

Analytical and Thermal Studies Of New Polyurethane Synthesis from Ziziphus SPINA-CHRISTI Leaves

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Abstract

This research aimed to alcoholic extract of leaves Ziziphus Spina –Chris and isolate the extract material as powder, then this isolated powder was converted to new polyurethane through condensation polymerization by reaction with methylene diphenyl diisocyanate . The Fourier Transform Infra-Red analysis was carried out to identify the functional groups present in the extracted material and prepared polymer. This polymer used to remove some metal ions from the mixing solution, and the result showing that the polymer uptake of zinc ion was 99 %,while the chromium ion was 97 % after 7 hours. Also the recovery of these ions from the polymer was done using column method and the result shown that the zinc ion removed at 89% using 1 Normality nitric acid solution , while the chromium ion remove at 79 % using 3 Normality nitric acid solution. On the other hand the thermal stability of the prepared polymer was evaluated using (Thermogravimetric analysis and Differential Scanning Calarometry) techniques.

Keyword: ZIZIPHUS SPINA-CHRISTI leaves, polyurethane , ,TGA ,DSC

Introduction

Buckthorn family has about 40 species of spiny shrubs and small trees , one of this genus is ZIZIPHUS SPINA-CHRISTI Buckthron found in the warm-temperate and subtropical regions throughout the word (1). The leaves are entire, with three prominent basal veins, some species leaves are deciduous, others evergreen. The flowers are small, inconspicuous yellow-green. The fruit is edible , yellow-brown, red, globose or oblong, have asweet and sugary taste. There are many types of Buckthorn one of these called Sea buckthorn (*Hippophae rhamnoides L.*) belongs to the genus *Hippophae*, Sea buckthorn has a warm, sour taste. In ancient times, doctors commonly use sea buckthorn as traditional Chinese medicine to treat diseases. Sea buckthorn contains abundant nutrients and biologically active ingredients. Sea buckthorn plays an important role in promoting metabolism and has ant fatigue,

anticaking, ant atherosclerosis, ant radiation, and ant scurvy properties Sea buckthorn has wide distribution and variety. It has become an important raw material for domestic and foreign drugs, health food, and cosmetics. Buckthorn is an edible plant resource that has long been the source of income of the People's Republic of China Pharmacopoeia, Various pharmacological activities such as cytoprotective, anti-stress, immunomodulatory, hepatoprotective, radioprotective, anti-atherogenic, anti-tumor, anti-microbial and tissue regeneration have been reported. (2, 3).

The chemical structures in the sea buckthorn pulp and seed are bioactive constituents, which are good for our health and have antioxidants, hepatoprotective, and immunomodulatory properties. There are five essential compounds in the extract of sea buckthorn (4, 5). The fruits and leaves of buckthorn have been demonstrated their important nutritional and medicinal values (5). So a high-performance liquid chromatography (HPLC) method based on the distribution and relative amount of seven flavonoids was established for the food quality evaluation of buckthorn leaves(6, 7).

ZIZIPHUS SPINA called Seder or Cidir is an old plant that grows in tropical and subtropical regions especially in middle east. Its extracts are important uses in the middle east and south east of Asia for a long time with huge amounts (8) Cidir leaves extract to decrease the skin sensitivity both in normal and effectively in atopic patients. The mean stimulus required to induce each one of cutaneous sensation (tingling, pricking, itching and pain) was higher after the application of the cidir leaves extract. Cidir leaves found good treatment for skin sensitivity and used as a cooling agent for eczema and pruritus (9).

Cidir can be reduced heavy metals in water resources which important environmental problems. Heavy metals have hazardous effects on human health. In the study was investigated the adsorption of Cd^{+2} from aqueous solution using adsorbents of cedar leaves by batch experiments at the variation of impressive parameters such as pH, contact time, and adsorbent amount. The results showed indicated the high ability of cidir leaves in adsorption of Cd^{+2} from aqueous solution(10).

Phytochemical profiling of ZIZIPHUS SPINA leaves led to the characterization of 10 dammarane-type saponins and 12 phenolic compounds(11). These polyphenolic compounds in the (leaves) juice were 510.00 and 722.00 ppm . Aliquots of the concentrated Cidir juice (leaves), represent 200, 400, 800 and 1600ppm and studied hydroxyl toluene (200ppm) . The data of the mentioned measurements indicate that the conduct of Cidir juice (leaves) did not cause any changes in kidney and liver functions(12).

Agricultural by products have unique chemical compositions a more efficient and feasible option for pollutant removal. These by-products are mainly composed of lingo cellulosic materials that consist of three main structural components, which are lignin, cellulose, and hemicelluloses also Agriculture wastes, contains lipids, proteins, simple sugars, water, hydrocarbons, and starch. Other polar functional groups of lignin may be also included, such as alcohols, aldehydes, ketones, carboxylic, phenolic, and other groups. The seeds of *Zizyphus spina* contain Moisture, Ash,

protein, and fiber, with little fat around and a moderate amount of carbohydrates include sucrose, fructose, glucose(13, 14).

In another study, activated carbon was prepared from Ziziphus seeds to remove Mn^{+2} from aqueous solutions. The chemical characterization of activated carbon on the surface was studied by Fourier's variable infrared spectrometry. Parameters such as pH, primary metal ion concentration and temperature on the seed absorption performance of Mn^{+2} ions were examined in batch method. Found good adsorption capacity Mn^{+2} ion from its solution(15).

In other research deals with the utilization of agriculture waste biomass of cidir seed as natural cation exchanger for removal of cationic pollutant from aqueous solution. Methylene blue dye method was used to determine the cation exchange capacity of the stone it highest dye sorption capacity, with high vacancy(16).

Extraction of polyphenols from buckthorn leaves. The polyphenolic extracts were analyzed in order to determine the total phenolic content by high-performance liquid chromatography (HPLC) (17). The polyphenol can benefit from its interaction with other material

The literature does not show research about preparation of foam from Buckthorn leaves with analytical study

2. Experiment

2.1- Chemicals

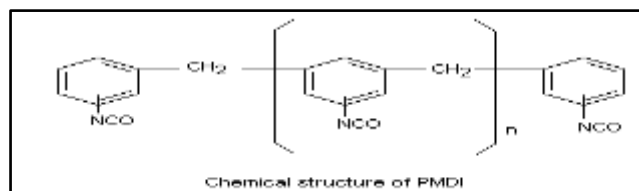
Ethanol, Acetone, triethyl amine, were supplied from merck company, tin octanoate as catalyst was supplied from Fluka company, while polymeric methylene diphenyl diisocyanate (PMDI) was supplied from DOW Company, it was used without any extra treatment with the following specification

Density : 1.23 gm/ cm³

NCO content: 31 %

Functionality : 2.7

Viscosity: 200 mp.s



n= 0- 4

While Leaves of ZIZIPHUS SPINA were collected from the garden city of Basrah , leaves were washed with distill water several times and then with sterile water, left to dry at room temperature grind using electrical blending mill to get dry powder. shown.

2.2 Instruments

2.2.1. Fourier transform infrared (FT-IR) Spectroscopy test:

The FTIR spectra of cured phenolic resins were performed in Shimadzu, FTIR-8400S. Each spectrum was recorded in a frequency range of 400 – 4000 cm⁻¹ using a potassium bromide (KBr) disc. The KBr was previously oven-dried at 300 °C to reduce the interference of water.

2.2.2. Differential Scanning Calorimetry (DSC) test:

DSC measurement was conducted on Shimadzu DSC-60. Dynamic scans were conducted in a temperature range 25-350 °C, at a constant heating rate of 10 °C/min, under a nitrogen atmosphere at a flux rate of 20 mL/min. About 10-15 mg of uncured resin was used in an aluminum crucible.

2.2.3. Thermogravimetric Analysis (TGA):

TGA measurement was evaluated on TGA Q50 V20 Dynamic scan were measure in a temperature range 25-700 °C, at a constant heating rate 50 °C/min, under a nitrogen atmosphere at flow rate 30 ml/min.

2.2.4. Swelling test :

The swelling ratio of 10 x 10 x 1 mm specimen was measured at laboratory temperture .Sample weight was measured every ten minutes within the first hour , every 20 minutes within the second hour and then every hour for the next 4 hours with the last measurement performed after 1 day since the beginning of the swelling experiment. Water uptake was calculated according to number of the equation below:

$$\text{Water uptake (\%)} = \frac{W_s - W_d}{W_d} \cdot 100 \quad (1)$$

Where w_s is the weight of the swollen sample at the given time and w_d is the weight of dry sample. From every sample three pieces were measured at each time point .

2.3 Preparation Methods

2.3.1. Extraction of ZIZPHUS SPINA-CHRISTI leaves:

The powder material (obtained from the leaves) was extracted by maceration using 3:1 ethanol and water mixture for 72 h at room temperature. The extract was then filtered and concentrated under vacuum in a rotary evaporator at 45 C⁰, The remaining solvent was placed in a water bath to evaporate the residual ethanol-water mixture to get brown powder .

2.3.2.Synthesis of polyurethane

New polyurethane based on ZIZPHUS SPINA-CHRISTI leaves was prepared by mechanically mixing MDI and the powder (obtained from the extraction of leaves) in the ratio (1.5 MDI : 1 natural grinding leaves) W/W for 5 min.at 100 rpm in 500 ml beaker at room temperature in order to get homogeneous mixture .The mixed polyurethane was poured into the cavities of an open silicone mold and cured was carried out at 50 C⁰ for 24 hours followed at 100 C⁰ for 8 hours and finally at 150 C⁰ for 3 hours in order to complete polymerization. The product was characterized and used for analytical study .

2.4.Analytical Study

2.4.1. Batch Method

This technique **used** to evaluated metal ion uptake of the resin for mixture ions. 10 ml of 50 ppm of each ions (Zn²⁺ , Cr²⁺) were added to (0.1) g of dry resin , and adjusted pH by (0.1 M) HCl or NaOH using digital pH meter , then shaken for a contact time 4 hrs. , at different pH values , at room temperature, the mixture ions was filtrate , and metal ion uptake was determined by flam atomic absorption spectroscopy (FAAS.) using standard solution for each ions. Under same experimental conditions , using different shaking time from (0.25) to (24) hrs. at optimum pH 4.5 value, in order to choose better time for maximum percentage metal ion uptake . On the other hand the percentage recovery of ions was calculated by treated loaded resin with different concentrations HNO₃ (in order to get suitable concentration of the acid to separate these ions), under shaken at room temperature for 4 hrs. filtered and determined the concentration using (FAAS).

2.4.2.Recovery of the ions using Column Method

The dimensional column (14 × 0.4 cm) is packed with (8 g) of loaded resin with ions, which is soaked with distilled water for activating. First 1N HNO₃ was added as eluent and equal fractions of the eluent were collected and the ion concentration was determined using (FAAS). Then 3N HNO₃ was used as eluent and the same procedure was done to isolate the second ion from the resin.

3.Results and Discussion

3.1.Reaction Scheme and FTIR study

First the FTIR of the powder obtained from the extraction of Spina leaves (extracted material) as shown in Figuer.1 indicate the presence of absorption band in the regain 3383 cm⁻¹ due to the presence of hydroxyl groups , also another strong and sharp band in the region 1608 cm⁻¹ indicate the presence of aromatic ring in their structures as reported in some literature's(18-20). On the other hand the test of the aromatic hydroxyl groups was done by using ferric chloride solution which react with phenolic group to form violet color complex indication of the presence of hydroxyl groups(21).

A new polyurethane sample was obtained using grinding extracted material which contains hydroxyl groups in their structures reacted with di-isocyanate compound (PMDI) in the presence of catalyst, the curing cycle was done first left the sample to cure overnight in the mold at room temperature followed by post curing. The scheme of the procedure is sketched in Figure 2.

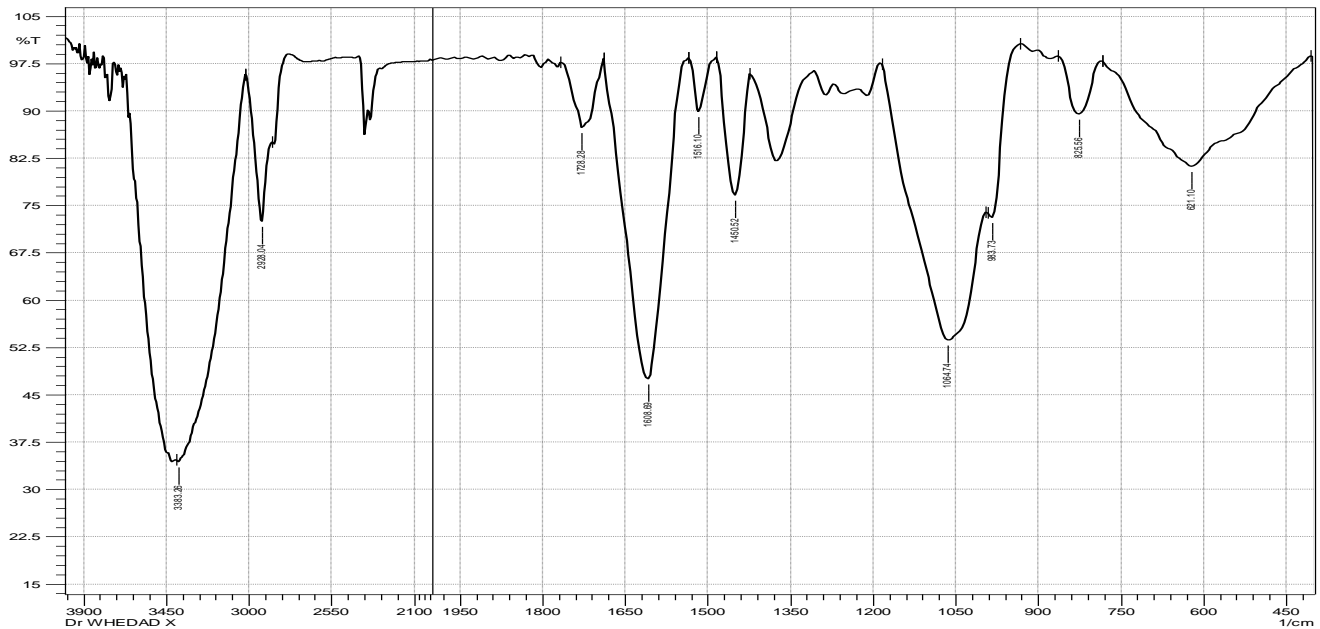
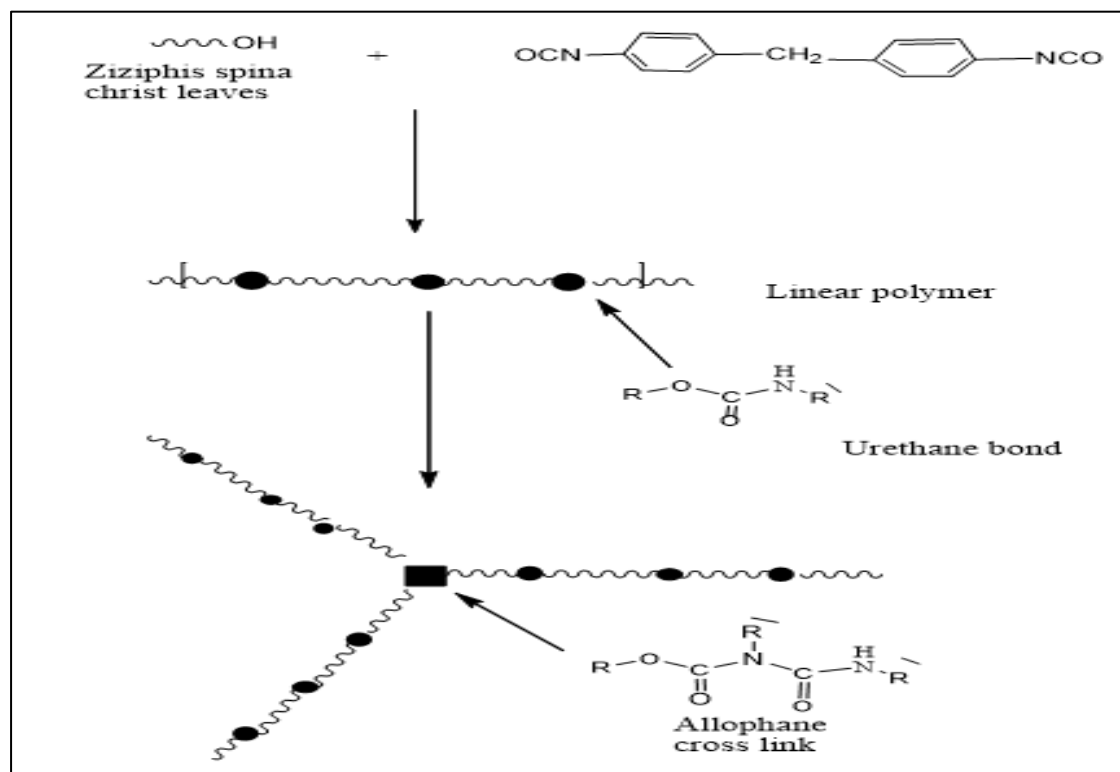


Figure 1. FTIR of the extracted material.



Figuer .2. procedure scheme for the preparation of polyurethane

The FTIR analysis confirmed the presence of groups characteristic for polyurethane. The FTIR spectra of the analyzed polymer are shown in Fig.3 & 4. Signal in the 3392 cm^{-1} due to stretching vibration of the N-H group present in the urethane groups included some unreacted hydroxyl groups(21). The signals at 2920 & 2852 cm^{-1} correspond to asymmetric and symmetric stretching vibrations of C-H bonds of $-\text{CH}_2-$ groups, respectively. The signal at 2275 cm^{-1} which correspond to unreacted isocyanate was observed only in the spectrum listed in Fig.3(22). And this band was disappear after complete curing of polyurethane as shown in Fig.4. Signals at 1653 - 1716 cm^{-1} indicates the presence of carbonyl bonds in urethane groups(20). The aromatic groups in materials correspond to 1618 cm^{-1} Signal at 1230 cm^{-1} due to C-N stretching vibrations. The multiple bands in the range ($1014 - 1101$) cm^{-1} is assigned to C-O bonds in flexible segments(23).

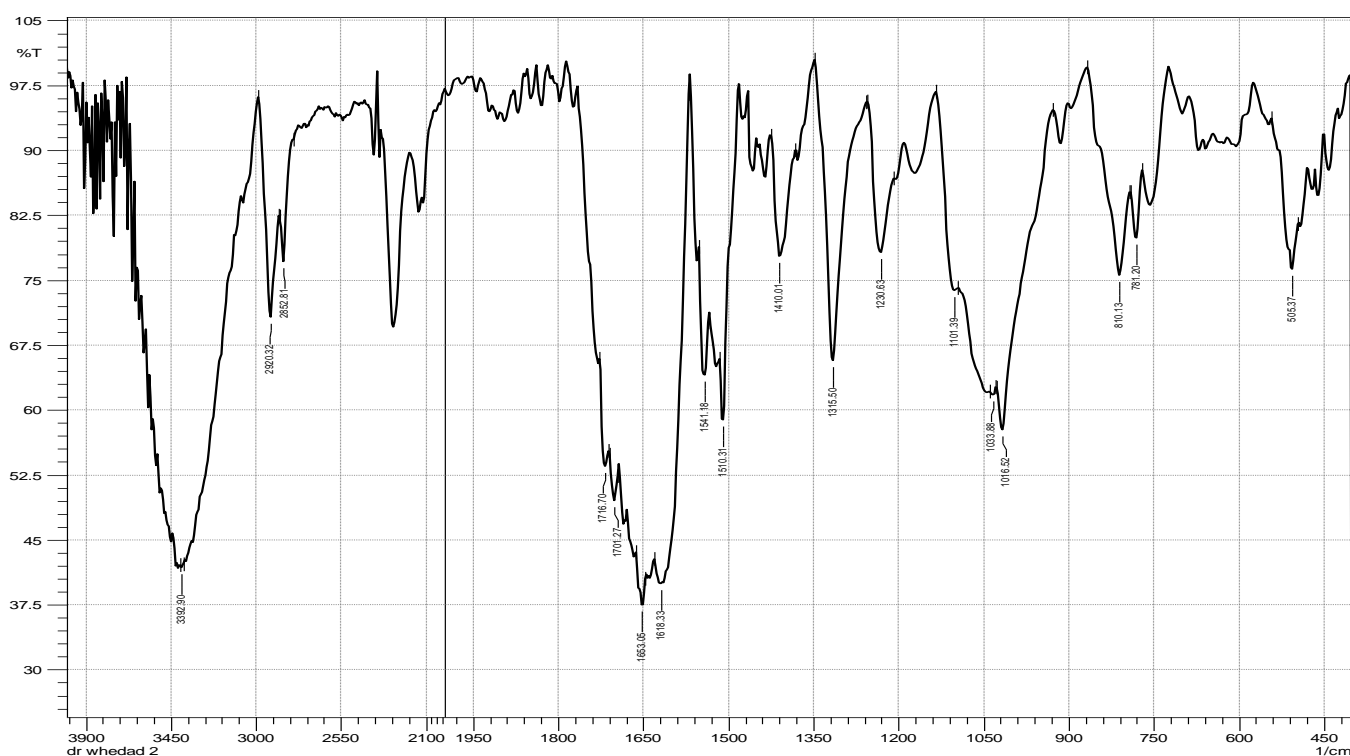


Figure 3. FTIR spectrum of polyurethane product after left 24 hours at room temperature.

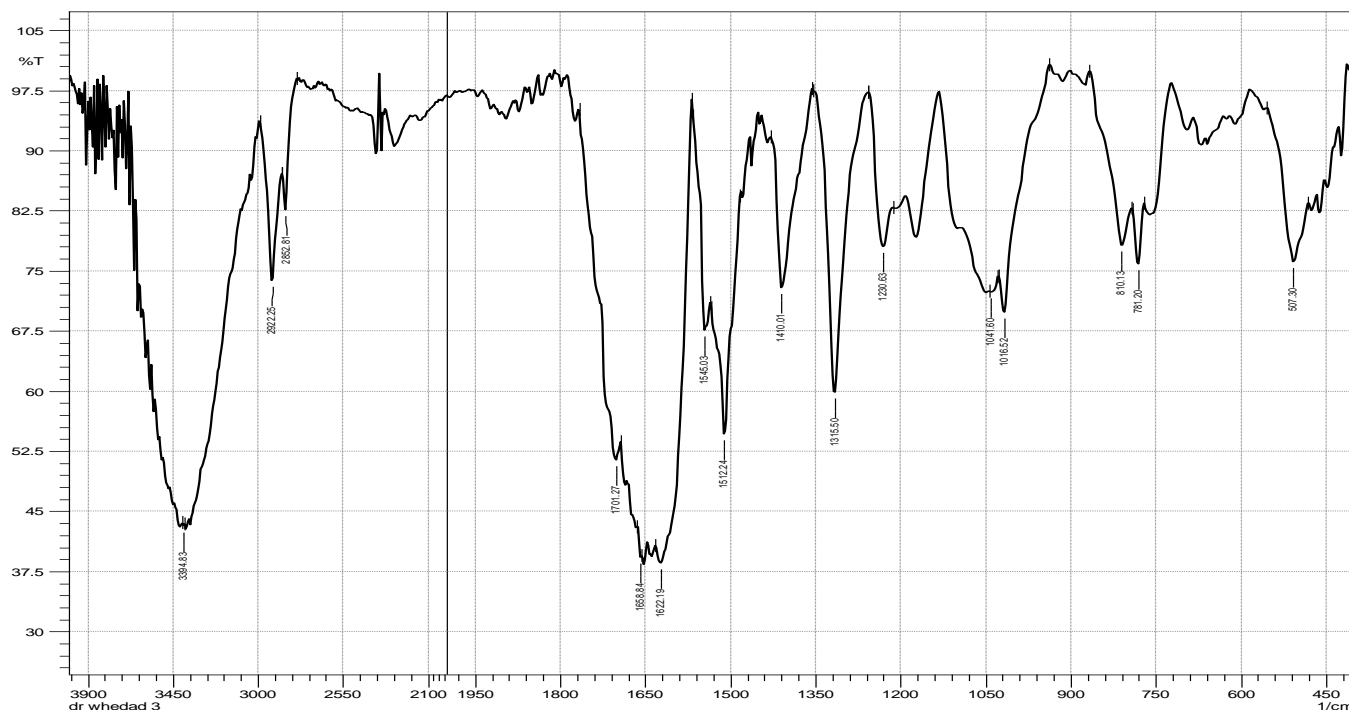


Figure 4. FTIR spectrum of polyurethane product after heat and complete curing

3.2. Thermal Analysis TG/DSC

The Thermal degradation of polyurethane has been investigated for the past 40 years(24, 25). The principle factors determining their thermal stability are related to the nature of the starting material and the conditions in which the way they are used. The predominant mechanism is strongly dependent on the nature of the substituents on the nitrogen and oxygen atoms(26, 27). Orlov and Tarakanov(28). give the free radical mechanism to explain the thermal degradation of polyurethane in three step :

- a . Dissociation of the urethane group into isocyanate and a polyol compound a reaction called depolarization.
- b. Dissociation of the urethane group into primary amine and an olefin with the release of CO₂.
- c. Formation of a secondary amine by the elimination of CO₂ .

So in this study the thermograms (TGA, DSC) are shown in Figure.5 From this thermogram, the prepared polyurethane was decomposition at a temperature (322C⁰) due to cleavage of urethane linkage, also the char residue at (650C⁰) was (42%) due to formation of fused structure containing benzene ring, while the temperature of (50%) weight loss (475C⁰). Figure (6). Show the proposed mechanism for the degradation of polyurethane

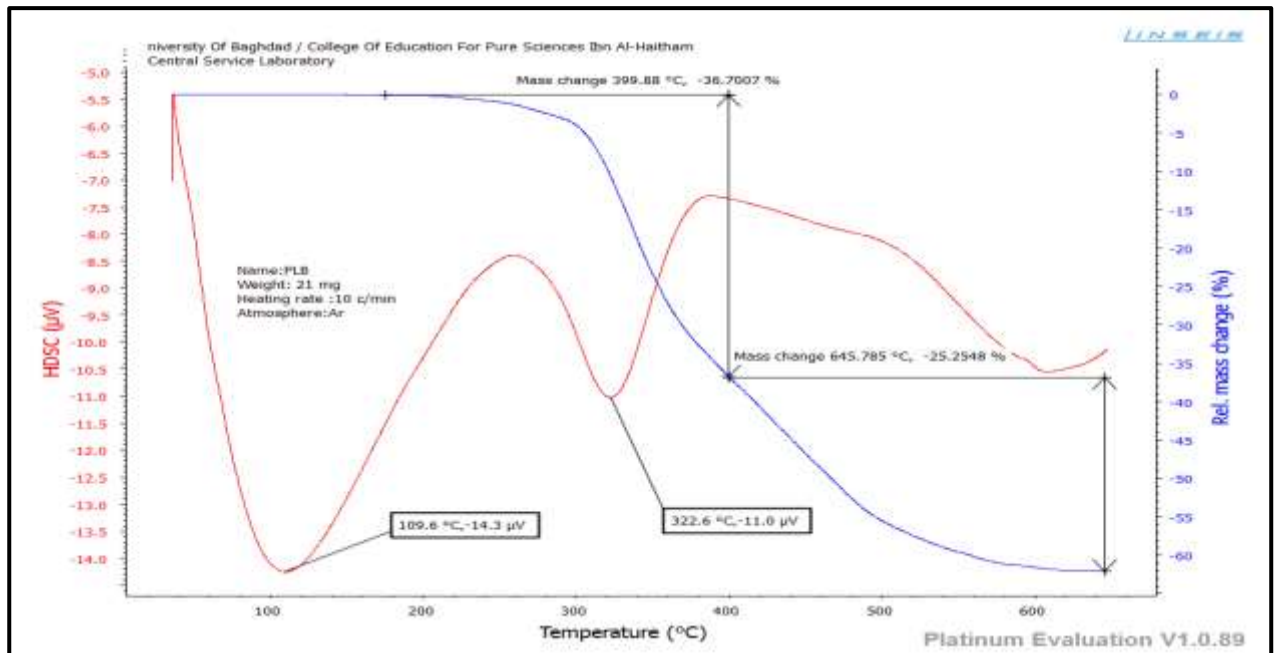


Figure.5 TGA and DSC thermogram of the prepared polyurethane

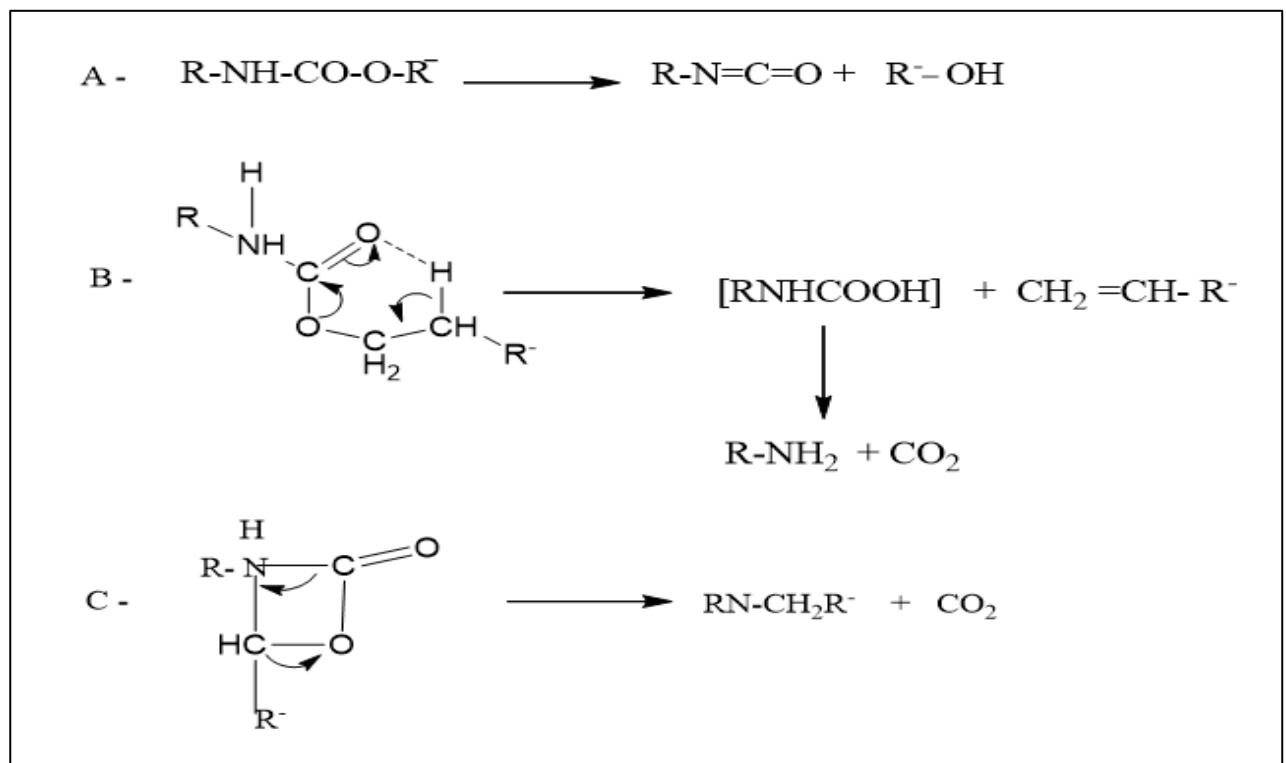


Figure (6) : proposed degradation mechanism of polyurethane based on leaves

On the other hand heat treatment was done of the prepared polymer at different temperature and the results was shown in table .1.

Table.1. Results of heat treatment for polyurethane sample at different temperature

Sample No.	Heat Temperature (C)	Weight Loss %	Remarks
1	150	3.23	No change in color (brown) of powder
2	200	4.11	No change in color (brown) of powder
3	250	9.42	No change in color (brown) of powder
4	300	28.56	Change in color of powder to (dark brown)
5	350	35.59	Change in color of powder to (dark brown)

Note : Hold temperature (1hrs.)under air conditions.

While for the DSC study , the thermogram showed an exothermic peak at (255C⁰) corresponding to the some of microcrystallinity of the polymer chains ,while the endothermic peak at 322 C⁰ correspond to the melting of this microcrystalline hard segment. After (300C⁰) the polymer will be degradation as shown from the TGA curve , either decompose of unstable biurets and allophanates bonds between polymer chains, or the H-bond of hard segment bonded near the multifunctional component and urethane may be broken

3.3 – Swelling and Extraction study.

Swelling behavior of the prepared polyurethane was investigated in the water at room temperature , the results presented in Figures 7 &8 .

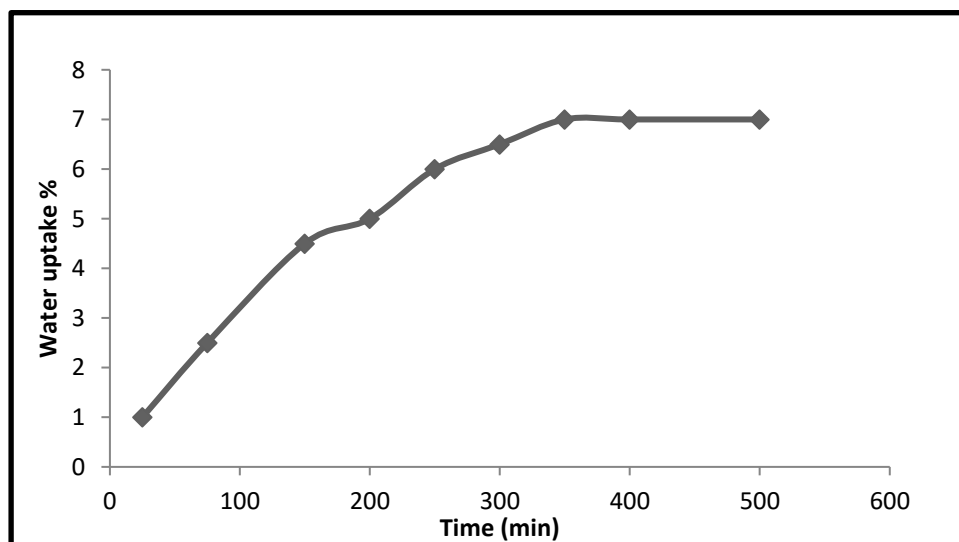


Figure 7. Water uptake as a function of time (600 min.)

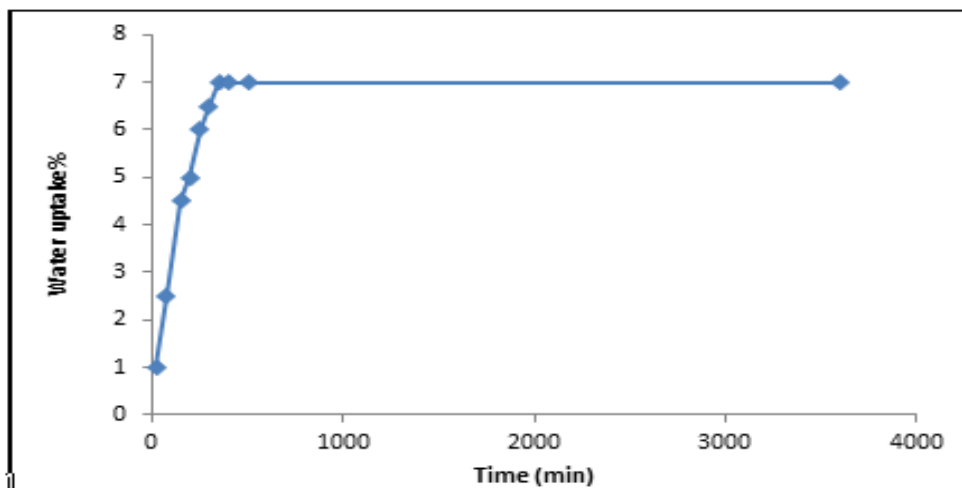


Figure 8. Water uptake as a function of time (4000 min.)

3.4. Analytical Analysis

3.4.1. Calibration curves and analytical parameters

Ions solutions of zinc and chromium with concentrations ranging (0-2.2)ppm were prepared in deionized water, to build the calibration curves.

3.5. Batch Method

In the batch system the optimization of parameters is presented such as equilibrium time, pH, and concentrations of HNO_3 on the recovery of these ions.

3.5.1. Effect of time

Metal ion uptake against time show that the first quarter hour 93% and 89% for zinc and chromium ions respectively. The increasing of metal ions uptake by the polymer was increase with increasing time until reached four hours, the metal ion uptake were 98 % and 93 % at seven hours was 99.3% and 97 % for zinc and chromium ions respectively. From the result and in order to not waste time we select the four hours as a good time for testing.

3.5.2. Effect of pH

The pH is an important and influential factor in the metal ions uptake by the synthetic polymer. Uptake percentage for zinc and chromium ion with different pH values was shown in Figure 9. and Table 2. From the figure the increasing in pH, the metal ions the uptake was increase until the maximum metal ions uptake reach 99.6 and 94.5 % at, pH 4.5 for zinc and chromium respectively. At pH 7 the metal ions uptake was decreased to 99.3% and 93.8% for zinc and chromium respectively. According to the above results the PH 4,5 is chosen as the optimum value because

the negative charges on the surface of polymer increases with increasing pH value. Also it was found that the effective groups of the polymer chains were affect on the uptake zinc ion higher than the chromium ions(29).

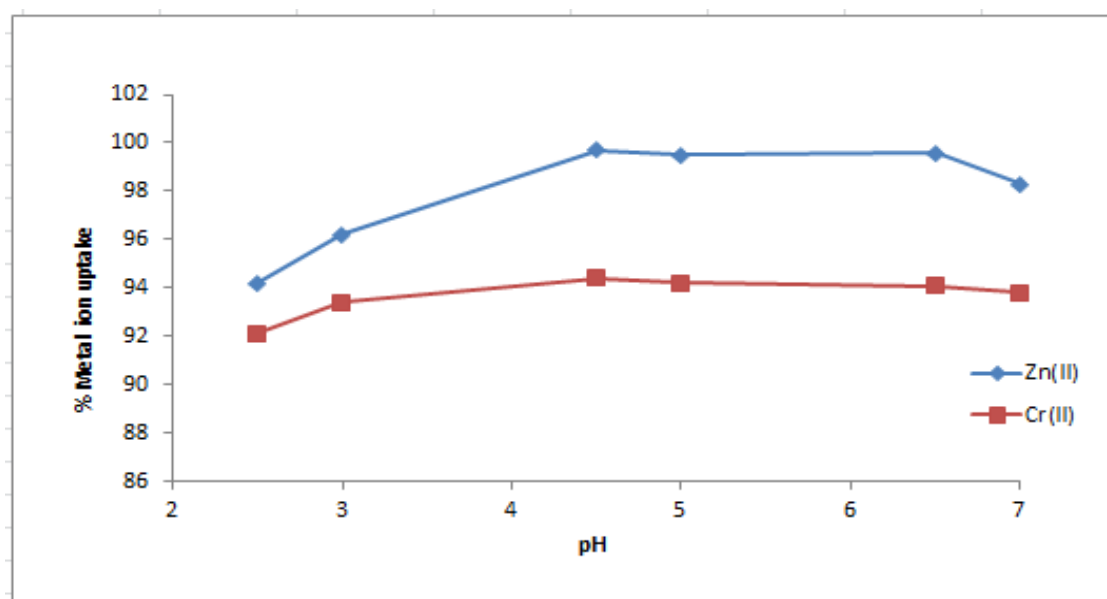


Figure 9. Effect of pH on the ions uptake by the polymer.

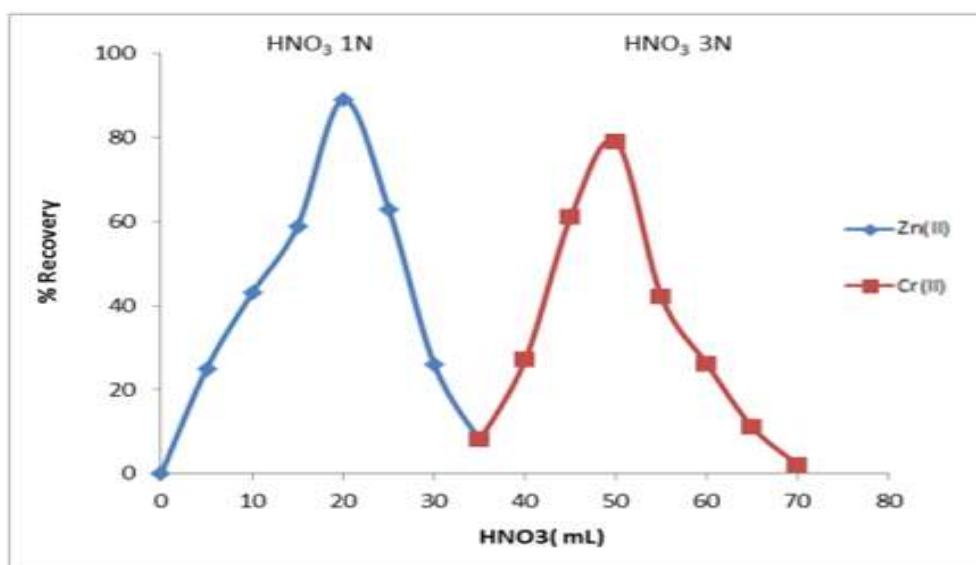
Table 2. The percentage of ions uptake by polymer againts pH

Cr(II) %	Zn(II) %	pH
94.1	99.2	2.5
94.4	99.2	3
94.5	99.6	4.5
94.2	99.5	5
94.1	99.6	6.5
93.8	99.3	7

3.5.3.Effect of eluent concentration (recovery of ions using column method)

Nitric acid was chosen as a modal acid for the recovery of ions in this study. Different concentrations of nitric acid were selected between 0.5 to 3 N as eluent.. The recovery ratio of zinc ions is higher than chromium ions at 1N nitric acid, while 3N of nitric acid was found as a suitable concentration for high recovery of both ions , so the concentration 1N and 3N of nitric acid was used for washing and separation of ions by using column method.

The quantitative separation of the metal ions was achieved on a glass column (height 50 cm, internal diameter was 0.6 cm) , packed with 2 g (100-200 mesh) loaded polymer. The flow rate of the effluent was maintained 1 ml/min throughout the elution process and the results were shown in Figure 10.



Figuer 10.Show the separations of ions from their mixture ions

From the results the first ion was separated at 89 % at 1N HNO₃ solution was zinc ion may be due to the large size and more atomic weight than chromium ion, while the chromium ion was separated at 79 % by washing the column with 3N HNO₃ solution because the active functional group presented in polymer chains was more attractive forces for chromium ion than zinc ions.

Conclusion

New polyurethane resin was prepared from the reaction of extracted material obtained from ZIZPHUS SPINA-CHRISTI leaves and methylene diphenyl diisocyanate, the resin was characterized using FTIR, TGA and DSC techniques. The prepared resin was used to uptake ions from solution and the result shown that the percentage of zinc and chromium ions was more than 75 %, on the other hand the recovery of these ions was done using column methods and nitric acid solution as eluent the optimum concentration of nitric acid used in this study was 1 and 3 normality. Also the thermal stability of this resin was evaluated using thermogravimetric analysis and the result shown that the temperature of decomposition was more than 300 °C and the char residue was 42 % at 650 °C.

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دراسة تحليلية وحرارية لنوع جديد من البولي يوريثان المحضر من أوراق نبات السدر

م.د.ميادة عبد الله عدنان، م.م. مها عبد الحسين حميد، أ.د.وداد صالح حنوش

الخلاصة :-

يهدف البحث الى استخلاص وعزل مادة فعالة من أوراق نبات السدر نوع -ZIZPHUS SPINA CHRISTI بالاستخدام الايثانول ومن ثم تحويل هذه المادة الى نوع جديد من البولي يوريثان من خلال البلمرة التكتيفية ومن خلال تفاعلها مع مادة المثيلين ثنائي فينيل ثنائي ايزوسيانات . شخص البوليمر المحضر باستخدام تقنية ال FTIR لتحديد نوع المجاميع الفعالة الموجودة في المادة المستخلصة والبوليمر المحضر . استخدم البوليمر لسحب بعض الايونات من محاليلها المائية وبينت النتائج بان نسبة سحب ايونات الزنك من قبل البوليمر المحضر كانت 99% في حين نسبة سحب ايونات الكروميوم كانت 97% بعد مرور 7 ساعات . ومن ناحية أخرى استخدمت طريقة الكولوم لبيان كفاءة إزالة الايونات من البوليمر وأثبتت نتائج الاسترجاع بنسبة 89% لايون الزنك في حين كانت 79% لايون الكروم باستخدام حامض النتريك بتركيز 1 و 3 نورمالي على التوالي .ومن ناحية أخرى تم تقييم البوليمر المحضر حرارياً من خلال استخدام تقنيتي التحليل الحراري الوزني والمسح التفاضلي المسعري .