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Synthesis and thermal stability study of polyurethane based on modified hydroxyapatite

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ABSTRACT

Hydroxyapatite (HA) is a ceramic made up of phosphate and calcium ions. Because of its bioactive properties, HA can interact with surrounding tissue after being implanted. This material is useful in several applications. So in this study we employed the sol-gel methodology to obtain a silica matrix modified with calcium and phosphate ions. We prepared the matrix by hydrolysis and condensation of the precursors HA with tetraethyl orthosilicate (TEOS), which were the sources of silicon, and the study included the chemical synthesis of new polyurethane based on modified hydroxyapatite (MHA). These polymers were prepared through the reaction of methylene diphenyl diisocyanate (MDI) with different percentages of MHA (10, 20, and 30%) and also the synthesis of polyurethane by direct reaction of MDI with MHA in the presence of a catalyst. Thermal stability of the prepared polymers was assessed using thermogravimetric analysis (TGA). The results show that the prepared polymers have one decomposition temperature over 200 °C. Also, the rate of decomposition decreases with an increasing percentage of MHA, and that means the presence of this modifier improves the thermal stability of polymers. On the other hand, the decomposition of polymers (after heat treatment) was more than 500 °C.

1. Introduction

Polyurethane is quite fascinating. It ticks off with a remarkable step – growth polyaddition reaction, where diisocyanate group (-NCO) meets and merges with a hydroxyl group (-OH), culminating in the formation of urethane bond [1]. As we delve into PU synthesis, we are greeted by a variety of long-chain Polyols such as polyesters, polyethers, and polycarbonates that can step into the spotlight. On the flip side, the second key players in this dynamic di iso cyanates [2], what makes (PU) truly specialist its intriguing Segmental structure / consisting of distinct hard and soft segments. The soft segments are born from polyols, while the hard segments are crafted from diisocyanates along with short chain extenders [3]. Polyurethan had a wide range of applications, such as thermal and sound insulation applications for buildings, as well as in automotive tires, coatings, adhesives, sealants, and elastomers [4]. The world of sports has also embraced the amazing capabilities of polyurethane, playing a pivotal role in formulating running surfaces that gently soothe athletes' joints and in creating mats for various sports. Moreover, the material lends its durability to boat construction, protecting ships from the ravages of water and petrochemicals [5].

Recent studies on hydroxyapatite have unveiled its remarkable role in bolstering the fire resistance and smoke suppression capabilities of polyurethane fire retardant. Composites fused with nano-HAP recently synthesized have been paving the way for new applications; for example, skin treated with hydroxyapatite showed Significant improvement in five resistances when placed over its untreated counterpart [6]. This leather was only partially burned. This behavior confirms the profound effect of hydroxyapatite on the fire resistance of leather. This improved performance is likely due to its exceptional thermal stability, which represents a major advance in fire resistance technology [7]. Several research studies used hydroxyapatite as internal and interaction crosslinks as well as a reinforcing filler to augment thermal and surface properties [8]. Hydroxyapatite (HA) (Ca_{10} (PO_4)$_6$ (OH)$_2$) is among the ceramic materials that are biocompatible.

belong to a class of tunable bioactive materials called

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calcium phosphate cements (CPCs) [9]. HA, collagen, and water make up the majority of natural bones. The natural substance found in teeth and bones is similar to the synthetic one.

Furthermore, synthetic hyaluronic acid (HA) is bioactive and biocompatible. It is a nontoxic as well as osteoinductive substance; hence, this mineral has several applications [10].

Polymeric materials have been used in several applications, so in this paper, new polyurethane was prepared from MHA using tetraethoxysilane (TEOS) as a precursor material for silicon, and their thermal stability was studied.

2. Materials and Methods

2.1. Materials

Methylene diphenyl diisocyanate (MDI) was supplied from Sigma Company with the following specification: Looks like a dark brown liquid, NCO content (wt %): 30.0-32.0, Viscosity (cps/250°C): 115-120 SP. Gravity (at 25°C): 1.23-1.25; Acidity (wt. %): Max 0.1. Polyester polyol was supplied from the Merck company with the following specification: pale yellow, Hydroxyl content: 60 mg KOH/g, Acid Value: 1.4 mg KOH/g, Viscosity: 9000 + 1500 at 35°C CP. Tetraethoxy silane (TEOS): This material was supplied from the Merck company with the following specifications: Appearance: colorless transparent liquid Density: 0.92-0.96 g/cm³, Purity: 99%, Refractive index (n25 D): 1.3788-1.3888

2.2. Instrumental

Fourier transform infrared (FT-IR) spectroscopy test: The Shimadzu FTIR-8400S was used to perform the FTIR spectra of cured phenolic resins. Each spectrum was captured using a potassium bromide (KBr) disk in the 400–4000 cm^{-1} frequency range. Previously, the KBr was oven-dried at 300 °C to lessen the water's interference.

Thermogravimetric Analysis (TGA): The TGA Q50 V20 Dynamic scan was used to analyze the TGA measurement. It was conducted in a nitrogen atmosphere at a flow rate of 30 ml/min, with a temperature range of 25-700 °C and a constant heating rate of 50 °C/min.

2.3. Synthesis methods

The prepared of the materials were donated in Table 1.

2.3.1. Synthesis of hydroxyapatite

Nano hydroxyapatite was prepared by mixing two solution, the first one was prepared by dissolve 11.4 g of diammonium hydrogen phosphate ($(NH_4)_2HPO_4$) in 125 ml of distill water and adjust the pH to while the other solution was prepared by dissolve 35.4 g of calcium nitrate tetrahydride in 125 ml of distilled water and adjust the pH to 7.4, then mixing these two solutions under good stirring at room temperature for 4 hours, taking into account that the pH should be equal 10.8 using a 0.1 M solution of sodium hydroxide (NaOH). After this the solution was left at room temperature overnight, a white precipitate was formed, which was washed several times with distilled water and ethanol to remove unreacted material then left to drying at laboratory temperature [9].

2.3.2. Synthesis of modified hydroxyapatite

This mineral was synthesis using tetraethyl orthosilicate (TEOS), so 1 g of the prepared hydroxyapatite was added to 15 ml of a (50:50) mixture of ethanol and water and left under a stirrer for half an hour at room temperature, then 7 ml of TEOS and 1 ml of sulfuric acid (1 M) was added to the solution, stirrer at 24 hours at room temperature. After that, the precipitate that developed was filtered. and washed with distilled water several times and left to dry at laboratory temperature.

2.3.3. Synthesis of polyurethane (PU)

Polyurethane alone was synthesis by a condensation reaction between polyester polyol and MDI, so 3 g of polyol was mixed with 3 g MDI Gentilly in the presence of a catalysts (tertiary amines, mixing in order to obtained homogeneous mixture, then leave it at laboratory temperature for 24 hours, after which the reaction mixture is placed in an air oven at 80 °C for 2 hours to complete the reaction, then at 100 °C for post-curing.

2.3.4. Synthesis of polyurethane based on modified hydroxyapatite

This polymer was prepared by adding 2 g of MHA to 8 g of MDI in the presence of triethylamine as a catalyst and mixing well at room temperature until the mixture becomes homogeneous, then pouring it into a polyethylene mold and leaving it for 24 hours to cure. Then the product is placed in an air oven at 80 °C for two hours to complete curing, then at 100 °C for post-curing.

Table 1. The prepared materials and their symbols

Symbol	Product Name
NHA	Nanohydroxyapatite
MHA	Modified hydroxyapatite
PU	Polyurethane prepared from polyol and MDI
PUM	Polyurethane prepared from MHA and MDI
$CPUM_{10,20,30}$	Composite polyurethane prepared from polyol and MDI in the presence of 10, 20, and 30% NHA

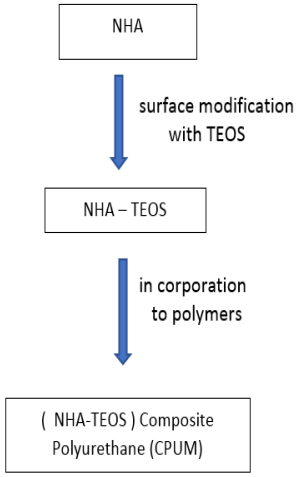
2.3.5. Synthesis of polyurethane composites based on modified hydroxy apatite

The CPU was synthesized first by mixing 5 g from Polyol with 0.55 g (10% MHA) until obtained of homogeneous mixture, then 5 g from MDI and 0.1% of catalyst was add to the mixture above and leave the reaction mixture over night at room temperature to curing, then the product was placed in an air oven at 80 °C for two hours to complete reaction, then at 100 °C for post curing The other percentages were prepared in the same way, where the weight of the MHA was 20% (1.2 g) and 30% (2.14 g).

3. Results and Discussion

3.1. Reactions and characterization

In this study, several types of polyurethane were prepared. Initially nano hydroxyapatite (NHAP) was synthesis form the reaction between diammonium hydrogen phosphate with calcium nitrate [1,2]. On the other hand, and in a different approach, NHAP was prepared using bone waste through heat treatment of bone at a temperature between 800 and 900°C [3, 4]. Modified HAP (MHAP) with silicon was prepared using TEOS in an acidic medium [5]; this reaction lead first to hydrolysis of TEOS to obtain silanol group Si-OH coated the surface of HAP. Also, the modification of HAP with TEOS involves the substitution of some phosphate groups (PO_4^{-3}) with in the hydroxyapatite structure with silicate groups (SiO_4^{-2}) presented in TEOS, and this substitution leads to a material with enhanced properties. Figure 1 is a picture showing the prepared NHA and MHA.



Scheme 1. Modification of HA.

Also, a new polymeric composite based on polyurethane was prepared using different percentages of MHAP (10, 20, and 30%). In order to improve thermal properties of the final products, this reaction was shown in Scheme 2.

However, new polyurethane was prepared by direct reaction of MHAP with MDI in the presence of triethylamine as a catalyst. This reaction was carried out through the reaction between the OH group presented on the surface of MHA with isocyanate groups in MDI, as shown in Scheme 3.

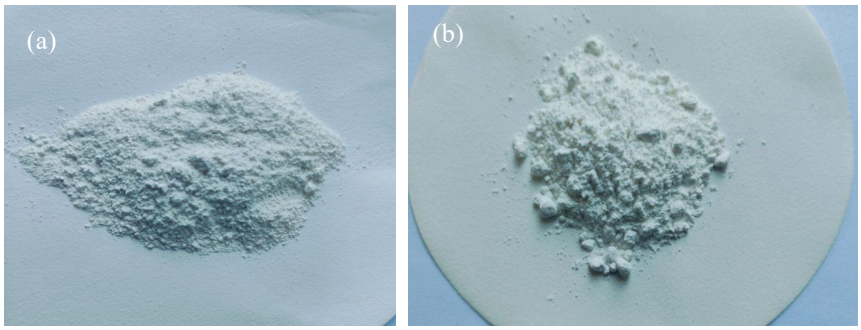
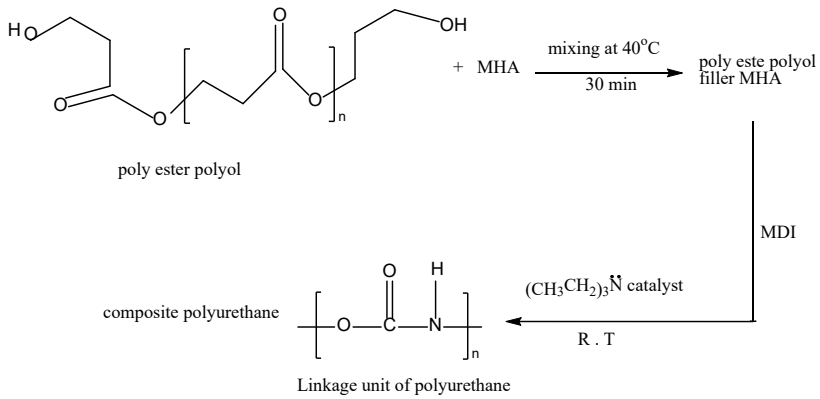
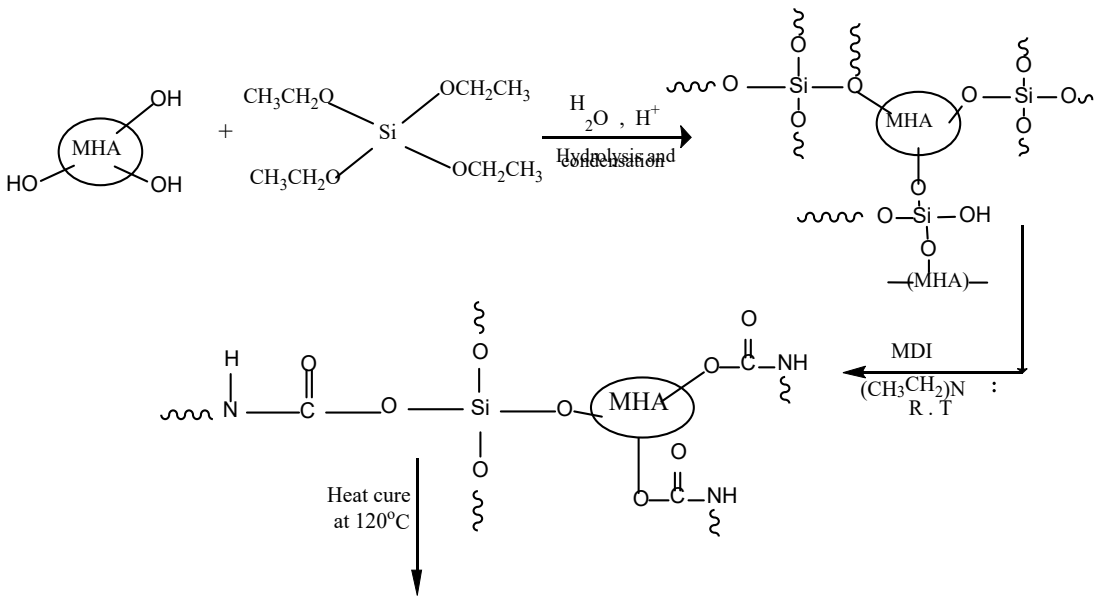


Fig. 1. (a). HA, (b). MHA



Scheme 2. Synthesis of composite PU based on MHA



Polyurethan modified HAP network structure

Scheme 3. Synthesis of PUM.

HAP and MHAP and their composites were characterized using FTIR (Figs. 1 and 2 Supp.)., show the FTIR of HAP and MHAP, respectively in the case of HAP, the characteristic bands was 1023, 600, and 559 cm^{-1}, corresponding to the phosphate groups [11], while bands at 1646, 872, due to the carbonate groups. Also, a small broadband at 3300 cm^{-1} corresponds to the hydroxyl group presented in their structure of HAP. While MHAP (4) shows a strong band due to the stretching and bending vibration of Si-O-Si presented in SiO_4 tetrahedral at 1041 cm^{-1} and which are overlapping with the band of PO_4 groups, also a new band at 800 and 667 cm^{-1} due to Si-O [12] and bands at 2924, 2854 cm^{-1} due to the stretching vibration of C-H aliphatic presented in the TEOS structure. For polymers (Fig. 3 Supp.), show the FTIR of polyurethane, small bands at 3290 cm^{-1} due to the stretching vibration of (N-H) urethane groups, also there is some small bands of an reacted OH and NCO groups can be found at around 3400 cm^{-1} [13] and 2125 cm^{-1}, and a band at 1720 cm^{-1} due to carbonyl groups for urethane. Also, bands at 1600 and 1526 cm^{-1} lead to C-C stretching vibration of aromatic rings and C-N stretching of urethane groups, and band at 1095 cm^{-1} is due to C-O-C stretching. while (Fig. 4 Supp.), show the FTIR spectrum of the polyurethane prepared by direct reaction between MDI and MHAP, (Fig. 4 Supp.) show the same characteristic band observed in polyurethane alone, except the presence of broad band at 1055 cm^{-1} due to stretching vibration of the Si-O-Si bond presented in MHAP [12], which is may be overlap with the band due to C-O-C observed in poly urethane. For heat treatment polyurethane samples prepared in this study at 500°C for 2 hrs., the FTIR spectrum as shown in (Fig. 5 Supp.), show different absorption band due to the thermal decomposition of the essential bonds.

3.2. Thermogravimetric analysis (TGS) of the prepared polymers and composite

The TGA techniques used to evaluate the thermal stability of many materials, polymers and polymer composites. TGA measures the amount and rate of change in sample weight as a function of temperature or time [14]. A solid matter's TGA can be performed either non-isothermal (dynamics) or at isothermal conditions (constant temperature), condition (generally the temperature varies with time), so in this study non-isothermal TGA was used to study the thermochemical behavior of the prepared polyurethane and their composite with MHA under an inert atmosphere. Figures (2-6) exhibits the TGA thermograms of the prepared polyurethane and their composites. TGA findings including temperature of 25%, 50%,, and 75 % weight loss (T25, T50, and T75) obtained. Also, the optimum decomposition temperature was evaluated from these thermograms and presented in Table 2. From the results, all the samples listed in Table 1. exhibit one decomposition temperature above 250℃, and the char residue of the PUs composites increase with increasing weight percentage of MHA (i.e in the case of PU alone the chare content was 10%, while in the presence of 30%, it was 29%). On the other hand, the rate of decomposition decreases with increasing MHAP presented in PU composites, 2.78 % /min in the case of PU alone, while in the presence of 30 %. MHA was 2.27 % /min., and for polyurethane prepared from MHA (PUM) was 1.76 %/min, this means that, the thermal stability improved in the presence of MHA. The presence of one decomposition temperature of PU means the complex portion of PU crosslink and backbone decomposed [15].

In the other study, some of the prepared PU and their

composites was heat treatment at 500°C, and evaluate the thermal behavior using TGA technique, so Figures 7-9, show TGA thermograms of PU, PUM_1 and PUM_2 respectively, and the thermal parameter obtained were listed in Table 3, from the results all the samples have one decomposition temperature around 550°C, and the char content at 800°C was 62-69%, while the rate of decomposition was 1.76, 1.51, and 1.46%/min for PU, PUM_1 and PUM_2 respectively. In addition, the same study was done for the polymeric composite prepared from polyurethane containing different percentage of MHA (CPUM), Figures 10-12 show TGA thermograms of these samples, white the thermal parameter obtained from these thermograms were listed in Tables 4. The results indicate these composites have one decomposition temperature 550°C, and have high char reside around 550°C (more than 60%, at 800°C).

The presence of MHA leads to an increase in the decomposition temperature, also increasing char reside, while the rate of decomposition was decrease, this means the presence of MHAP leads to an improvement in the thermal stability of PU [16].

Table 2. Thermal stability parameter for prepared polyurethane obtained from TGA thermograms.

Sample name	Decomp. Temp. °C	Wight loss at decomp. Temp. %	Temp. of 25% wt. loss °C	Temp.of 50%wt. loss °C	Temp. of 75% wt. Loss°C	Char. at 800°C %	Rate of decomposition%/min
PU	272	17	281	323	614	10	2.78
$CPUM_{10\,\%}$	250	13	293	357	670	14	2.40
$CPUM_{20\%}$	275	20	299	334	673	16	2.71
$CPUM_{30\%}$	270	10	312	371	>800	29	2.27
PUM_1	295	14	336	537	>800	40	1.96
PUM_2	300	9	349	743	>800	48	1.76

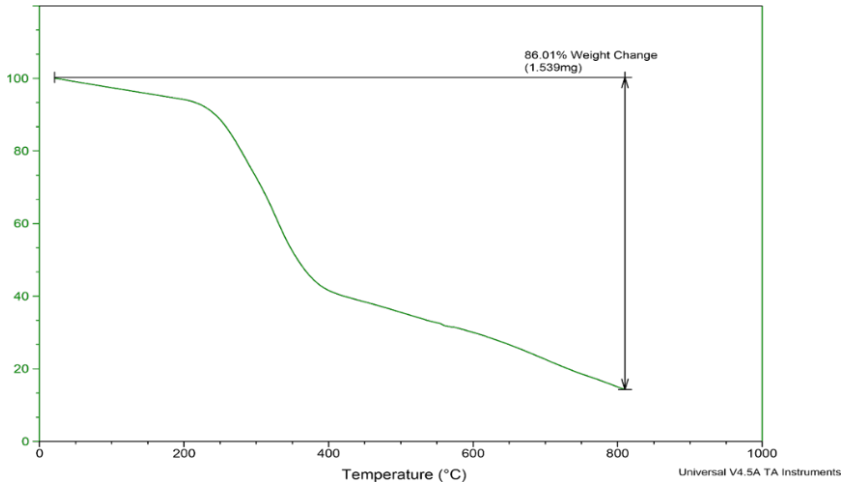


Fig. 2. TGA thermogram of PU

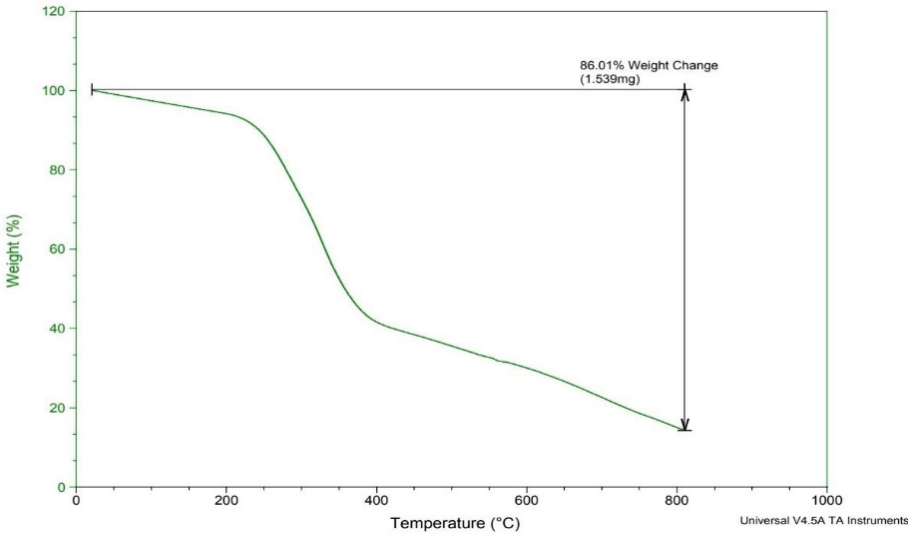


Fig. 3. TGA thermogram of CPUM20%

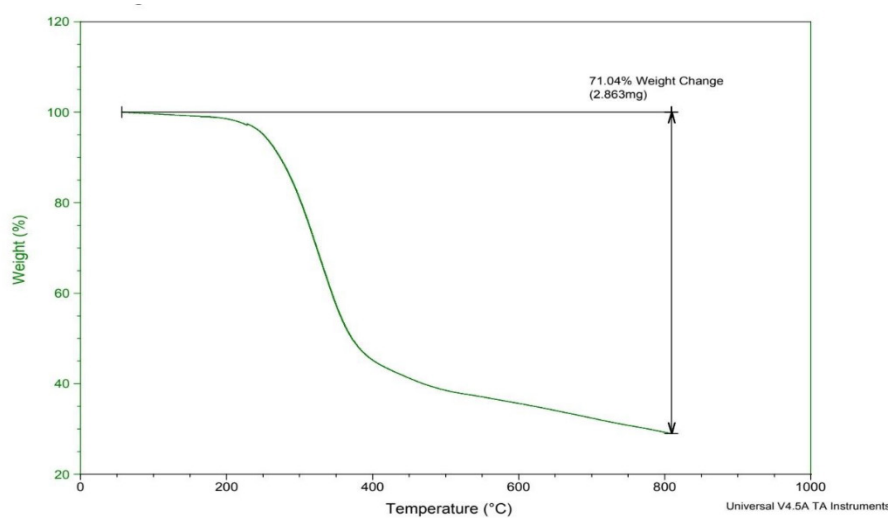


Fig. 4.TGA thermogram of $CPUM_{30\%}$

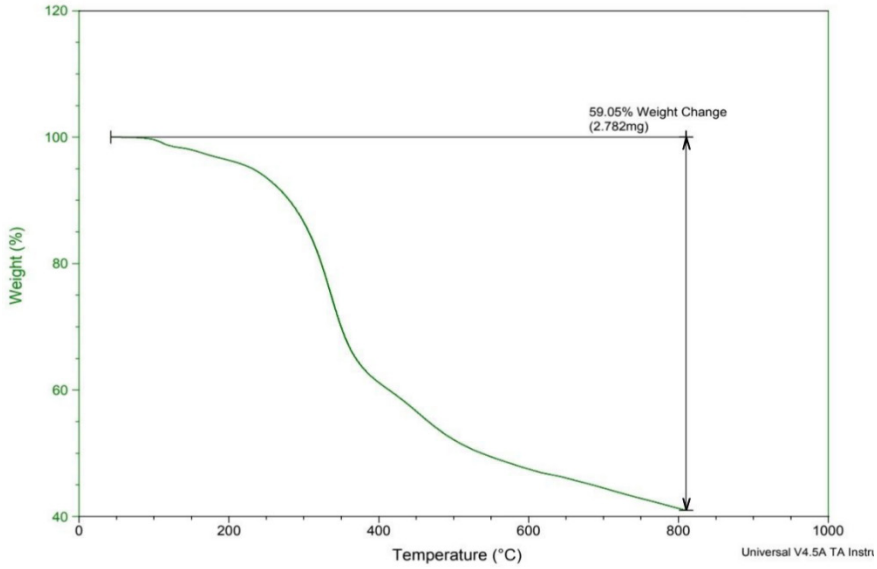


Fig. 5. TGA thermogram of PUM_1

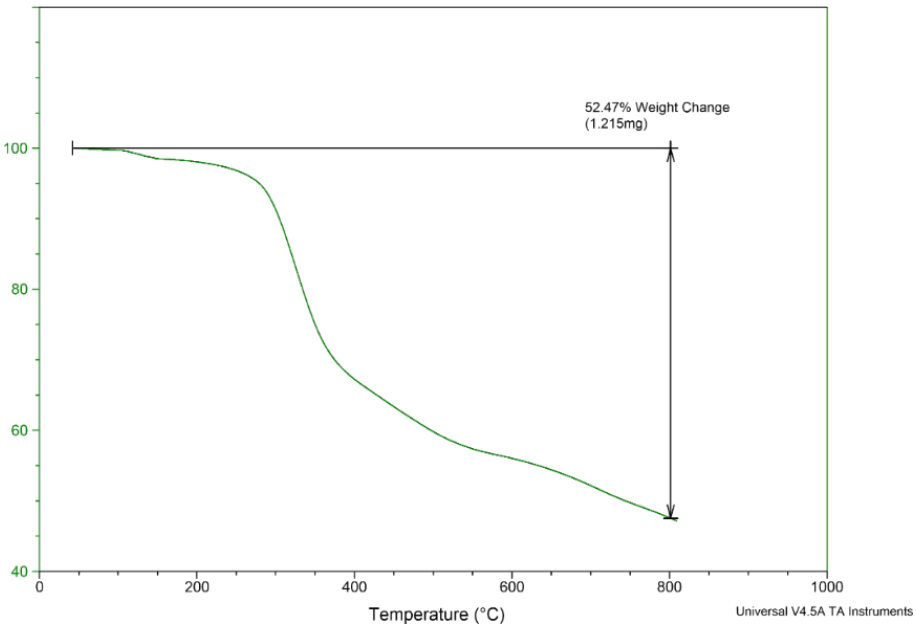


Fig. 6. TGA thermogram of PUM_2

Table 3. Some thermal stability parameter for prepared polyurethane (after heat treatment at 500ºC for one hrs.), obtained from TGA thermograms.

Sample name	Decomp. Temp. °C	Wt. loss at decomp. Temp. %	Temp. of 25% wt. loss ºC	Temp.of 50%wt. loss ºC	Temp. of 75% wt.loss ºC	Char. at 800ºC %	Rate of decomposition%/min
PU	550	17	664	>800	>800	62	1.76
PUM_1	565	19	654	>800	>800	65	1.51
PUM_2	537	12	716	>800	>800	69	1.46

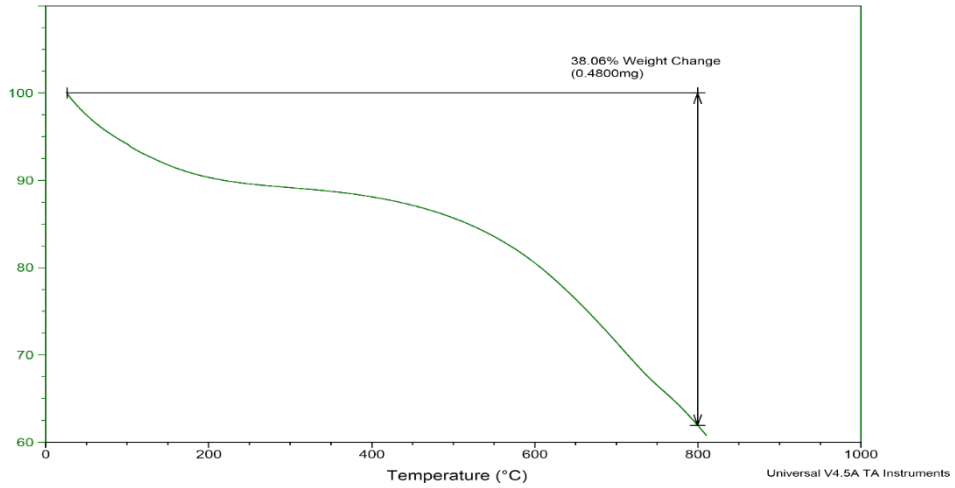


Fig. 7. TGA of PU after heat treatment at 500 °C

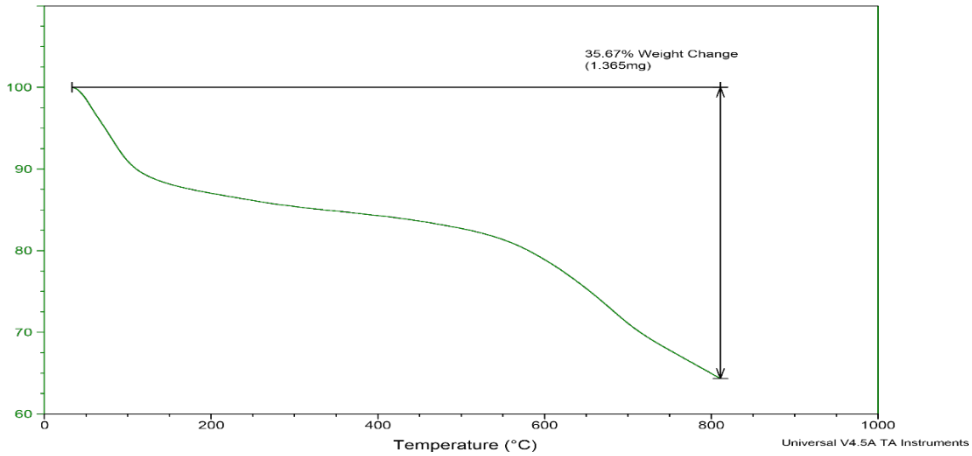


Fig. 8. TGA of PUM1 after treatment at 500 °C

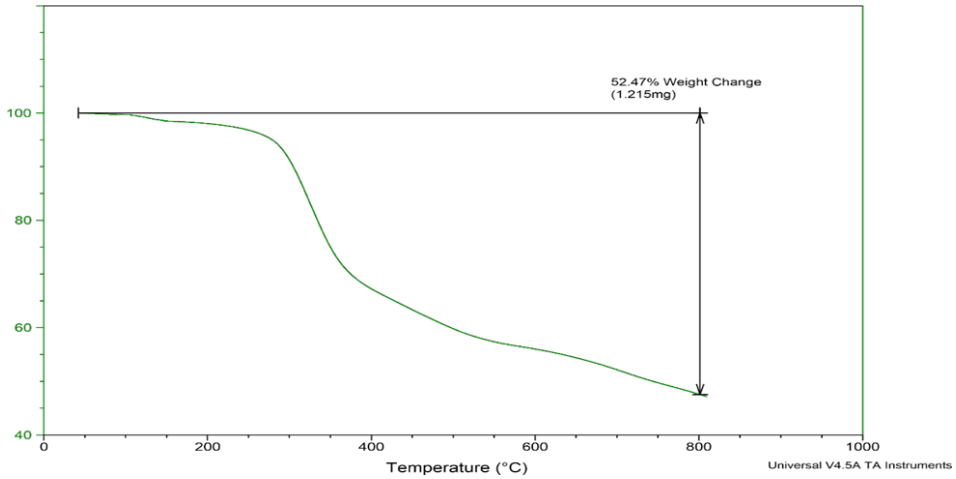


Fig. 9. TGA thermogram of PUM2

Table 4. Some thermal stability parameter for prepared polyurethane (after heat treatment at 500 °C (obtained from TGA thermograms

Sample name	Decomp. Temp. °C	Wt. loss at decomp. Temp. %	Temp. of 25% wt. loss ºC	Temp.of 50%wt. loss ºC	Temp. of 75% wt. Loss ºC	Char. at 800ºC %	Rate of decomposition%/min
PU	550	17	664	>800	>800	62	1.76
$CPUM_{10\%}$	562	15	676	>800	>800	65	1.74
$CPUM_{20\%}$	556	11.5	699	>800	>800	67	1.77
$CPUM_{30\%}$	575	11	690	>800	>800	69	1.66

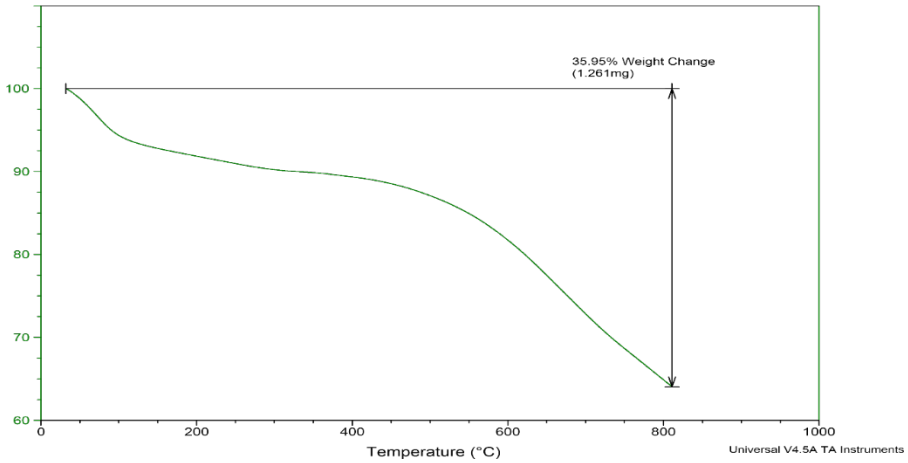


Fig. 10. TGA thermogram of $PUM_{10\%}$ after heat treatment at 500 °C

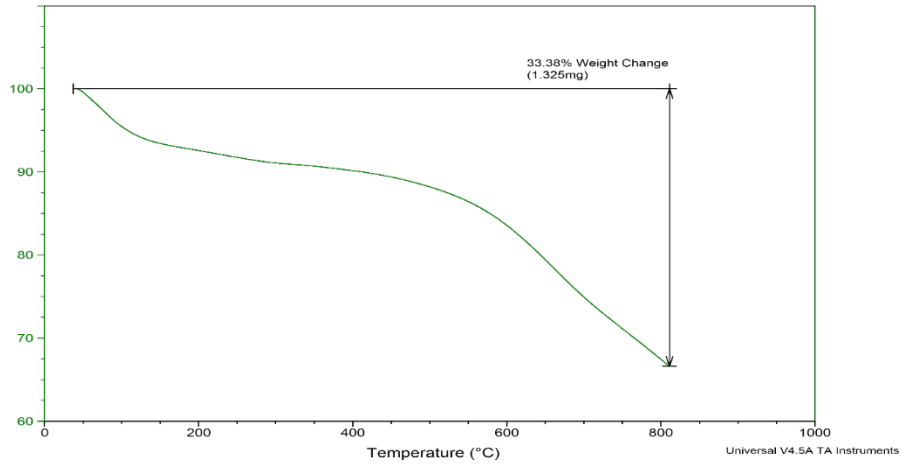


Fig. 11. TGA thermogram of $PUM_{20\%}$ after heat treatment at 500 °C

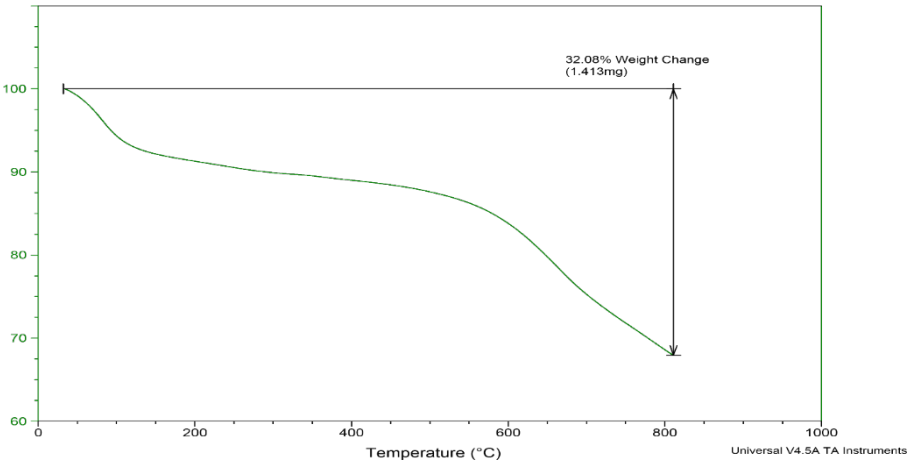


Fig. 12. TGA thermogram of $PUM_{30\%}$ after heat treatment at 500 °C

4. Conclusion

The present work describes the preparation of nanohydroxyapatite and modified hydroxyapatite using (TEOS) as precursor of silicon. Also, polymers and polymer composites based on polyurethane were prepared using modified hydroxy apatite to increase the thermal properties of polymers.

The thermal stability was evaluated using TGA technique, and the results show that the prepared materials have decomposition more than 200°C, and no meit up to 5oooC. on the other hand, the decomposition temperature and char content increase with increase percentage of modified hydroxyapatite in the polymer composites, so it is anticipated that this study may open the way for future investigations in the use of modified hydroxy apatite with other polymers.

Supplementary files

Supplementary file 1.

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