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Microwave synthesis and thermal properties of polyacrylate derivatives containing itaconic anhydride moieties

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Abstract

Background: Microwave irradiation as an alternative heat source is now a well-known method in synthetic chemistry. Microwave heating has emerged as a powerful technique to promote a variety of chemical reactions, offering reduced pollution, low cost and offer high yields together with simplicity in processing and handling. On the other hand, copolymers containing both hydrophilic and hydrophobic segments are drawing considerable attention because of their possible use in biological systems. Various copolymer compositions can produce a very large number of different arrangements, producing materials of varying chemical and physical properties. Thus, the hydrophilicity of copolymers can be modified by changing the amount of incorporated itaconic anhydride.

Results: A series of methyl methacrylate (MMA) and acrylamide (AA) copolymers containing itaconic anhydride (ITA) were synthesized by microwave irradiation employing a multimode reactor (Synthos 3000 Aton Paar, GmbH, 1400 W maximum magnetron) as well as conventional method. The thermal properties of the copolymers were evaluated by different techniques. Structure-thermal property correlation based on changing the itaconic anhydride ratio was demonstrated. Results revealed that the incorporation of itaconic anhydride into the polymeric backbone of all series affect the thermal stability of copolymers. In addition, the use of the microwave method offers high molecular weight copolymers which lead eventually to an increase in thermal stability.

Conclusions: Microwave irradiation method showed advantages for the produced copolymers compared to that prepared by conventional method, where it can offer a copolymer in short time, high yield, more pure compounds and more thermally stable copolymers, rather than conventional method. Also, microwave irradiation method gives higher molecular weight due to prevention of the chain transfer. Moreover, as the itaconic anhydride content increases the thermal stability and T_g increase due to the decrease in the crystallinity.

Background

Microwave-assisted organic synthesis has been recognized as one of the most interesting areas of current chemical research [1-5]. Microwave heating has emerged as a powerful technique to promote a variety of chemical reactions, offering reduced pollution, low cost and offer high yields together with simplicity in processing and handling [1,6-11]. The application of microwave irradiation to organic synthesis has been the focus of considerable attention in recent

years and is becoming an increasingly popular technology [12-19]. Recently, there has been growing interest in applying microwave irradiation to polymer synthesis [20-23] as it can accelerate many syntheses providing selective activation with short start-up phase and can allow fast optimization of reactions.

Itaconic anhydride (ITA) is an unsaturated dicarboxylic organic anhydride with one carbonyl group conjugated to the methylene group. It can be regarded as a substituted acrylic or methacrylic derivatives. In addition, it can be obtained from renewable resources [24,25]. Also, it can be polymerized [26,27] or copolymerized with various other monomers by free radical reactions [28-33].

As a result of increasing studies of acrylate derivatives and itaconic acid polymers or copolymers, various methods

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Table 1 Yield % of MMA/ITA and AA/ITA copolymers using microwave irradiation

Method	Copolymer Code	MMA/ITA ratio	MMA (mmol)	ITA (mmol)	Yield (%)
Conventional	PMITA 1	10: 2	50	10	44.6
Microwave	PMITA 4	10: 1	100	10	76.9
	PMITA 5	10: 2	100	20	86.0
	PMITA 6	10: 4	100	40	63.8
	Method	Copolymer Code	AA/ITA ratio	AA (mmol)	ITA (mmol)
Conventional	PAITA 2	10: 4	50	20	09.3
Microwave	PAITA 7	10: 1	50	5	92.5
	PAITA 8	10: 2	50	10	69.8
	PAITA 9	10: 4	100	40	57.0

for the synthesis of these polymers and their copolymers with the monomers such as acrylonitrile, styrene and acrylic esters have been reported, in most cases radical polymerizations [34-42]. Various copolymer compositions can produce a very large number of different arrangements, producing materials of varying chemical and physical properties. Thus, the hydrophilicity of copolymers can be modified by changing the amount of incorporated itaconic anhydride [27].

The present work described copolymerization of acrylic monomers such as methyl methacrylate (MMA) and acrylamide (AA) with different ratios of itaconic anhydride (ITA) through free radical copolymerization in the presence of α,α' -azobisisobutyronitrile (AIBN). Copolymerizations were carried out in both conventional and microwave conditions. The chemical structure of the prepared copolymers was confirmed by FTIR and size exclusion chromatography (SEC) and the thermal stability were studied using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC).

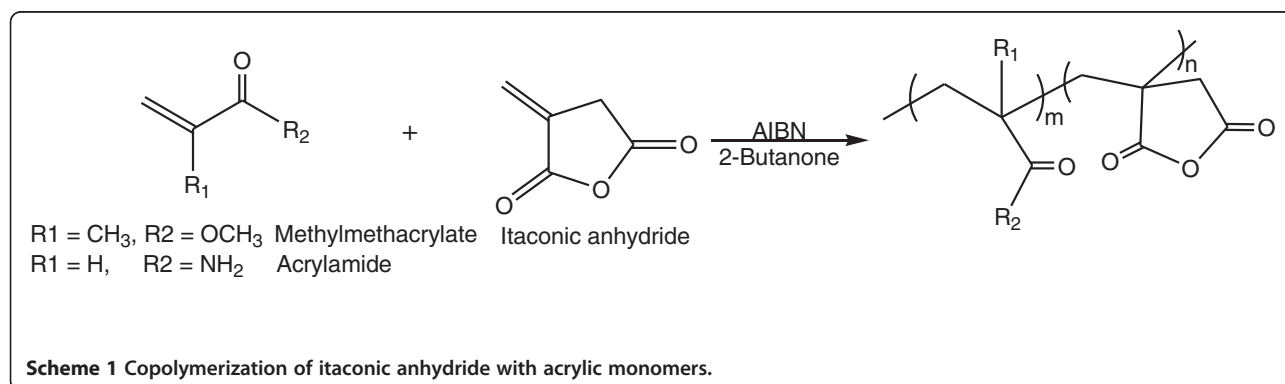
Experimental

Materials & Equipments

Itaconic anhydride (ITA) was purchased from Fluka. Acrylamide (AA) was purchased from LKB. Methyl

methacrylate (MMA) was purchased from M & B. α,α' -Azobisisobutyronitrile (AIBN) was purchased from Aldrich and was recrystallized from absolute ethanol before use. All solvents were dried before use.

The microwave irradiation employing a multimode reactor (Synthos 3000, Aton Paar GmbH, 1400 W maximum magnetron) was used to prepare the copolymers. Fourier transform infrared spectroscopy (FTIR) Spectra was recorded on Nicolet 560 Magna spectrometer. Thermal properties of the copolymers were examined through using thermogravimetric analysis (TGA) under nitrogen, from room temperature to 800°C with heating rate of 10°C/min and differential scanning calorimetry (DSC) which was carried on TA-Q500 in which Specimens of (5-10 mg) were encapsulated in aluminum pans and were heated or cooled between -25°C and 400°C under dry nitrogen atmosphere with heating rate of 10°C/min. Number-average molecular weight (M_n) and molecular weight distribution (M_w/M_n) were estimated by size exclusion chromatography (SEC) which was carried on Viscotek, HT-GPC. Polystyrene standards were employed for calibration. The analysis was performed at 35°C using high-performance liquid chromatography-grade THF as the eluent. Polystyrene standards were used to calibrate the molecular weight.



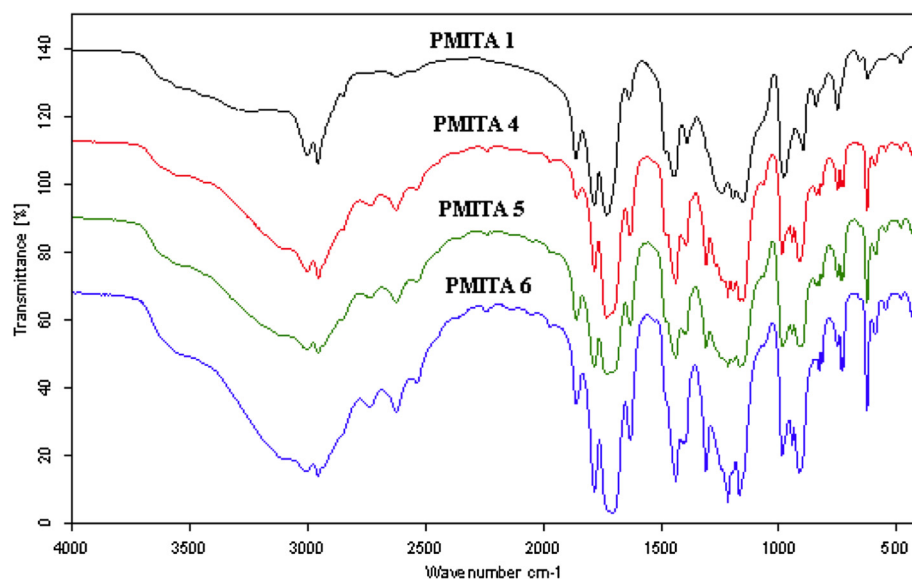


Figure 1 FT-IR spectra of PMITAs copolymers.

Synthesis of Copolymers

General Procedure for Conventional Method

In a general procedure, in a three-neck round bottomed flask fitted with a condenser, methyl methacrylate (MMA) (5.00 g, 50.0 mmol) or acrylamide (AA) (3.55 g, 50.0 mmol) was mixed with itaconic anhydride (ITA) (1.12 g, 10.0 mmol) in 2-butanone (20 mL). The reaction mixture was thoroughly purged with nitrogen for 10 min. Copolymerization was initiated by adding AIBN (0.1 g, 0.01% w/w) and then was heated at 60°C, under nitrogen atmosphere for 24 h. The reaction was cooled down to room temperature, and then the copolymer was

precipitated in diethyl ether. The precipitated copolymer was filtered, washed by excess of diethyl ether and was dried in oven under vacuum at 40°C for 24 h, Table 1.

General Procedure for Microwave-Assisted Synthesis

Employing a multimode reactor (Synthos 3000, Aton Paar GmbH, 1400 W maximum magnetron); the initial step was conducted with 4-Teflon vessels rotor (MF 100) that allow processing four reactions under the same conditions. Each vessel has itaconic anhydride (ITA) was mixed with methyl methacrylate (MMA) or acrylamide (AA) in specific ratio (10:1, 10:2, and 10:4), in the presence of (0.1 g) AIBN and

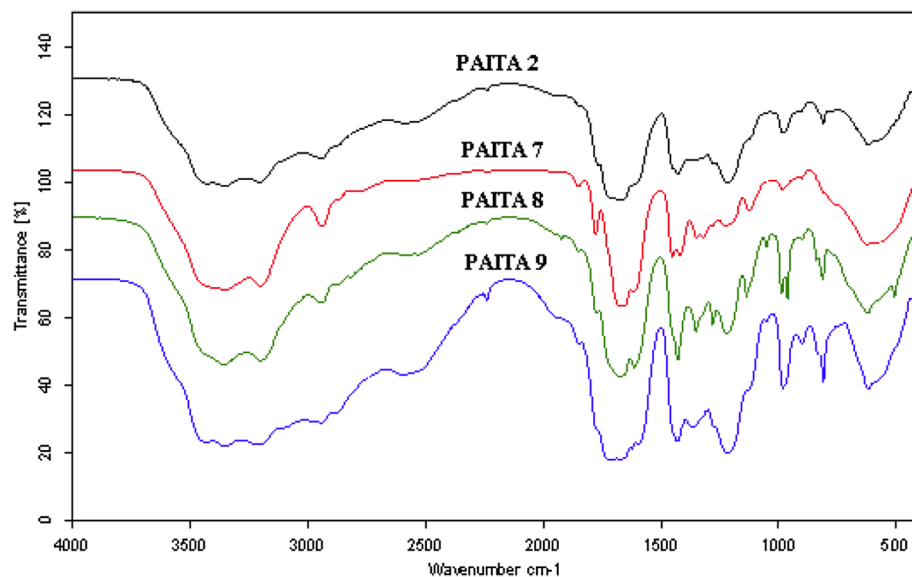


Figure 2 FT-IR spectra of PAITAs copolymers.

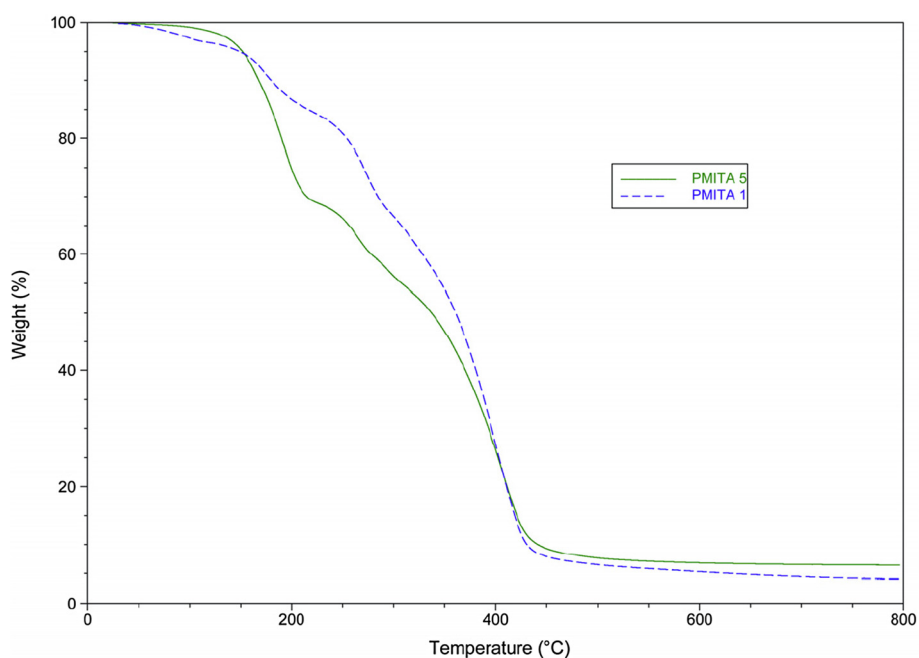


Figure 3 TGA of copolymers PMITA1 and PMITA5.

2-butanone as a solvent (Table 1). The individual vessels were purged with nitrogen gas for 10 min and then were placed in the corresponding rotor, fixed by screwing down the upper rotor place, and finally the rotor was closed with a protective hood. After heating the vessels for 5 min. at 140°C and hold at the same temperature for 5 min (~2 bar pressure, 400 W). Cooling was accomplished by a fan

(5 min). The final product was washed with diethyl ether, and was dried in oven under vacuum at 40°C for 24 h.

Results and Discussion

The copolymerization reaction of methyl methacrylate and acrylamide with itaconic anhydride was carried out by two different methods: a) conventional method using of 2-

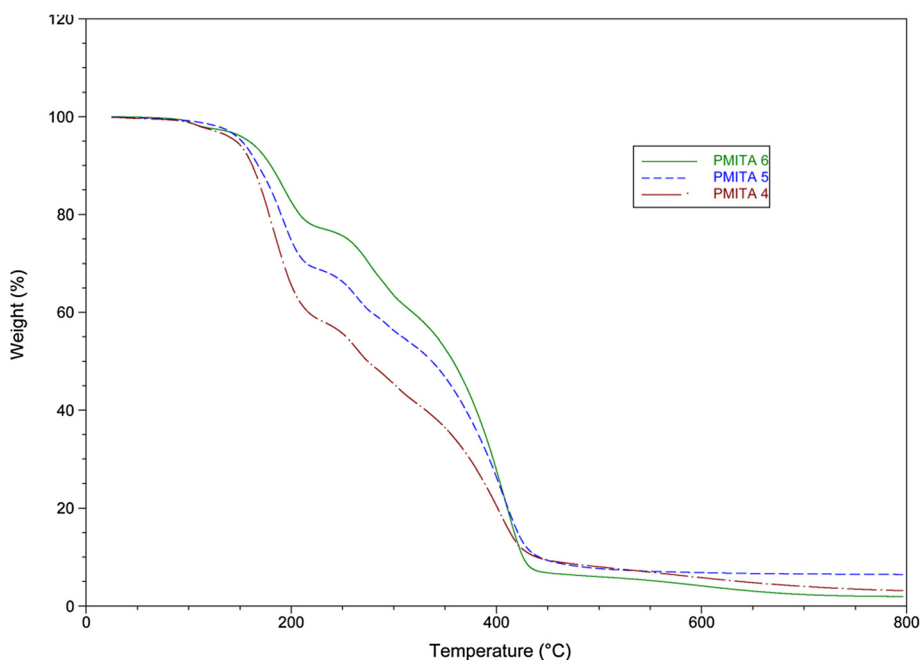


Figure 4 TGA thermogram of copolymers PMITA4-6.

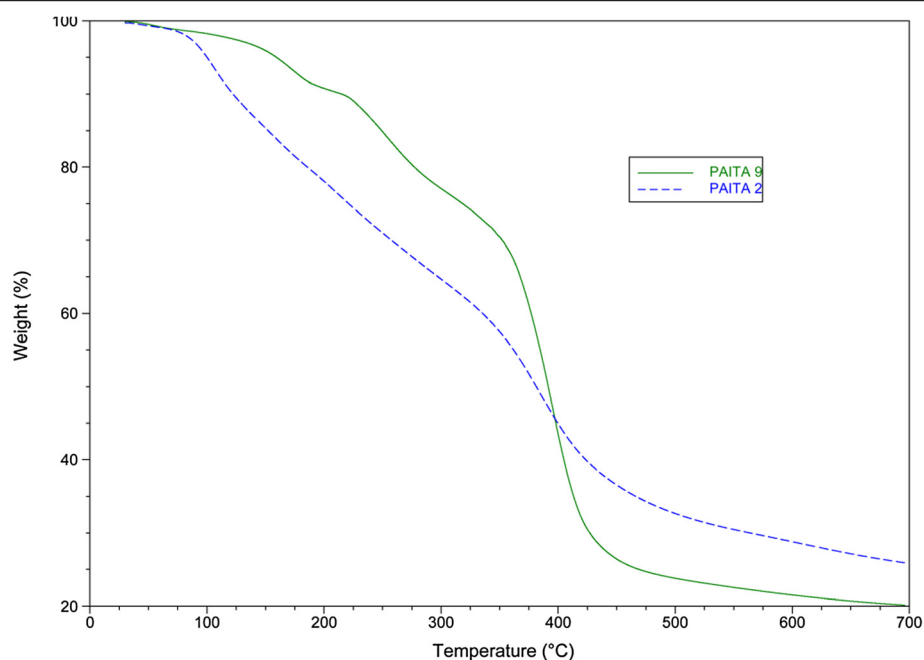


Figure 5 TGA thermogram of copolymers PAITA2 and PAITA9.

butanone as a solvent and AIBN as initiator for free radical polymerization at 60°C, under nitrogen atmosphere for 24 h and b) Employing a microwave synthesis using a multimode reactor (Synthos 3000, Aton Paar GmbH, 1400 W maximum magnetron), in the presence of AIBN for 5 min. at 140°C and 400 W, Scheme 1. The prepared copolymers

were characterized using FT-IR spectra, TGA, DSC, SEC, and elemental microanalysis.

Fourier Transform Infrared Spectroscopy (FT-IR)

The FTIR spectra of itaconic anhydride copolymers, Figures 1 and 2 showed the absorption peaks for the ITA at

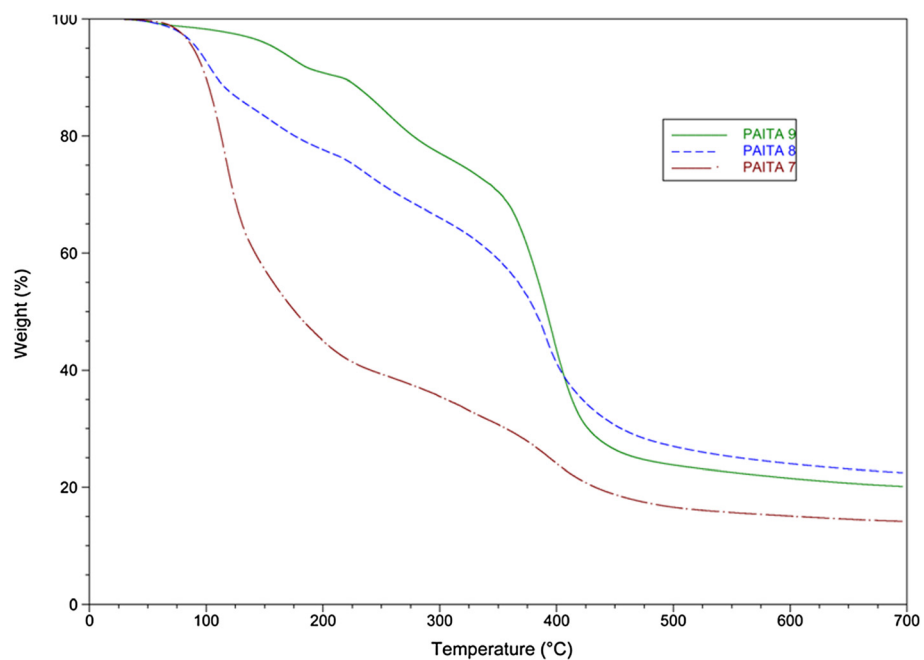


Figure 6 TGA thermogram of copolymers PAITA7-9.

Table 2 Information derived from TGA measurements of degradation of copolymers PMITA 1, 4-6 and PAITA 2, 7-9

Copolymer Code	T_{onset} (°C)	T_{max} (°C)	T_{50} (°C)	Residue (%)*
PMITA1	154	363	360	6.5
PMITA4	165	369	358	6.0
PMITA5	158	365	337	7.6
PMITA6	158	367	275	8.0
PAITA2	86	351	381	32.7
PAITA7	94	371	176	16.6
PAITA8	84	366	382	27.0
PAITA9	145	366	391	23.8

* At 500 °C.

1783 and 1860 cm^{-1} (C=O symmetric and asymmetric stretching of the 5-member anhydride unit), 1662 cm^{-1} (C=C stretching), and 1400 cm^{-1} (=CH₂ in plane deformation), and for methyl methacrylate at 1731 cm^{-1} (C=O) and for the acrylamide at 1640 cm^{-1} and at 3349 cm^{-1} originates from the secondary amines in acrylamide. The anhydride peaks indicate that the anhydride remained intact in the copolymer, and the shift to higher frequency (higher energy) of the ester and amide carbonyl stretch is consistent with the elimination of the conjugation of the vinyl and carbonyl double bonds.

The intensity of the anhydride peaks was increased as the ratio of ITA increased in the copolymer, Figures 1 and 2. Copolymers prepared by both conventional method and microwave irradiation showed the same spectral characterization.

Thermogravimetric Analysis (TGA)

The thermal properties of the prepared copolymers were evaluated by thermogravimetric analysis (TGA) Figures 3-6,

in which the weight of a sample is measured as a function of temperature whilst it is subject to a controlled heating program. Thermal results revealed that the prepared copolymers have high thermal stabilities. Structure thermal property correlation based on changing of the itaconic anhydride ratios, as a single structural modification, demonstrated an interesting connection between itaconic anhydride ratios and thermal properties.

For copolymers PMITA1 & PMITA5, the thermogram, Figure 3, showed that the prepared copolymers were degraded in four similar degradation steps. The first step with weight loss of 2.42% for conventional method at 70-115°C and 21.27% for microwave-assisted method at 130-225°C which is attributed to water evaporation and decarboxylation which is more clear in the prepared copolymers by microwave irradiation, respectively. The last stage exhibited subsequent major degradation process between 331-445°C (87.13%, wt loss) and between 331-445°C (80.71%, wt loss) for PMITA1 and PMITA5, respectively.

For the effect of the percentage of itaconic anhydride, Figure 4, the thermogravimetric data showed that the thermal stability of copolymer PMITA6 is higher than both copolymers PMITA4 and PMITA5 due to the increase in the itaconic anhydride content as a bulk group which leads to an increase in the thermal stability.

For copolymers PAITA2 and PAITA9, the thermograms, Figure 5, showed that they were degraded via two main stage degradation processes; the first step for copolymer PAITA2 is between 100 to 110°C with weight loss of 7.53%, while the last step occurs in a range of 380 to 440°C with weight loss of 62.39%. For copolymer PAITA9, we find the first degradation step is between 115 to 120°C with weight loss of 6.19%, while the last degradation step is between 490 to 445°C with weight

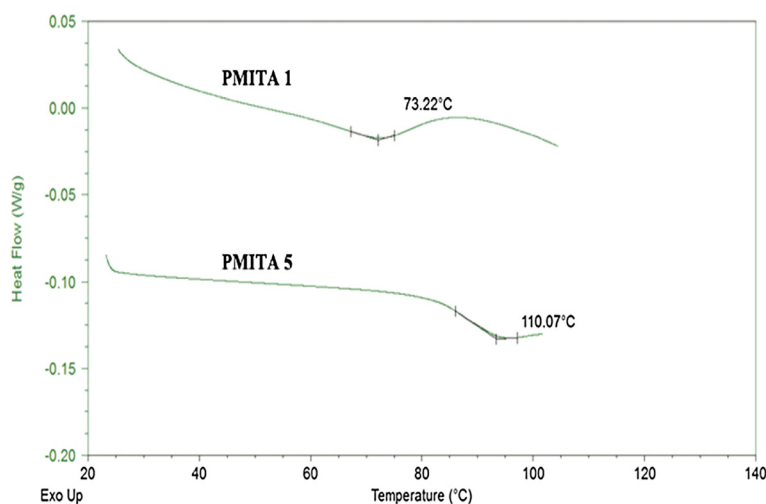


Figure 7 DSC thermogram of copolymers PMITA1 and PMITA5.

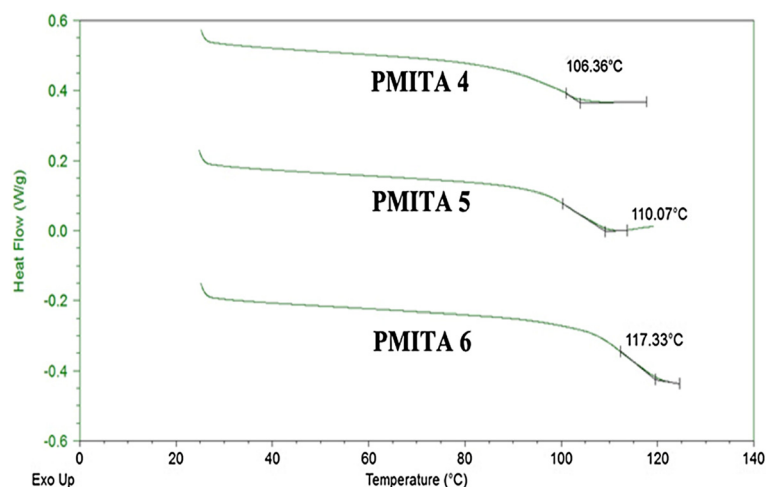


Figure 8 DSC thermogram of copolymers PMITA4-6.

loss of 61.68%. The thermogram showed the major weight loss within the temperature range 331–507°C and the temperature for a maximum decomposition was 559 and 661°C for copolymers PAITA2 and PAITA9, respectively.

For the effect of the percentage of itaconic anhydride, Figure 6, the thermogravimetric data showed that the thermal stability of copolymer PAITA9 is higher than copolymer PAITA7 due to the increase in the itaconic anhydride content as a bulk group which leads to an increase in the thermal stability as described earlier.

Considerable differences in the thermal decomposition of the prepared copolymers are observed in terms of degradation temperature are shown in (Table 2).

The copolymerization using microwave irradiation led to an increased T_{onset} of PMITA1 compared to PMITA5 which prepared by conventional method, however, T_{50} and T_{max} decreased. While on increasing the itaconic anhydride content (PMITA4-6), T_{onset} and T_{50} decreased and T_{max} are too close.

Similarly, PAITA2, 7-9, T_{onset} , T_{50} and T_{max} increased from PAITA2, which prepared by conventional method, to PAITA9, which prepared by microwave irradiation. The same conclusion was observed with increasing the itaconic anhydride content (PAITA7-9).

This could be explained by that microwave irradiation gave chance for increasing the itaconic anhydride content more than conventional method. As expected from the literature, the molecular weight of the copolymers

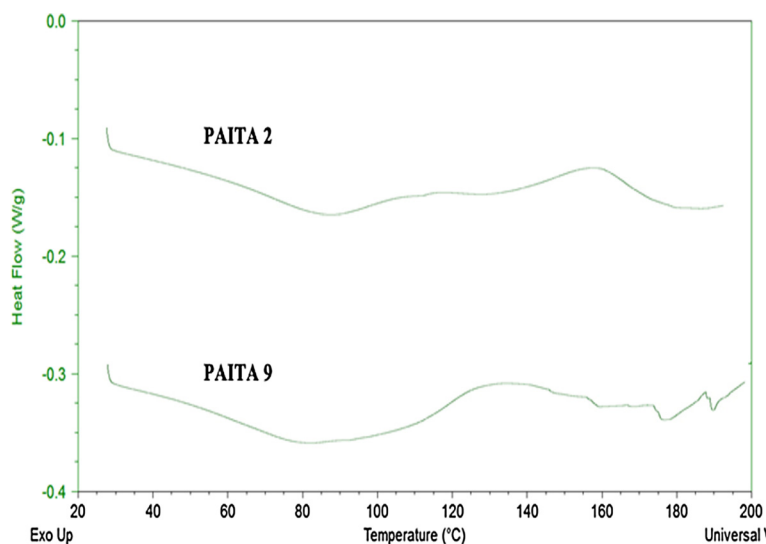


Figure 9 DSC thermogram of copolymers PAITA2 and PAITA9.

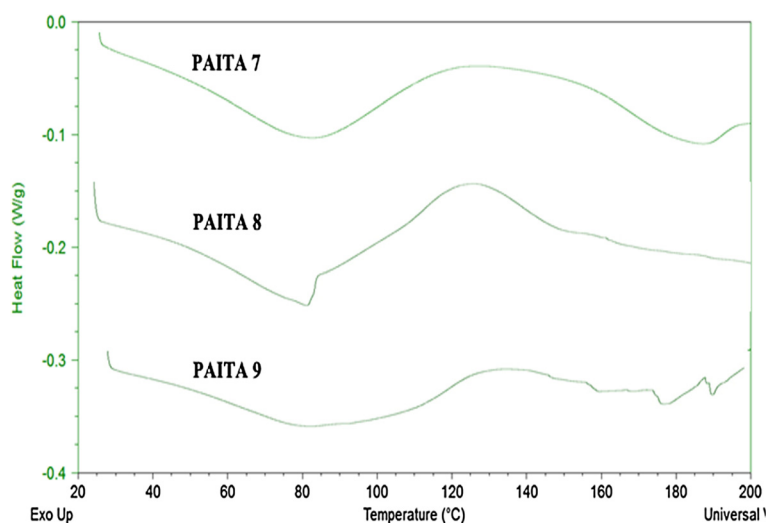


Figure 10 DSC thermogram of copolymers PAITA7-9.

decreased with increasing ITA concentration in the feed, which may be a consequence of the allylic hydrogen in ITA that can act as a chain transfer agent in radical polymerization [43,44].

Differential Scanning Calorimetry (DSC)

The effect of itaconic anhydride ratio in the prepared copolymers changes on the glass transition temperature (T_g) and the copolymer crystallinity upon thermal treatment were investigated by DSC at a heating rate of 10 °C/min, and the results are shown in Figures 7-10. The DSC traces, one transition was observed. The glass transition temperature (T_g) was found to be 73 and 110°C, for copolymers PMITA1 and PMITA5, respectively. It was noticed that T_g of copolymer PMITA5 is higher than that of copolymer PMITA1, which gives another evidence for enhancing the thermal properties by using the microwave technique due to increasing in the itaconic anhydride content. On the other hand the glass transition temperature (T_g) was found to be 106, 110, and 117°C, for copolymers PMITA4, PMITA5, and PMITA6, respectively, Figure 8.

The T_g increased as the ITA content increase due to the decrease in crystallinity [45].

Figure 9 showed the DSC thermograms of copolymers PAITA2 and PAITA9 copolymers prepared by conventional and microwave method. Both show no glass transition temperature, but copolymer PAITA2 shows a hump from 30 to 100°C, this hump is assigned to the evaporation of water attached to the polar amide groups NH_2CO - [46]. While for copolymer PAITA9 this hump starts from 30 to 120°C for the same reason.

Moreover, for the degradation process of the copolymer PAITA2, PAITA7-9 it is clear that the effect of the hydrogen bond so during the first degradation only dehydration followed by deamination process and this stage there is no differences between the prepared polymer by convention method and microwave irradiation while at higher temperature most of the hydrogen bond were broken and the difference in the thermal stabilities and the effect of the ITA was clear [47].

For AA/ITA copolymers series PAITA7-9 prepared by microwave method, the DSC thermograms, Figure 10, showed that copolymers PAITA7-9 has no glass transition

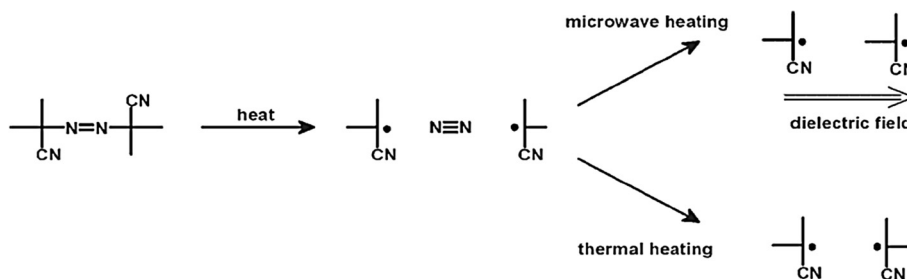
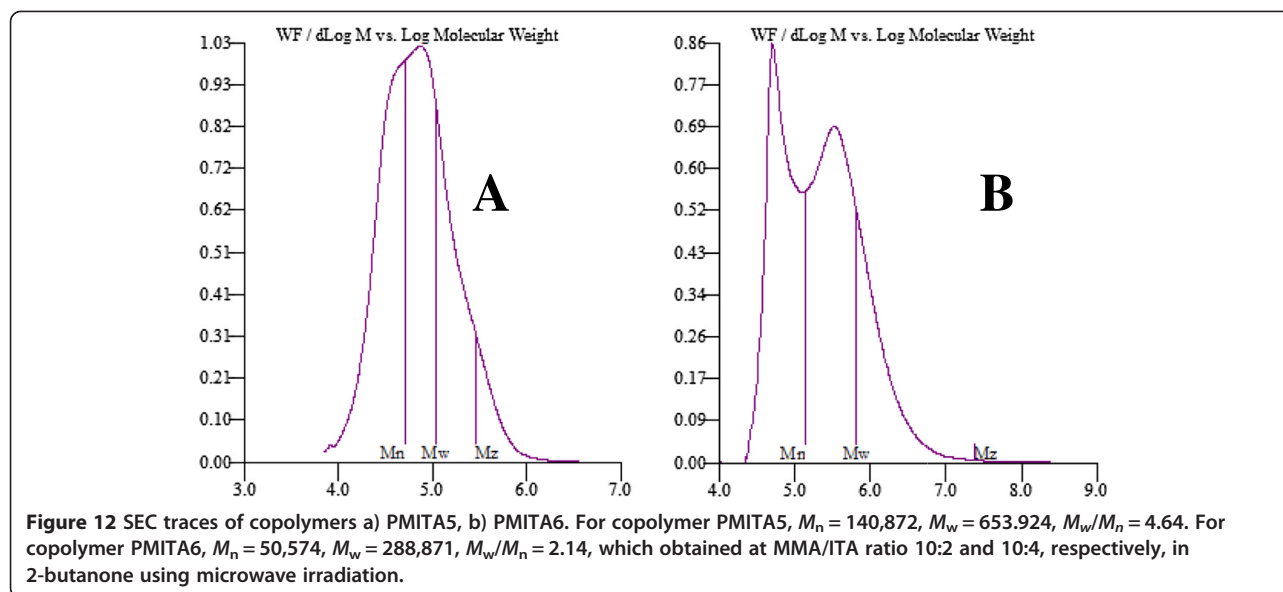


Figure 11 Schematic representation of the direct orientation of radicals that are formed from the decomposition of α,α' -azoisobutyronitrile (AIBN) under microwave irradiation [47].



temperature, the absence of T_g may be due to that itaconic anhydride makes a steric hindrance for the segmental motion of the copolymer, which lead to the absence of T_g .

Generally, the increase in the thermal stability for copolymers prepared by microwave irradiation technique compared to those prepared by conventional method is attributed to the increased radical flux under microwave irradiation as well as increasing of the itaconic anhydride content and prevention of the chain transfer in free radical polymerization. This result from the rapid orientation of the radicals that are formed from the decomposition of the α,α' -azobisisobutyronitrile as depicted in, Figure 11 [43,44,48], this orientation reduces the number of direct terminations via recombination of the formed two radical fragments under microwave irradiation and thus cause a higher radical flux. The obtained high radical flux leads to the formation of high molecular weight copolymers, which result in increase in their thermal stability of the copolymers synthesized by microwave irradiation method rather

than copolymers synthesized by conventional solution polymerization.

The molecular weight and Elemental microanalysis Determination

The molecular weight of itaconic anhydride copolymers have been studied, again it is obvious that the molecular weight decrease with increasing the ratio of itaconic anhydride in the copolymer with a significant change using microwave irradiation method due to the prevention of chain transfer and increasing of the itaconic anhydride in the copolymer composition (PMITA 5), Figure 12. For SEC measurements, SEC traces showed that for copolymer PMITA5, M_n was 140,872 g/mol with polydispersity 4.64, while for copolymer PMITA6, M_n was found to be 50,574 g/mol with polydispersity of 2.14.

Moreover Figure 12B not 12A showed a bimodal population in which the first one is related to the formation of itaconic anhydride homopolymer due to the increase in itaconic anhydride ratio.

In addition, the copolymers structures and ratios were confirmed by elemental microanalysis which in a good agreement with the calculated values (Table 3).

Table 3 Elemental microanalysis of the prepared copolymers using microwave irradiation

Copolymer code	Calculated (%)			Found (%)		
	C	H	N	C	H	N
PMITA4	59.3	7.6	-	58.6	7.8	-
PMITA5	58.8	7.2	-	58.3	7.6	-
PMITA6	58.0	6.6	-	55.2	5.5	-
PAITA7	51.1	6.6	17.0	48.4	7.0	15.9
PAITA8	51.4	6.5	14.4	49.3	6.6	14.1
PAITA9	51.8	6.0	11.7	49.8	6.4	9.8

Conclusions

Itaconic anhydride based copolymers with both methyl methacrylate and acrylamide with different ratios were successfully prepared using microwave irradiation technique. For comparison study, similar copolymers were prepared by conventional method. The obtained results showed the advantages of microwave irradiation method over conventional method. Microwave irradiation method offers the possibility of the preparation of copolymer in short time with high yield, high molecular weight and

high thermal stability. Moreover, as the itaconic anhydride content increases the thermal stability and T_g increase due to the decrease in the crystallinity.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

SMO carried out all the experimental work. MHE, SSE and AEF designed the proposed methods and analyzed the data statistically together. All authors read and approved the final manuscript.

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