، 300 درجة مئوية، 350 درجة مئوية، و400 درجة مئوية؛ كان لديه خطوة تحلل واحدة. من ناحية أخرى، تمت معالجة البوليمر حراريًا في درجات حرارة وأوقات مختلفة، وتم تحديد العديد من المعلمات الحرارية.

DOI: 10.52571/PTQ.v17.n34.2020.130_P34_pgs_112_124.pdf

عند)6113، 1051، 1111، و 560(سم-1. الاضافة الى، أظهر الطيف حزمة عند 6105سم-1 بسبب مجموعة الهيدروكسيل وحزمة عند 1511سم-1 بسبب مجموعة الكربونيل التي أنشئت بواسطة انشطار حلقة الفيوران تحت البلمرة في الوسط الحامضي. تحليل CHN، ظروف معالجة البوليمر أُتبعت بتقنية DSC وكانت درجة حرارة التحول الزجاجي للراتنج المعالج)613 درجة مئوية(. يفيد تحليل الجاذبية الحرارية (TGA) طريقة جيدة لتسريع اختبار عمل البوليمر لمراقبة التغيرات الوزنية في المواد. إن TGA و DSC لمركبات راتنج الفيوران بعد التسخين عند 605 درجة مئوية ، 655 درجة مئوية ، 605 درجة مئوية ، و 155 درجة مئوية ؛ خطوة تحلل واحدة. من ناحية أخرى تمت المعالجة الحرارية للبوليمر في درجات حرارة وأوقات مختلفة وتم تحديد العديد من المعلمات الحرارية.

الكلمات المفتاحية: ظروف التفاعل، فقدان الاشتعال، الفقد في الاشتعال، التركيب، اللزوجة، اختبار الشد.

1. INTRODUCTION

Furfural has captured extreme attention as one of the most important organic compounds, and it is exclusively produced from lignocelluloses biomass by dehydrating pentose (Dashtban *et al*., 2012; Ahmad *et al*., 1995; Feather *et al*., 1972; Win, 2005), Lignocelluloses biomass has drawn a lot of attention because of its high carbohydrate content. It contains approximately 42-45 % cellulose, 23-36 % hemicelluloses and 22-28 % lignin (Orlov *et al*., 2017; Cai, *et al.,* 2017). Furfural can be produced by a one-step or a two-step process. In a one-step process, pentosan is hydrolyzed into xylose and then dehydrated into furfural simultaneously. However, in the two-step process, hydrolysis of pentosan occurs under mild conditions followed by dehydration of xylose into furfural (Poddar *et al*., 2014; Agirrezabal-Telleria *et al*., 2014a).The commercial production of furfural is by the acid hydrolysis of pentose polysaccharides from non-food residues of food crops and wood wastes from fibrous residues of food crops, in terms of industrial production batch or continuous reactors has been used for that. The reaction condition for furfural production is 3% acid solution to lignocelluloses mass ratio of between 2:1 and 3:1 at 170 -185 ^oC, the maximum furfural yields are within (45 - 50) % (Barbosa *et al*., 2014; Agirrezabal-Telleria, 2014b) . The schematic for this process is presented in Figure 1.

Furfural (furan–2 – carboxaldehyde) is a viscous, colorless liquid with a boiling point of about 160 °C. It has a pleasant aromatic Oder and turns dark, brown, or black when exposed to air (Morone *et al*., 2005; Lakra *et al*., 2014), furfural is natural precursor to arrange furan – based chemicals and solvents (Lakra *et al*., 2014) as it is illustrated schematically in Figure 2. Additionally, furfural is the sleeping beauty of all the bio-renewable chemicals, bioplastics, and polymers, it as well as its derivatives, has been utilized in a wide range of applications, for instance, plastic, pharmaceuticals, and agrochemicals (Tsanaktsis *et al*.,2015; Aït-Aissa and Aïder, 2014). Furthermore, it can be used for the synthesis of particular polymers depending on the chemistry of the furan ring. Moreover, furfural alcohol is most of these derivatives that have been used in reinforced carbon-carbon composites (Aït-Aissa and Aïder, 2014; Mascal, 2015; Fleute-Schlachter *et al*., 2015; Chen *et al*., 2014). The aim of the proposed research was to synthesizing poly furfural using acid polymerization process, also studying some physical and thermal features of the prepared polymer. Furthermore, the new composite material was synthesized by using industrial waste silica and the polymer as a binder material.

2. MATERIALS AND METHODS

2.1. Materials

Furfural 98%, absolute ethanol, and sulphuric acid 98% were purchased from Fluka company, whereas other chemicals such as acetone, methanol were obtained from Merck company. Silica reinforced filler was obtained from fluorescence tube waste as powder form with a density of 2.33 kg/m^3 and a mesh size of 200 micrometers.

2.2. Viscosity Measurements

The viscosity of the furan resin prepared in this study was measured using brook-field rotary viscometer type Alpha series code V 100002 with a spindle at 252 °C. An average value of three replicated measurements was reported (Alshawi and Hanoosh, 2019).

2.3. Fourier Transform Infra-Red (FTIR) spectroscopy

The FTIR spectra of cured resin were performed using Shimadzu FTIR – 84005, and each spectrum was recorded in a frequency range of 500 – 4000 cm using potassium bromide (KBr) disc. The KBr was previously oven-dried at

300 °C to lessen the interference of water (Bobrowski *et al*., 2018).

2.4. Differential Scanning Calorimetry (DSC)

DSC measurements were conducted by Shimadzu DSC-60. Dynamic scans were performed within the temperature range (25 – 350) °C at a constant heating rate of 10°C /min. Under the N_2 atmosphere at a flow rate of 20 ml/min, about 10 – 15 mg of uncured resin was used in an aluminum crucible (Abdul Razak *et al*., 2016).

2.5. Thermo Gravimetric Analysis (TGA)

TGA measurements were computed using TGA Q SOV 20-13. The dynamic scan was measure within temperature range (25 – 700) °C, at a constant heating rate of 10 °C/min. All measurements have been performed under the nitrogen atmosphere at a flow rate of 30 ml/min (Garay *et al*., 2011).

2.6. Resin Solid Content

The percentage of solid resin content (s) was calculated by the Equation 1:

%5 = S1/S0 x 100% (Eq. 1)

Where S0, and S1 are the weight of the resin before and after dried 3 hours at 105 °C, respectively (Lin *et al*., 2014).

2.7. Samples Preparation for Primitive Mechanical Test

Amounts of silica filler and resin were weighted by using a digital scale and poured into mixer Shimadzu were blended for 1 minute, then an amount of generated mixture was put into molds for tensile, compression, and harden test. The samples were subjected to the curing process before they were extracted from the molds. A total of three samples were prepared for numbering, tensile testing of the sample in a shape of dumbly was carried out after 24 hours of the molding and curing processes. Tensile force and elongation were applied until the samples were broken into two parts for compressive strength test samples, for the test were cylindrical shape with 50 mm diameter and 50 mm height. Each sample was pressed at a load of 1200 N until the breakdown occurred (Arbaoui *et al*., 2016).

Synthesizing of Poly Furfural Resin

Furan resin was prepared by using 50g of freshly distilled furfural in 250 ml round flask fitted with a reflux condenser, mechanical stirrer, and a digital thermometer. To furfural monomer 5ml of Sulfuric Acid (2M) was added and heated at 100 °C with a suitable stirrer until a brown viscose material was obtained (after 2.5 hours) then this resin was soluble in hexane and washed several times with water in order to remove untreated monomer and acid residue. Finally, a hexane layer was separated and dried under magnesium sulfate then it filtrated and evaporated to provide a brown viscose material. Sample from the produced furan resin will be subjected to DSC study (Choura *et al*., 1996).

2.8. Final Mechanical Test

The silica and furan resin amounts were weighted by using a digital scale (different weight ratio of waste industries silica 10,20,30 and 40% with furan resin as a binder. They were mixed for 3 minutes, then the amount of the generated mixture was put into molds for tensile and compression. Finally, the sample was cured at 130 °C for about 4 hours and post-cure at 150 °C for about 3 hours (Arbaoui *et al*., 2016).

2.9. Curing Process of Poly Furfural

The curing process of the prepared furan resin was achieved by heating, and firstly the resin was heated at 100 °C for 5 hours then at 120 °C for 3 hours. Finally, post-cure at 150 °C for 3 hours, a sample from the processed resin will be taken to the TGA study (Tasan and Kaynak, 2004).

2.10. Weight Loss Investigation Test

For study purposes, the cured poly furfural resin was subjected to the heating test under temperatures of 200 °C, 250 °C, 300 °C, 350 °C, and 400 °C respectively. The test was conducted to investigate the influence of heating on the loss of weight of the processed resin. The quantitative weight of the cured resin was put in the oven at a constant time (1 hour), then weight residue was measured (Dhinakaran *et al*., 2010).

2.11. Loss on Ignition (LOI)

Loss on ignition was performed in order to determine the presence of organic or other gas-

forming materials in the silica furane resin. A 10 g of sample was weighted and put into a crucible, and then it was heated at 1000 °C for 30 minutes. After that, it was taken out of the oven and placed immediately in desiccators. The sample was weighted for a second time, LOI was measured according to the initial weight minus final weight and divided by the initial weight in unit gram.

3. RESULTS AND DISCUSSION:

3.1. Reaction scheme

Schematically as demonstrated below, furan resin was obtained by the polymerization process of freshly distilled furfural under acid medium, leading to the formation of brown viscose material with some physical properties presented in Table 1.

Then this polymer after heating over than 100 °C cross-linked polymer may be obtained as shown schematically is infusible and non-soluble in most organic solvents.

3.2. Mechanical Study.

Tensile testing of the processed samples in a shape of dumbbell was performed after 24 hours of the curing process, and the samples were subjected to clamp between attachments, as shown in Figure 3. The tensile force was applied until the samples were broken into two parts. Table 2 shows the results for tensile strength, elongation, compressive strength and hardness, and these values were averaged from 3 samples for each set of percentage ratio except for hardness test

It was observed that the value of tensile strength for cured poly furfural alone was 7.8 kg/cm2, while this value increased consistently with an additional percentage of waste silica. It was 21 kg/cm^2 for a 40% weight percentage of silica filler. In the case of elongation, the value will be reduced with increasing fillers due to the most brittle behavior of polymers. While a compressive strength test was performed using a universal testing machine (UTM), samples were cylindrical shapes with 50 mm diameter and 50 mm height, as shown in Figure 4. Each sample was placed between compression holders until it was broken. The result was displayed from the aided software. The obtained results have shown that the compressive strength of the composite increased consistently with a rising percentage of silica filler due to the more bonded between silica and furan resin after curing.

3.3. FTIR-Study

The FTIR spectroscopy is an important and common technique to identify the most functional group. So, the FTIR spectrum of the furan resin prepared in this study showed several bands due to the furan ring, which appeared at (3116,1508,1149 and 735) cm^{-1}. Also, band at 3450 cm^{-1} due to the presence of hydroxyl group and band at 1714 cm^{-1} due to carbonyl group presence through cleavage of some furan ring under polymerization at acid medium. Figure 5 shows the spectrum of uncured furan resin.

3.4. Heat treatment of cured poly furfural resin

Table 3 shows the weightless of the cured polymer at different temperatures (250, 300, 350, 400 °C) within a range of times. According to Table 3, the maximum weight loss was 46% at 400 °C when the cured polymer was held for 1 hour at this temperature, then this percentage decreased to 10% at the same temperature but at 6 hours holding. That means these types of polymers have a good char yield due to the presence of fused aromatic structure after heating.

3.5. TGA and DSC study

Polymers can be employed to predict the lifetime of 16 products; Thermo Gravimetric Analysis (TGA) provides a good approach to accelerate the polymer lifetime testing that monitors weight changes in materials. Figures (8 -11) and Table 4 show the TGA and DSC of the furan resin compounds after heating at 250 °C, 300 °C, 350 °C and 400 °C, furan resin compounds had one decomposition step. The percentages of the char content of cured resin at 600 C0 were more than 35%, which indicates that those materials can be used as thermal insulators. Furthermore, the glass transition temperature (Tg) of the cured furan resin was 246 °C as explained in figure.7 that also may indicate more cross-linking occur after curing. Figure 6 shows the DSC thermogram of uncured furan resin, and the uncured sample presented an exothermic peak may due to the reaction among hydroxyl groups in polymer chains. The optimum curing temperature was around 125 °C. Also, the values of Tg are directly proportional to

temperatures of the resin, which attribute to more crosslink formation (Li *et al*., 2017; Saha *et al*., 2014; Kandola *et al*., 2015; Celikbag *et al.,* 2017). Table 5 illustrates that.

3.6. Loss on Ignition (LOI)

Table 6 shows the LOI results for furan resin and furan resin-bonded silica, LOI obtained was less than 2 %, and that confirms the distinct values of LOI according to (Anthonia and Philip, 2015; Nastac *et al*., 2016; Sun *et al*., 2019). LOI must be kept below 3%, and the results demonstrate that LOI is inversely proportional to the added percentage of silica.

4. CONCLUSIONS:

The proposed research has utilized the polymerization process to convert furfural to ploy furfural under acidic medium, and particular mechanical and thermal characteristics were investigated. The tensile strength for processed poly furfural alone was 7.8 kg/cm^2, and it was directly proportional to added percentages of waste silica, which was used as a filler. In terms of thermal features, the thermal decomposition of prepared polymers showed that the char content of cured resin was more than 35% at 600 ^{o}C, the thermal study was implemented within a considerable range of temperatures.

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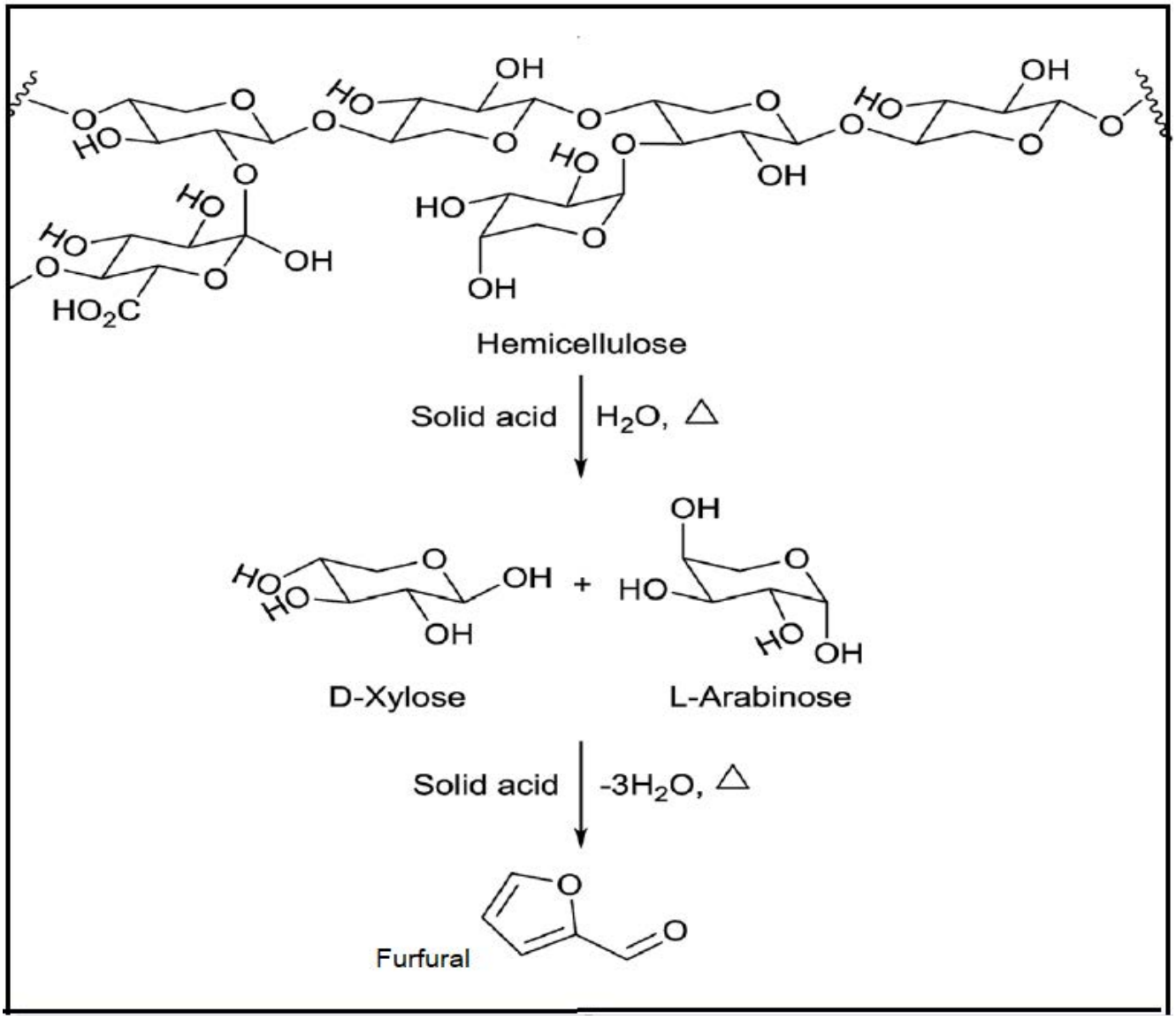

Figure 1. The reaction for the acid-catalyzed the conversion of hemicellulose into furfural

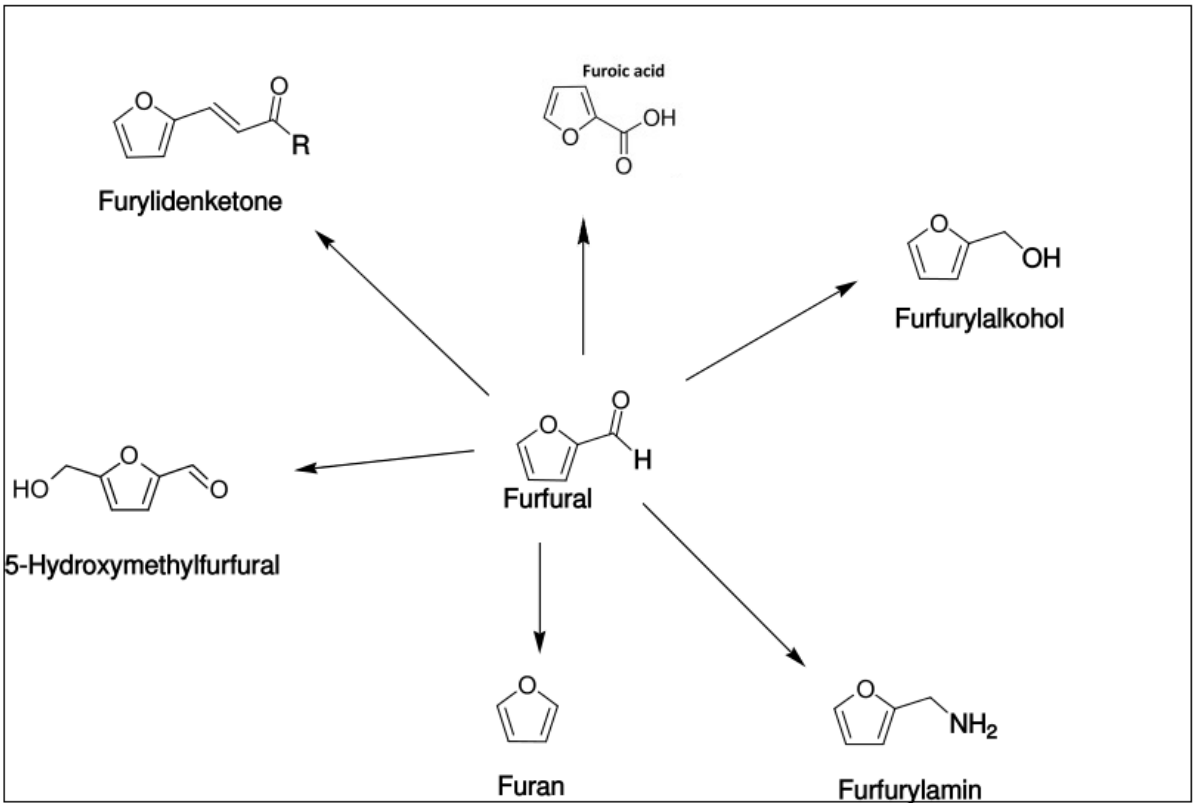

Figure 2. Conversion of furfural into some important chemical compounds

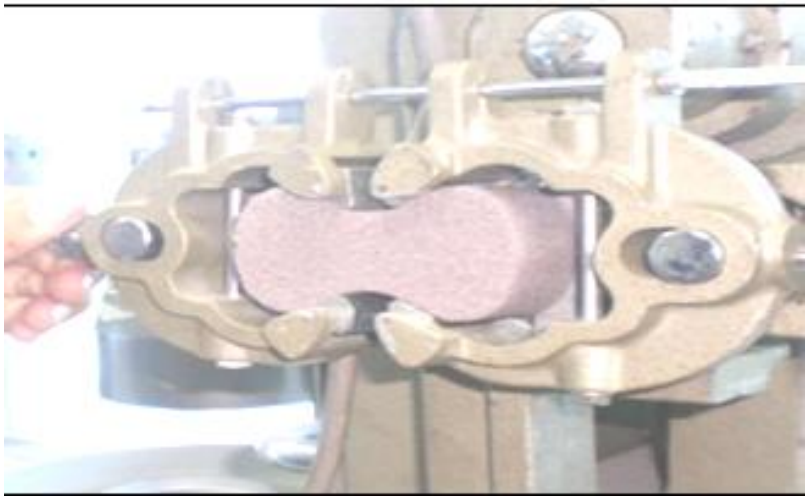

Figure 3. The sample was clamped between the attachments of the universal silica strength machine

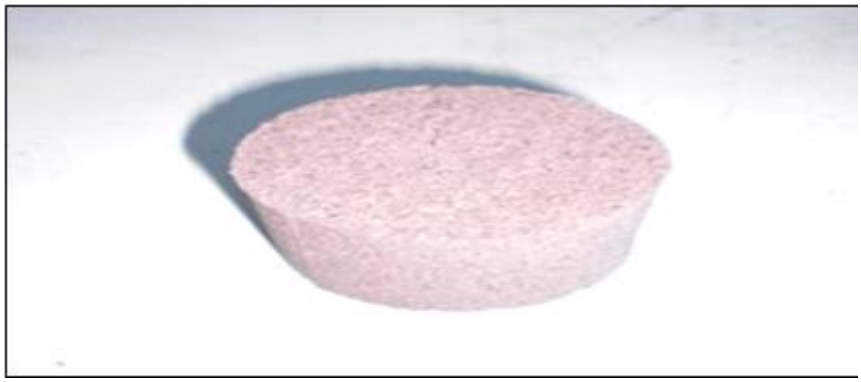

Figure 4. Sample for compressive strength.

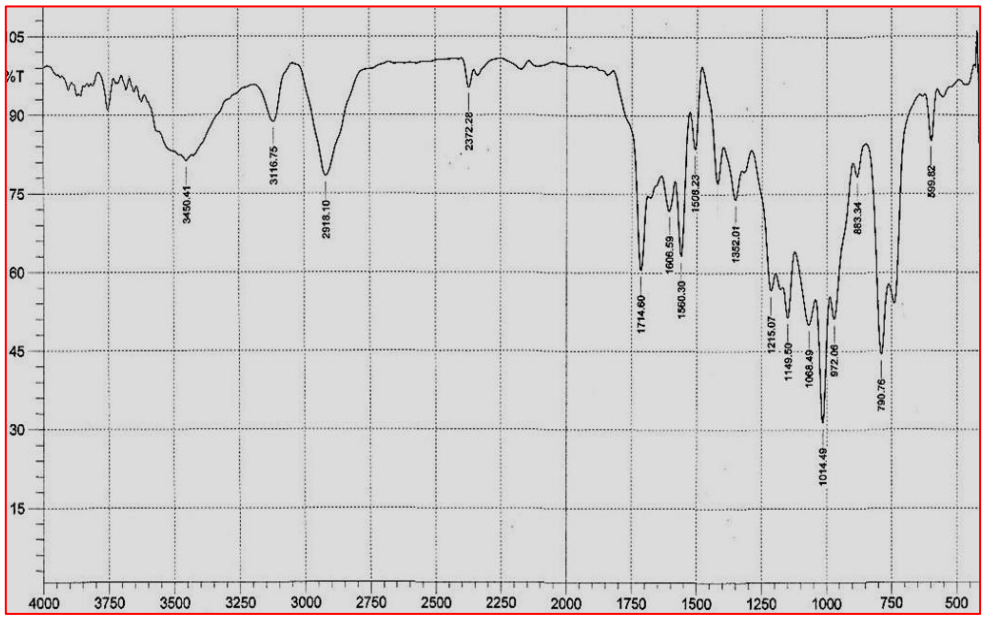

Figure 5. FTIR spectrum of polyfurfural

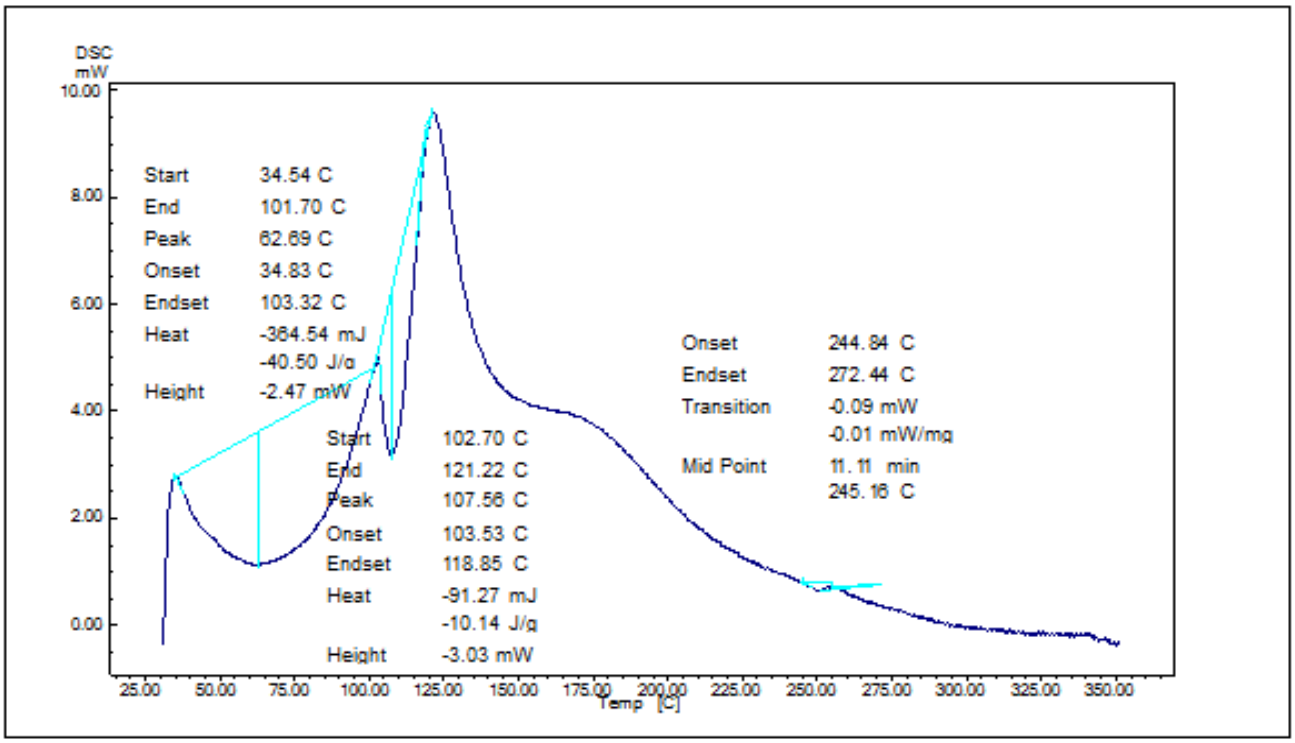

Figure 6. DSC thermogram of furan resin

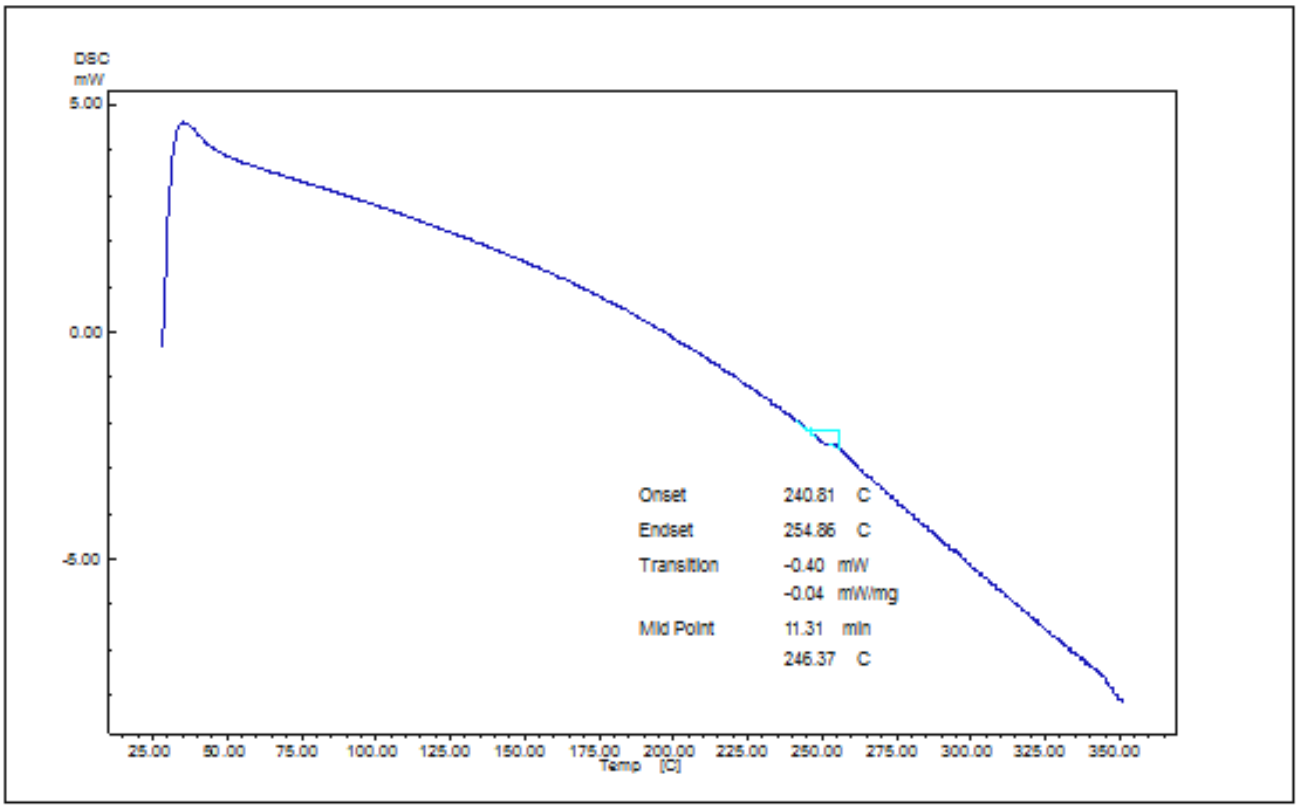

Figure 7. DSC thermogram of Cured poly furfural.

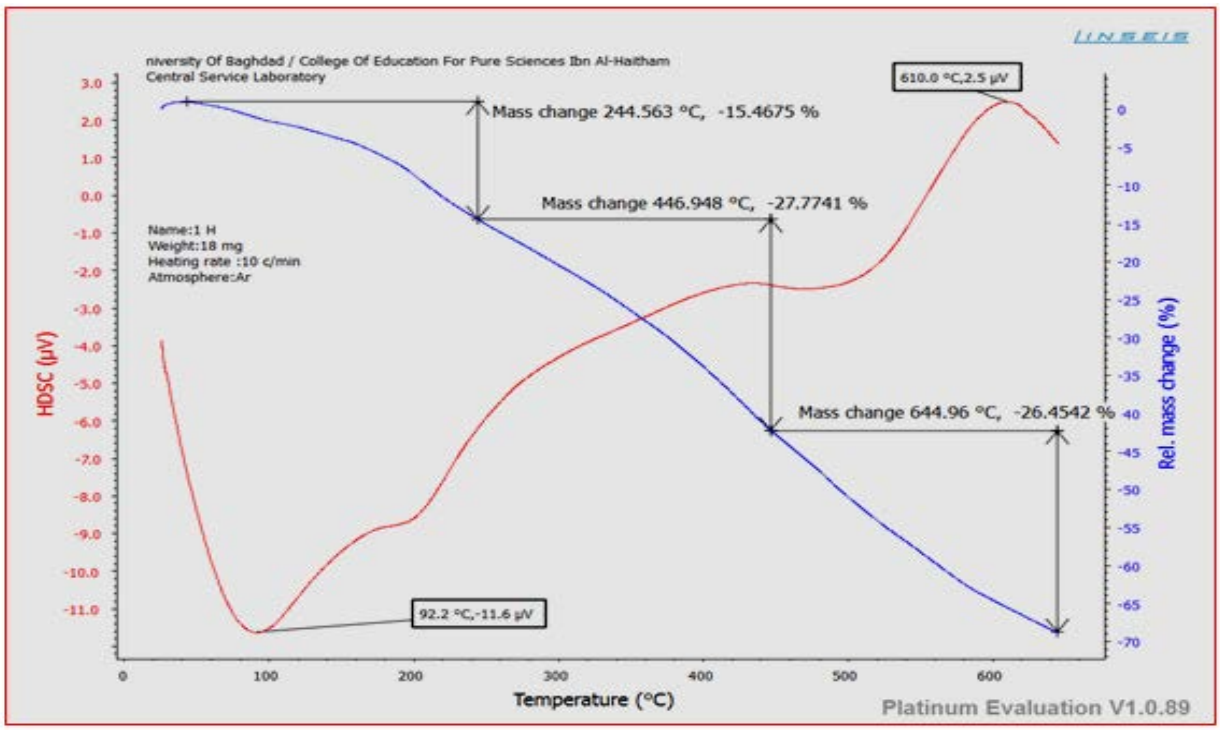

Figure 8. TGA and DSC thermogram of furan resin after heat treatment at 250^{0}C

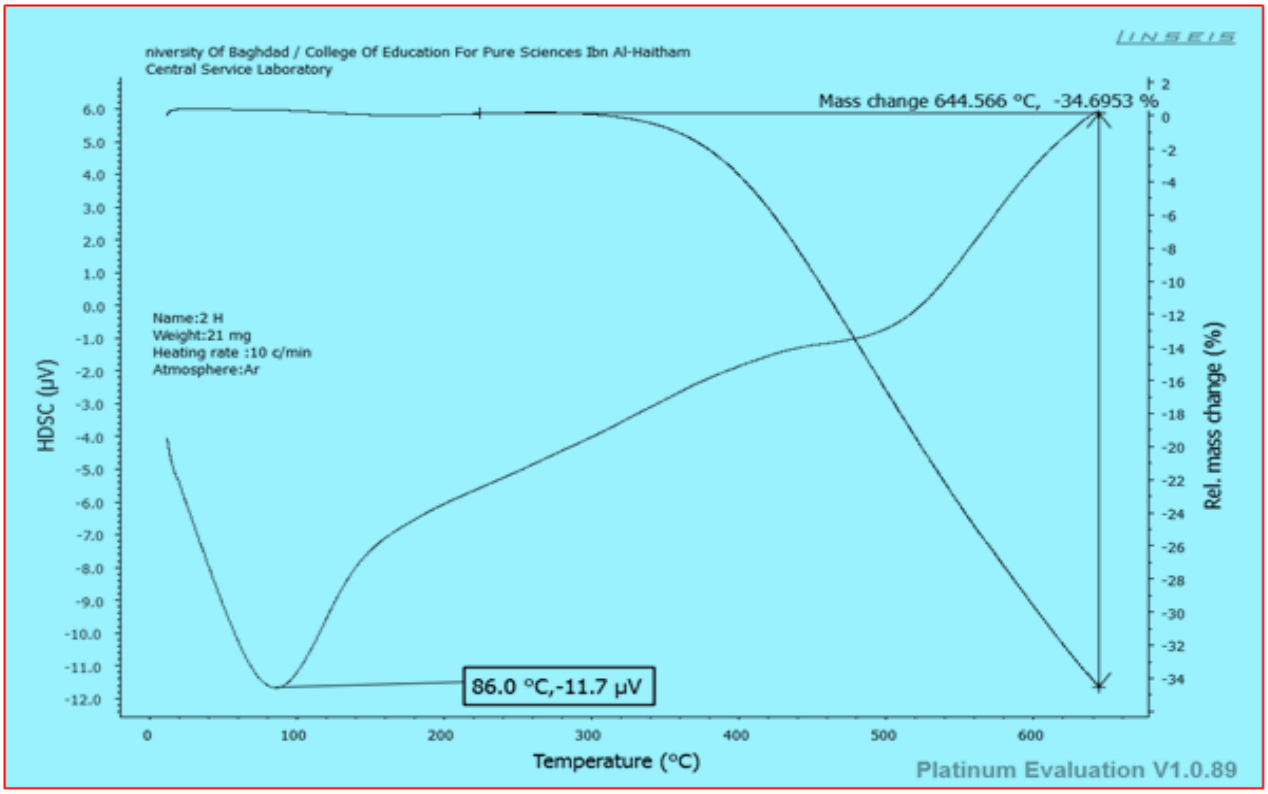

Figure 9. TGA and DSC thermogram of furan resin after heat treatment at 300 ^{0}C

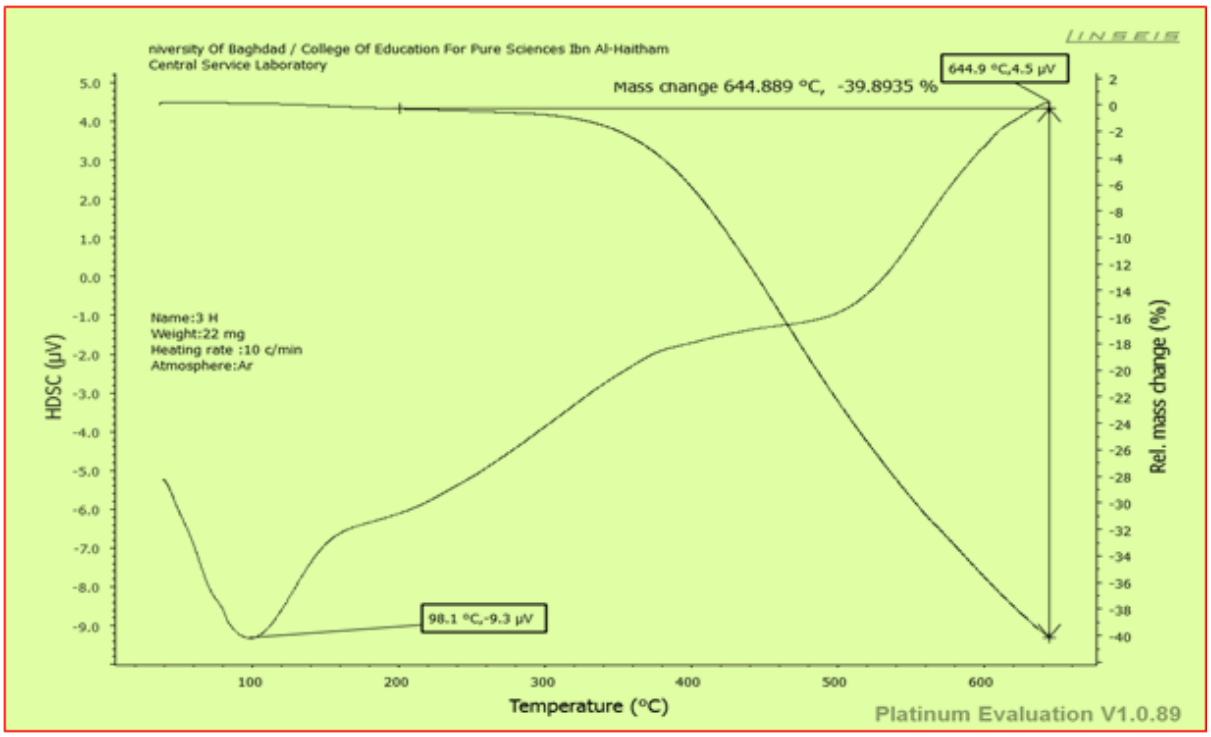

Figure 10. TGA and DSC thermogram of furan resin after heat treatment at 350 ^{0}C

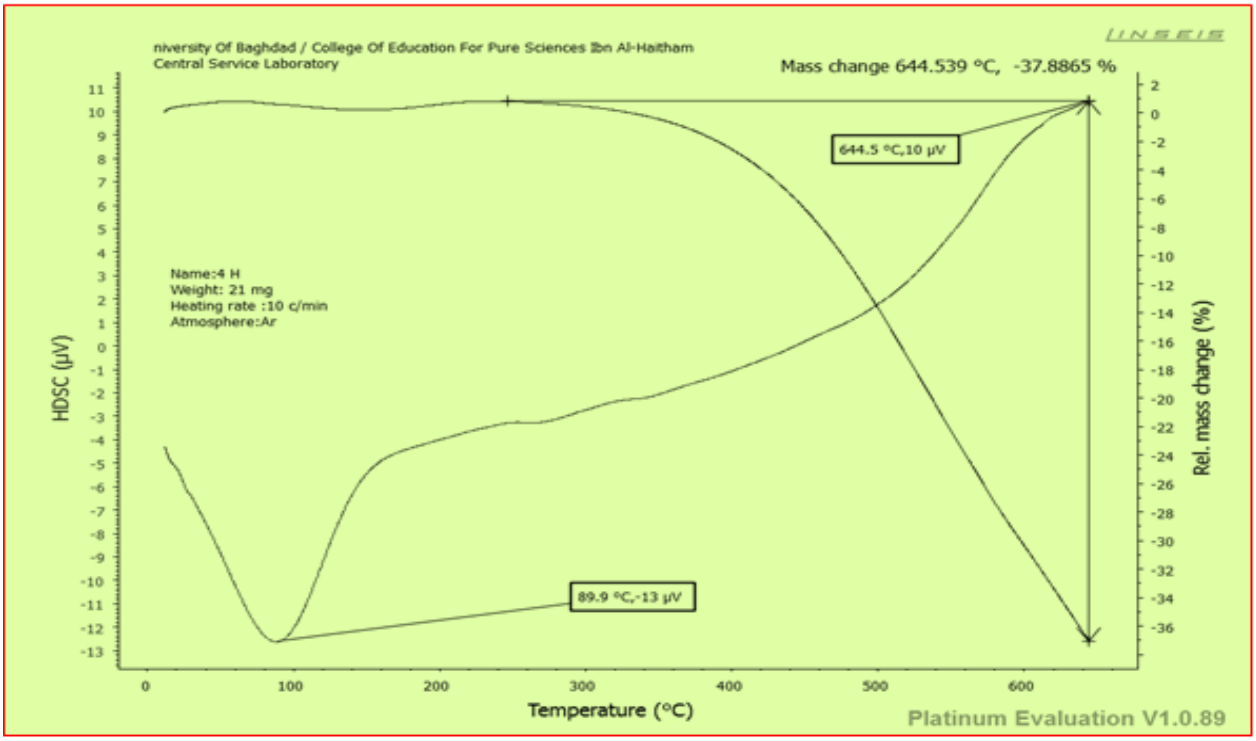

Figure 11. TGA and DSC thermogram of furan resin after heat treatment at 400 ^{0}C

Table 1: Some physical properties of uncured furan resin

Gel time min. (loss of fluidity) at 100 ^{0}C	Viscosity (mpa.s) at 25 ^{0}C	Dry solid content % at 105 ^{0}C. 3hrs
21 min	327	51.32

Table 2. Tensile strength, elongation, compressive strength and hardness of silica casting mold

Sample No	Tensile strength Kg/cm^2	Elongation %	Compressive strength Kg/cm^2	Hardness Shor D
Cured furan resin alone	7.80	2.1	14.72	63
10% silica	10.32	1.9	17.32	69
20%	12.73	1.8	20.11	71
30%	17.52	1.5	24.32	75
40%	21.00	1.4	28.56	77

Table 3: Weight loss of furan resin at different temperatures and times

Temperature ^{0}C	Weight loss %					
	1h	2h	3h	4h	5h	6h
250	6.38	0.49	0.42	0.24	0.19	0.08
300	10.56	5.57	1.91	0.41	0.37	0.23
350	14.16	10.08	4.79	2.32	2.06	1.22
400	46.45	28.15	27.94	23.46	12.17	10.74

Table 4: Thermal parameter obtained from TGA /DSC thermogram of cured poly furfural after heat treatment at different temperatures

Resin No.	Decomposition temperature ^{o}C	Char content at 600 ^{o}C %	Temp. of 50% weight loss ^{o}C
1H	244	35	495
2H	380	70	>600
3H	380	64	>600
4H	450	70	>600

Table 5. Tg value of cured poly furfural obtained from DSC thermogram

Resin No.	**Tg ^{0}C**
Cured polymer alone.	246
1H	200
2H	270
3H	256
4H	254

Table 6. LOI values for furan resin and its composites

Resin. NO.	**LOI %**
Cured polymer alone.	1.94
10 % silica	1.83
20 %	1.54
30 %	1.42
40 %	1.27