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Synthesis, characterization and Study of the physical properties of some new silicone polymers

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Abstract

This study involves the synthesis of a new class of silicon polymers, designated as P1-P7, derived from dichlorodimethylsilane (DCDMS) in combination with various organic compounds (Schiff bases prepared from different amines and appropriate aldehydes or ketones) [I-V] through condensation polymerization. The structures of all monomers and polymers were characterization by FTIR and ^{1}HNMR spectroscopy (for some polymers). The results of thermogravimetric analysis (TGA) and differential scanning calorimetry DSC test show stable thermal behaviour. Polymers with a higher concentration of aromatic rings in their repeating structural units exhibited a higher temperature for weight loss, indicating increased thermal stability. Thermal measurements reflect the fact that all the polymers prepared in this study possess thermal stability, and the most thermally stable are the polymers that contain more phenyl rings. The inhibitory feature of the prepared polymers is studied through many tests, which include measuring the erosion rate through methods known as weight loss and scanning electron microscopy tests. In the weight loss method, the inhibiter gives good efficiency in protecting aluminium metal, to reach the inhibition efficiency to 83% using polymer P_5 inhibitor with concentration of 0.15 in 0.1 M solution of NaOH. On the other hand, P4 it showed the lowest inhibition efficiency of 16.74% at a concentration of 0.05. Scanning electron microscopy (SEM) images showed that a high corrosion inhibition efficiency of the polymers in NaOH solution (0.1 M), while the metal surface under the corrosion containing the inhibitors showed lower corrosion than that which could be found on the same metal surface, that is located in a completely empty media of the barrier and also clearly showed the protective layer on the surface. Viscosity testing in dimethylsulfoxide solvent showed that the true viscosity increases three fold when the concentration increases from 0.1 to 0.7. The results also showed that copolymer P_7 has a higher viscosity.

Keywords silicone polymers, monomers, dichlorodimethylsilane, thermal stability, imine polymers

تحضير ،تشخيص ودراسة الخواص الفيزيائيه لبعض بوليمرات السليكون الجديدة

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الخلاصة

تتضمن هذه الدراسة تحضير فئة جديدة من بوليمرات السيليكون P_1–P_7 مشتقه من ثنائي كلوروثنائي ميثيل سيلان (DSMS) بالاشتراك مع مركبات عضوية مختلفه (قواعد شيف التي حضرت من امينات مختلفه مع الديهايدات او كيتونات مناسبه) [I–V] من خلال بلمرة التكثيف .تم تشخيص تركيب جميع المونومرات والبوليمرات بتقنية FTIR وكذلك التحليل الطيفي ^{1}HNMR(لبعض البوليمرات) .اظهرت نتائج اختبارالتحليل الحراري الوزني (TGA) وتحليل قياس السعرات الحرارية DSC سلوك حراري مستقر. أظهرت البوليمرات ذات التركيز العالي للحلقات العطرية في وحداتها الهيكلية المتكررة درجة حرارة أعلى لفقدان الوزن ، مما يشير إلى زيادة الاستقرار الحراري .تعكس القياسات الحرارية حقيقة أن جميع البوليمرات المعدة في هذه الدراسة تمتلك ثباتا حراريا ، والأكثر استقرارا حراريا هي البوليمرات التي تحتوي على المزيد من حلقات الفينيل.تم تقييم الأداءالتثبيطي للبوليمرات المحضرة من خلال عدة اختبارات ، والتي تشمل قياس معدل التآكل من خلال الطرق المعروفة كفقدان الوزن واختبار الفحص المجهري الإلكتروني(SEM). في طريقة إنقاص الوزن ،اعطت المثبطات كفاءة جيدة في حماية معدن الألمنيوم من التاكل ، اذ اعطى المثبط البوليمري P_5 عند تركيز 0.15 كفاءة تثبيط تصل إلى 83٪ في محلول قاعدي بتركيز 0.1 مولاري من هيدروكسيد الصوديوم بدرجة حرار الغرفه ، اما P_4 فقد اظهر كفاءة تثبيط الاقل 16.74 % عند تركيز 0.05 . أظهرت صور الفحص المجهري الإلكتروني بوضوح كفاءة تثبيط التآكل العالية للبوليمرات في محلول هيدروكسيد الصوديوم (0.1 م) ، حيث أظهر السطح المعدني تحت التآكل المحتوي على المثبطات تآكلا أقل من ذلك الذي يمكن العثور عليه على نفس السطح المعدني ،والذي يقع في وسط فارغ تماما للحاجز وأظهر أيضا بوضوح الطبقة الواقية على السطح. أظهر اختبار اللزوجة في مذيب ثنائي ميثيل سلفوكسيد ان اللزوجة تزداد بثلاث اضعاف النسبه عند زيادة التركيزمن 0.1 الى 0.7 ،كما بينت النتائج ان البوليمر المشترك P_7 يمتلك اعلى لزوجه.

1. Introduction

Phenyl-containing silicone polymers have garnered significant attention in both academic and industrial spheres due to their exceptional characteristics. These materials derive their unique properties from the synergistic combination of organic and inorganic components within their molecular structure. This hybrid nature allows them to bridge the gap between organic and inorganic systems, effectively merging properties from both realms [1,2]..Hybrid polymers containing metal-inorganic connected to organic molecules through covalent bonds may show improved performances in different fields such as optics, electrical and luminescence properties and ionic conductivity in addition to the field of medicine [2-3] . In such a case the synthesis of hybrid polymers with silicone has attracted the attention of researchers due to their physicochemical properties such as thermal stability and environmental protection, therefore, they employed in many applications in many products as coatings, paints and adhesives. Phenyl rings are used in silicon polymers which have a sequential electron-π -system since they have been increasingly used in photoelectric industry, it has been found that conjugated compounds containing silicon have effective electron transfer properties due to the direct relationship between the electronic structure of conjugated heterogeneous systems and their photoelectric properties [2, 4-6]. Also, the introduction of phenyl groups units and different substituents into the main chains of silicone polymers could improve their thermal stability [7-12]. Researchers have been extensively focused on the search for materials with high thermal stability and the development of high-performance polymers to meet increasingly demanding requirements. In this context, silicone polymers have consistently proven to be a reliable choice for thermally stable materials. However, researchers used several methods to improve the thermal stability and resistance to high temperature, for example, nanoparticles such as SiO_2 and Al_2O have been used as filler to improve thermal stability [13-15]. Organic coatings often suffer from significant/minute defects, resulting in damage to the metal surface, corrosion of aluminium can be prevented with the use of protective coatings, the silicone-based coating is primarily a barrier coating

[16]. Silicones are frequently used as material of choice for different coating applications. The effectiveness of barrier coatings in preventing ingress and accumulation of moisture and corrosives at the substrate generally improves with better packing of polymeric chains. The silicone resin combines the advantage of inorganic ceramics with that of organic polymers. Compounds containing alkyl or phenyl groups typically exhibit favorable properties [16], also the use of copolymerization technique enables obtaining materials with properties intermediate between those of the individual homopolymers. Polymer dissolution plays a key role in many industrial applications in a variety of areas where, it has been proven that polymer dissolution is very important to several applications such as microlithography, membrane science, plastics recycling, and drug delivery, newer applications such as tissue engineering. Thus, the physical and chemical properties of the final material can be controlled through the appropriate choice of monomers that form the polymer chain [17]. Many researchers have conducted many works to improve the properties of Silicon polymers [10-12], the work they discovered that the introduction of phenyl groups units and different substituents into the main chains of silicone polymers could enhance the thermal stability and engineering application properties of silicone polymers, Also Yanhua Huang and his colleagues [18] were synthesized two types of silicone polymers: Methyl Phenyl and diphenyl silicone rubbers. The results showed that the introduction of phenyl groups can improve the thermal properties of silicone rubber, and the thermal stability of diphenyl silicone rubber is better than that of methyl phenyl silicone rubber. However, there is little difference of mechanical properties in high temperature environment. Manping Ma et al [19] and Songqi Songqi Zhang et al [20] supported the results showing that silicon-containing aryl acetylene resins with phenyl have good thermal and mechanical properties. While Zhang Li et. al. [21] and other researchers [22-25] worked on using a hybrid polysiloxane structure, which includes Schiff base structures which imparted a rigid-elastic nature to other resins, leading to remarkable mechanical properties. It not only significantly improved the impact strength but also contributed to the enhancement of thermal stability, Patricio A. Sobarzo et al [26] and Patricio A. Sobarzo [27] have been observed that silicone polymers-containing more effective π-conjugation across the main chain act as semiconductors promising for optoelectronic applications.

Silicone polymer has excellent mechanical properties and thermal stability and is widely used in mechanical manufacturing. As industrial technology advances, the disparity between the thermal stability of silicon polymers and the demands of industry is growing, prompting scientists to explore new avenues for improving the durability and thermal stability of these materials, particularly in the presence of oxygen.

The purpose of this work was to improve the thermal stability of silicone polymers through the introduction of phenyl groups and preparation of copolymers. In this work we report the synthesis of the polymers P_1-P_7 obtained through condensation polymerization and two of them new copolymers (P6) and (P_7). Spectrometric measurements in the infrared region exhibited the expected bands, demonstrating the successful synthesis of polymers and copolymers. Thermal analysis and viscosity were also carried out. Measurements of the corrosion by these materials suggest their potentiality for application in devices as anti-corrosion agents.

Experimental

Material and method

Materials

4-Hydroxyacetophenone99%, Benzidine 98%, Dichlorodi(methyl)silane, pyridine 99.8%, Glacial acetic acid 100%, 4-Hydroxy-3-methoxybenzaldehyde, Ethanol absolute99.5% Benzene-1,4-diamine 98%, 4-Aminoacetophenone99%,4-Aminophenol 98%, Dimethyl sulfoxide 99.9% and all the raw materials were supplied from Merck and SIGMA-ALDRICH CO.

Characterization Instruments

The FTIR spectrum of samples were recorded using a Shimadzo (Ir prestige-21) over the range 4000 – 600 cm^{-1} and Nuclear magnetic resonance ^{1}HNMR spectrometer were carried out by company: Bruker, model: ultra-shield 400 MHz, Differential scanning calorimetry (DSC) and Thermal gravimetric analysis (TGA) were taken for silicon polymers and copolymers using DSC-60A, Serial No: C30955700083SA Shimadzo under Air atmosphere and temperature range 0-600 °C with a heating rate of 10 °C/min ,The scanning electron microscopy (SEM) via MIRA3 device.

Preparation of Monomers I-V

The monomers I-IV were prepared from the refluxing a mixture of substituted-benzaldehyde (0.02 mol) with the (0.01 mol), from different amino compounds (while the monomer V was prepared using the 0.01 mol from amine and ketone), 2 drops of glacial acetic acid and absolute ethanol (10 mL) for (6) hrs. Once placed under vacuum conditions, the solvent was removed by evaporation. The remaining residue was then induced to form crystals by dissolution in methanol [28, 29]. Fig.1shows the chemical structure of the prepared monomers (I-V), the physical data and Nomenclature of the prepared monomers I-V are listed in Table1

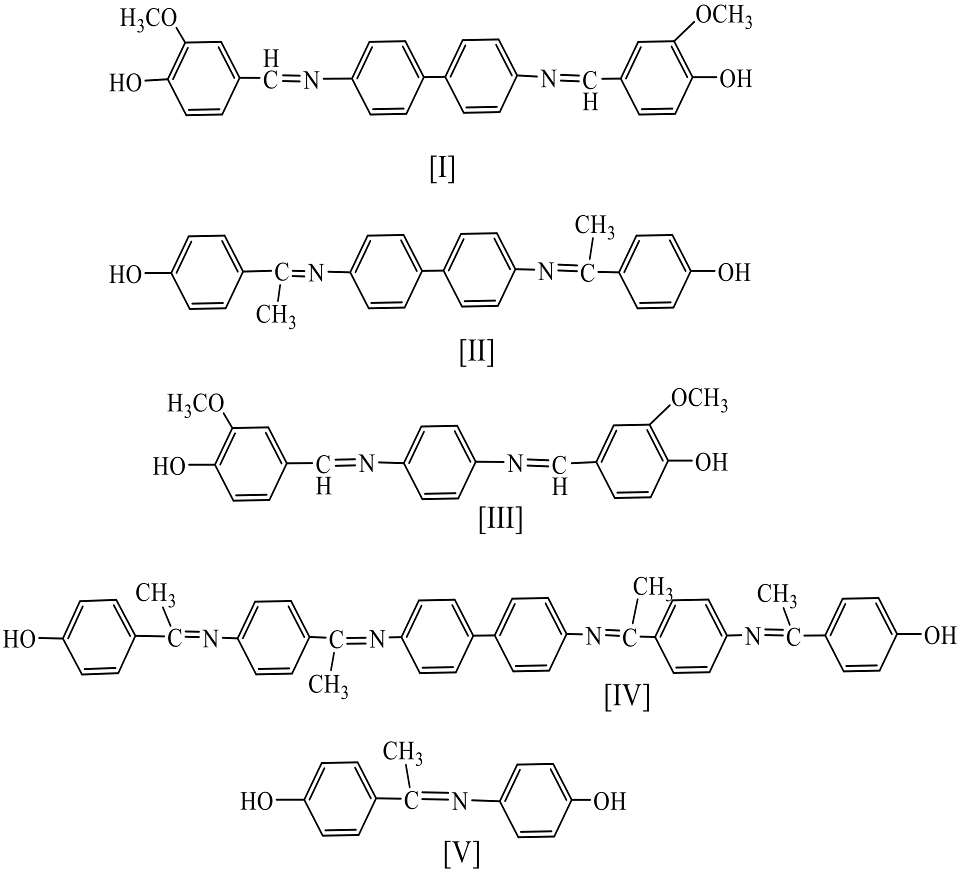


Figure 1: The chemical structure of the synthesized monomers I-V

Table 1: Summary of physical properties for monomers (I-V)

No.monomer	Nomenclature	Molecular formula	color	Melting point	Yield
I	4,4'-(([1,1'-biphenyl]-4,4'-diylbis(azaneylylidene))bis (methaneylylidene))bis(2-methoxyphenol)[	$C_{28}H_{24}O_4N_2$	orange	229-232	86%
II	4,4'-(([1,1'-biphenyl]-4,4'-diylbis(azaneylylidene))bis(ethan-1-yl-1-ylidene))diphenol	$C_{28}H_{24}O_2N_2$	orange	280 compos	79%
III	4,4'-((1,4-phenylenebis(azaneylylidene))bis(methaneylylidene))bis(2-methoxyphenol	$C_{22}H_{20}O_4N_2$	Dark brown	250-254	66%
IV	4,4'-((((([1,1'-biphenyl]-4,4'-diylbis(azaneylylidene))bis(ethan-1-yl-1-ylidene))bis(4,1-phenylene))bis(azaneylylidene))bis(ethan-1-yl-1-ylidene))diphenol	$C_{44}H_{38}O_2N_4$	Light green	71-76	76%
V	4-((1-(4-hydroxyphenyl)ethylidene)amino)phenol	$C_{14}H_{13}O_2N$	brown	182-187	84%

Synthesis of polymers [P_1-P_5]

These silicon polymers were synthesized by the condensation reaction (0.1 mol) of one of the monomers (I-V) in dry benzene with (0.1 mol) Dimethyldichlorosilane in the presence of 0.5mL pyridine with stirring under a temperature between (0 - 4) °C in an ice water bath for 48 hrs. The resulted solid was poured onto 10 mL solution cold from dilute 5% HCl, then the precipitate was filtered, dried and washed by use methanol [30]. Fig.2 illustrates general formula for synthesized polymers.

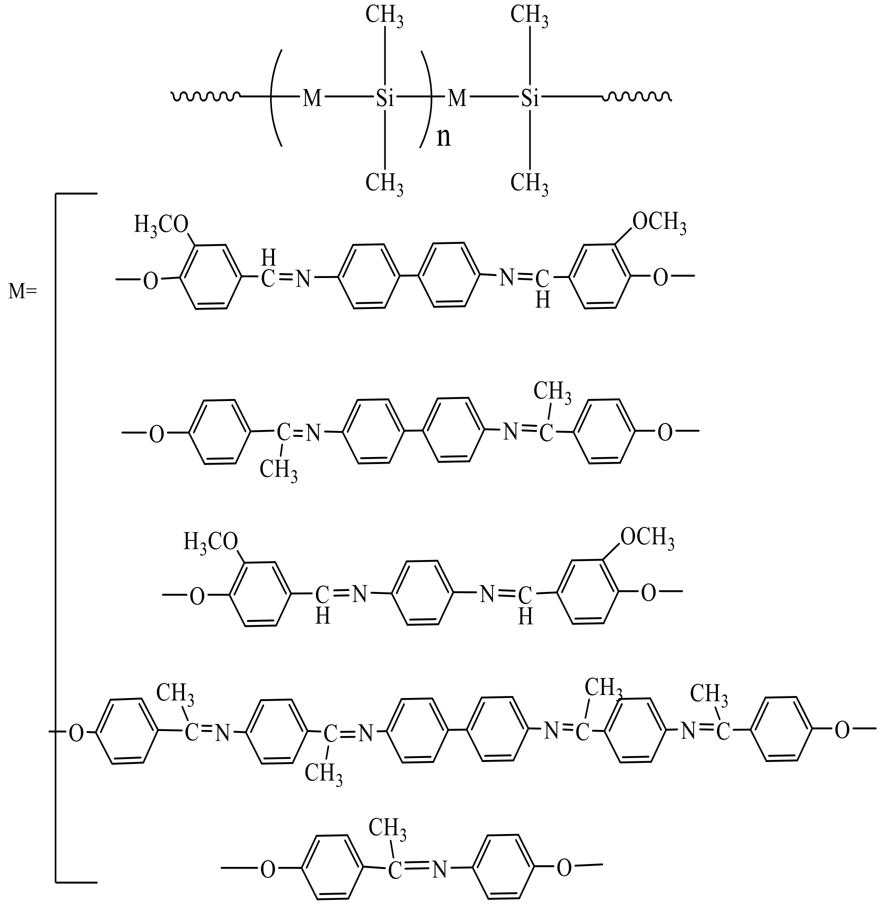


Figure 2: Structure of silicon polymers [P_1-P_5] with different monomers [I-V]

Synthesis of copolymers [P_6, P_7]

The copolymers P_6 and P_7 were prepared using a modified procedure similar to that employed for the synthesis of polymers P1-P5, with the exception that two monomers were used with DCDMS at a mixing ratio of 1:1:2 mol. monomers I,II with DCDMS and monomers III,V with DCDMS to synthesis copolymers P_6 and P_7 [30], respectively. Fig.3. illustrates general formula for synthesized copolymers P_6 and P_7 and the physical data of the synthesized polymer P_1-P_5 and copolymer P_6, P_7 are listed in Table 2

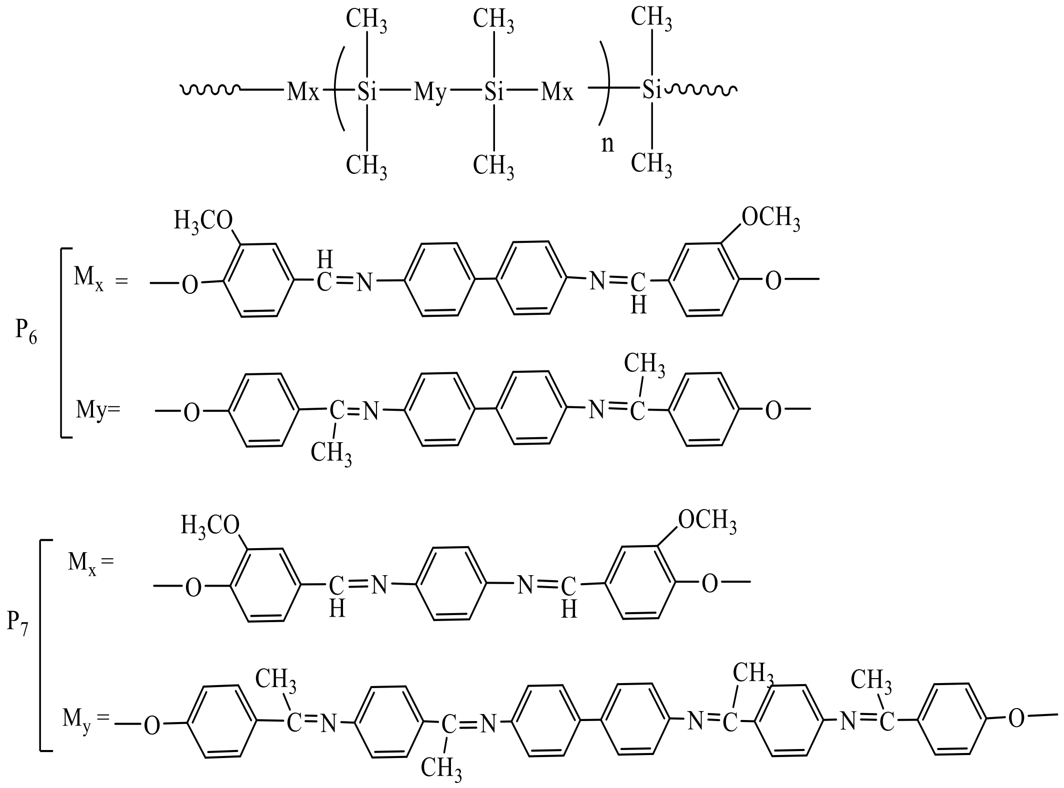


Figure 3: Structure of silicon copolymers P_6, P_7 with different monomers M

Table 2: Summary of physical properties for polymers (P_1-P_7)

NO. polymer	Color of polymer	Softing point of polymer
P_1	Reddish orange	273-275
P_2	brown	>300
P_3	Dark brown	205-210
P_4	Brown	234-240
P_5	Brown	208-210
P_6	Dark red	>300
P_7	Brown	>300

Viscometry

The average molar mass of a polymer is a crucial parameter that characterizes the average length of the polymer chain. This measurement is of great importance in studying the behavior of polymers, as the physical and chemical properties of the polymer are strongly influenced by the average chain length. The average chain length is very important in polymer processing and its overall performance. The method of dissolving a polymer in a suitable solvent is one of the most important scientific methods in calculating the molecular weight of a polymer. For this reason, by measuring the viscosity the molecular weight can be calculated using the Mark–Houwink equation [31]. The solubility of the polymer chain in the solvent has an impact on its rheological behavior, and a number of factors control the nature of the flow of the polymeric solution, making viscosity measurements are difficult to interpret. For this reason, viscosity measurements are performed using dilute solutions to minimize interactions between polymer molecules [32, 33]. The viscosity of the polymers was measured using Ostwald viscometer at 25°C temperature.

Measurement of corrosion inhibition

Corrosion is a widespread and expensive problem that threatens the service life of metals. Although organic coatings are applied to mitigate corrosion, which can provide a strong physical barrier against the penetration of corrosive media. However, organic coatings often suffer from significant/minute defects. Corrosive substances can thus reach the metal substrate through coating defects. In order to achieve long-term corrosion protection, the possibility of using prepared polymers as corrosion inhibitors to produce an active protective performance that can independently repair corrosion resistance when the coating is damaged has been verified [16,34]. The chemical material was used in this work is aluminium. The chemical composition of aluminium was: Si,0.007; Fe,0.2; Cu,0.007; Mn,0.004; Mg,0.001; Zn,0.001; V,0.005 and the rest was aluminium. In sodium hydroxide medium, the corrosion of metal happening by the action of local cells, which are established by metal, consisting a partial anodic reaction and a partial cathodic reaction occurring simultaneously on the metal surface [35].

$$Al + 3OH^- \longrightarrow Al(OH)_3 + 3e^- \text{ ------ } (1)$$

$$Al(OH)_3 + OH^- \longrightarrow Al(OH)_4^- \text{ ------ } (2)$$

A partial anodic dissolution reaction of aluminum in alkaline solution can be obtained by combining electrochemical film formation (1), and chemical film dissolution (2), which can be written as:

$$Al + 4OH^- \longrightarrow Al(OH)_4^- + 3e^- \text{ -------} (3)$$

The electrons produced by the partial anodic reaction (3) will be consumed immediately by such partial cathodic reactions as the oxygen reduction reaction.

$$3/4\ O_2 + 3/2\ H_2O \longrightarrow 3e^- + 3OH^- \text{ -----} (4)$$

The corrosion reaction of aluminum in alkaline solution can be obtained by combining reactions 3 and 4

$$Al + OH_3^- + 3/4\ O_2 + 3/2\ H_2O \longrightarrow Al(OH)_4^- \text{ ------ } (5)$$

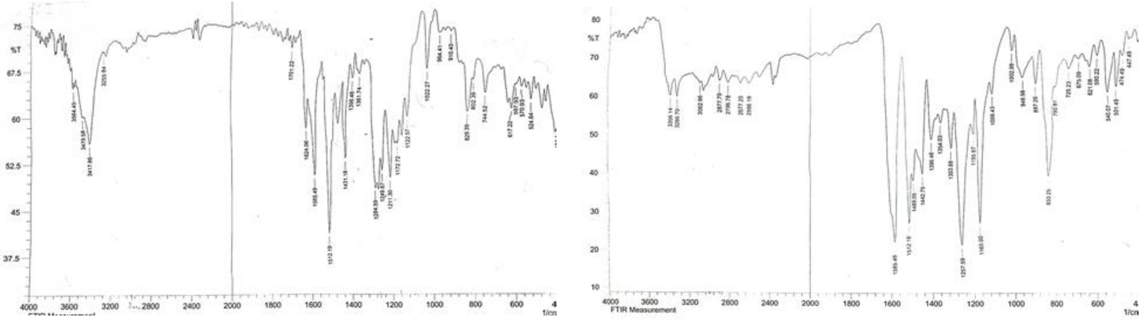
As a consequence of the overall corrosion reaction (5), aluminum metal dissolution occurs. The gravimetric approach (i.e. the weight loss) is the most widely used method of evaluate inhibition due to the reliability and simplicity of measurement, making it an essential measurement method used to evaluate inhibition, polymers concentrations were expressed using the percentage of concentration: mass to volume (m/ v%). In the weight loss experiment, which have been carried out under the total immersion at the room temperature, four 250 ml beakers containing (100 ml) of 0.1M NaOH solutions with 0,0.05, 0.1 and 0.15 % from polymer weight as additives respectively. The aluminium samples were cut into circular shapes of 2 cm as a diameter and a thickness of 3 mm with areas equal to 0.000628 dcm^2, then a small hole with a diameter of 2 mm was drilled into each sample, the surfaces of the body were slightly polished to remove traces of contaminants and achieve a flat surface at each of the cut edges and following that all the samples were then placed in beakers .The samples were retrieved from their corroding solutions at a one-hour period, a period of 7 hours, after each hour, it was cleaned using distilled water, dried then reweighed each time.

Results and Discussion

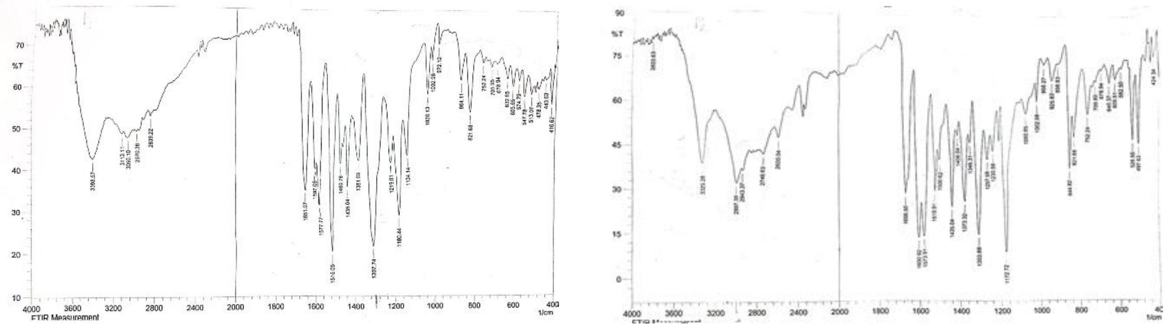
Characterization of the Silicone polymers

FTIR-Spectroscopy

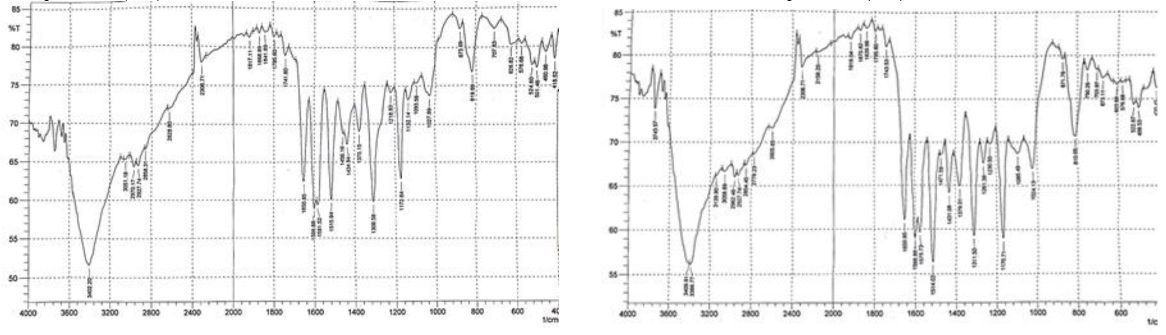
A characteristic broad peak was observed in the range of 968-972 cm-1 for all silicone polymers, corresponding to the Si-O-ph bond, which confirms the presence of a Si-O-ph backbone in the polymer structure. The characteristic peak near 1408-1473 cm^{-1} indicated the presence of Si-CH_3, and the peak around 2900-2970 cm^{-1} belonged to the C-H bond of Si-CH_3 groups. While, the absorption peak around 3000-3059 cm^{-1}, which belonged to the C-H bond of the benzene ring in silicone polymers, in addition a stretching band [20] for C=N appeared between 1604-1666 cm^{-1}**.** The characteristic FT-IR absorption bands for monomers I-V and polymers P_1-P_5 with copolymers P_6,P_7 were listed in the Table 3.[11,30,36] and Fig.4 shows the FTIR spectra of the monomer I,II , silicone polymers P_1 , P_2 and copolymers P_6, P_7 with different content of the organic monomers.



monomer (I) monomer (II)



Polymer (P_1) Polymer (P_2)



Copolymer (P_6) Copolymer (P_7)

Figure 4: the FTIR spectra of the monomer I,II , silicone polymers P_1 , P_2 and copolymers P_6, P_7

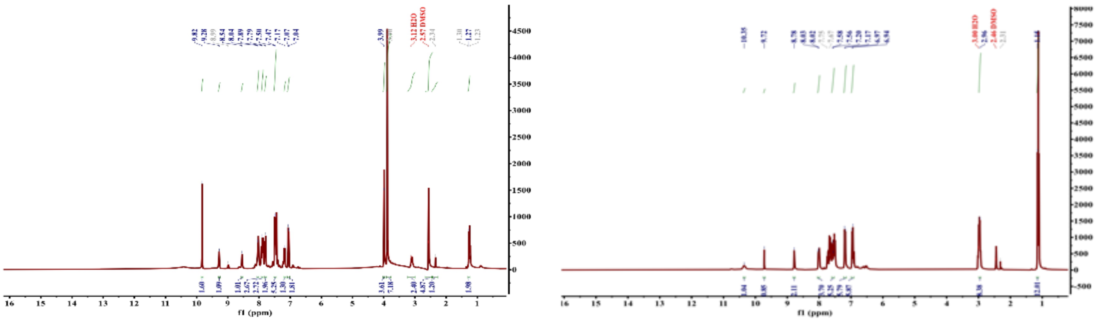
Table 3: Summary of infrared bands (cm^{-1}) for stretching band of monomers, Silicone polymers and their copolymers

Compound No.	OH cm^{-1}	C-H arom. cm^{-1}	C-H aliph. cm^{-1}	C=N cm^{-1}	C=C cm^{-1}	Si-CH3 cm^{-1}	C-O-C cm^{-1}	Si-O-ph cm^{-1}
I	3417	3000	2900,2800	1624	1585	—	1249,1122	—
II	3356	3062	2900,2877	1600	1585	—	—	—
III	3352	3005	2939,2877	1604	1593	—	1257,1099	—
IV	3332	3008	2954,2836	1620	1597	—	—	—
V	3340	3062	2900,2800	1602	1589	—	—	—
P_1	3398	3059	2970,2839	1651	1597	1450,1275	overlap,1134	972
P_2	3352	3000	2943,2890	1666	1600	1408,1230	—	968
P_3	3332	3050	2947	1604	1593	1473,1230	1261,1099	970
P_4	3352	3010	2935	1647	1598	1473,1261	—	968
P_5	3265	3026	2940,2800	1602	1589	1400,1230	—	960
P_6	3402	3051	2970-2858	1650	1598	1458-1218	1260-1093	940
P_7	3386	3058	2962-2854	1650	1598	1471,1230	1261-1095	910

^{1}HNMR-Spectroscopy

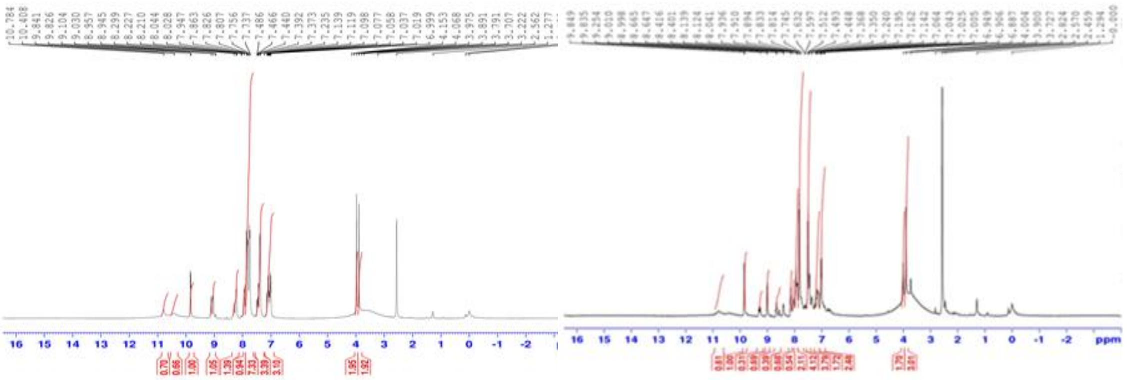
The ^{1}HNMR spectrum (in DMSO) for polymer P_1 showed the following signals: two singlet signals at δ 9.82ppm and δ 9.28ppm could be attributed for protons of OH in end of chain and protons of CH=N group, respectively, many signals in the region δ (8.99-7.04) ppm for protons of benzene rings, singlet at δ 3.99 ppm that attributed for 6 protons of $2OCH_3$ protons [37] and a signal at δ 1.27 ppm for to $SiCH_3$ protons. The ^{1}HNMR spectrum (in DMSO) for polymer P_4 exhibited two signals in δ 9.72 ppm and in the region δ (8.03-6.94) ppm that attributed to OH protons [30] and benzene rings, respectively. Besides to two singlet signals at δ 2.96 ppm and δ 1.15 ppm could be attributed for CH_3-C and $SiCH_3$ protons [11, 38].

The ^{1}HNMR spectrum (in DMSO) for copolymer P_6 showed the following signals: two signals at δ 10.784 ppm and δ 9.841 ppm could be attributed for protons of OH, and the CH=N protons appeared at δ 9.104ppm, multiple signals between δ (8.945-6.999) ppm and singlet at δ 3.97 ppm that attributed for protons of benzene rings and protons of OCH_3 group, respectively. Other signals appeared at δ 2.562ppm for $N=CCH_3$ and at δ1.277 ppm for to $SiCH_3$. The ^{1}HNMR spectrum for copolymer P_7 showed the following signals: two singlet signals at δ10.8ppm and δ 9.849 ppm could be attributed for protons of OH and at δ 9.254 ppm for CH=N protons, signals in the region δ (8.998-6.949) ppm that attributed for protons of benzene rings and singlet at δ 3.90 ppm and δ 2.56 ppm could be attributed for protons of OCH_3 and $N=CCH_3$ protons, respectively. Finally, a singlet signal appeared at δ 1.294ppm could be attributed for protons of $SiCH_3$. Fig.5 shows the HNMR spectra of the synthesized silicone polymers P_1, P_4 and Fig. 6 shows copolymers P_6 and P_7



Polymer (P_1) Polymer (P_4)

Figure 5: the ^{1}HNMR spectra of the silicone polymers P_1, P_4

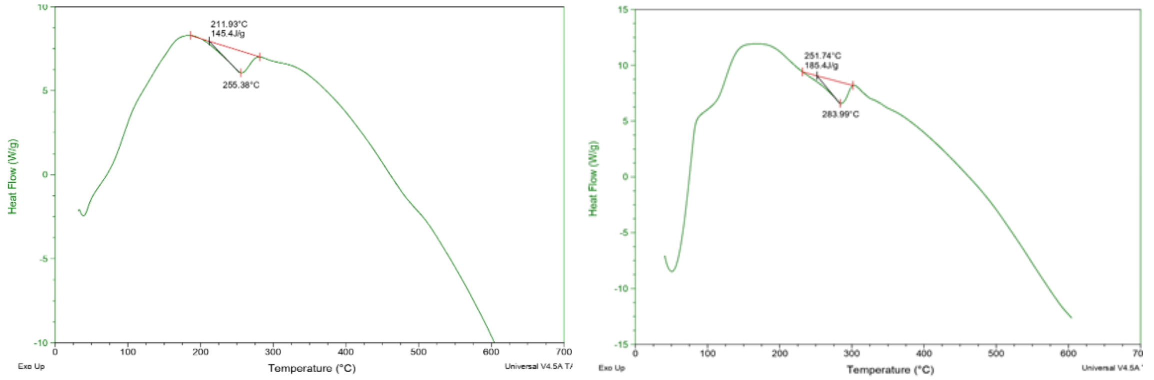


Copolymer (P_6) Copolymer (P_7)

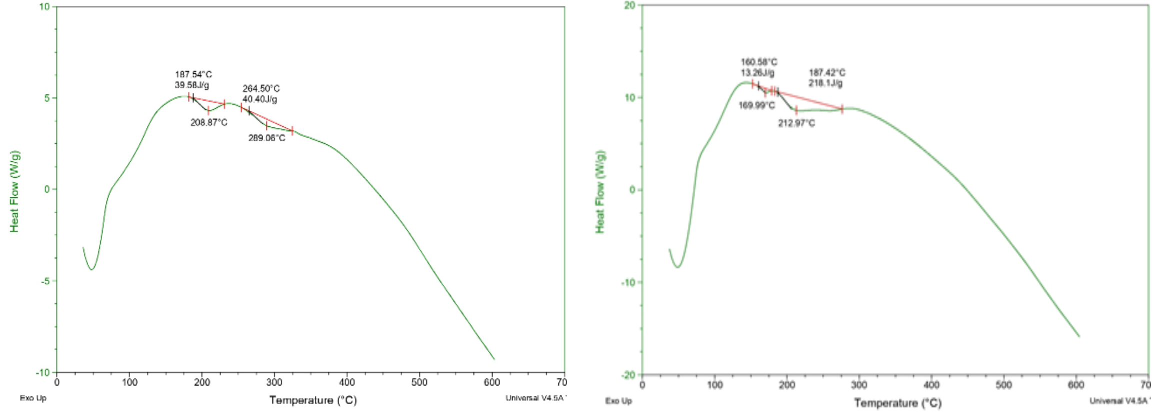
Figure 6: the ^{1}HNMR spectra of the silicone Copolymers P_6, P_7

DSC and TGA

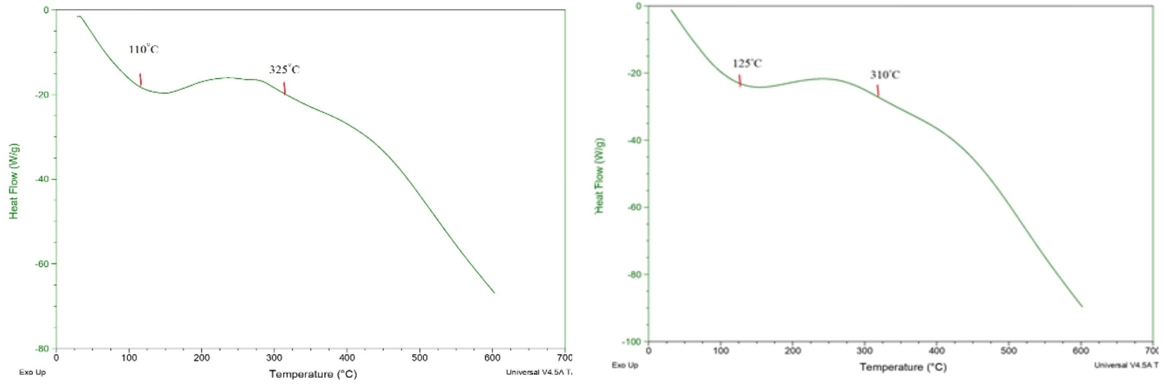
The DSC scan of silicon polymers and their copolymers showed a similar thermal degradation trend for most polymers. Physical glass transformations (Tg) for silicone polymers appeared in the range of 160- 251ºC and in 110 ºC, 125ºC for silicon copolymers P_6 and P_7, respectively as shown in Fig.7. The thermogravimetric analysis (TGA) of silicone polymers showed two to four main weight loss stages. The first stage at around 100ºC with a weight loss of 3-6% for polymers P_2, P_3, P_4 while polymer P_1 showed a weight loss of 31%, at 160ºC, which may be due to evaporation of moisture content, it was observed that the main slope of the weight loss curve occurred in the range of 200- 300ºC for P_1, P_2, P_3 and P_4. Although the structure of the repeating units of polymers P_1 and P_3, P_2 and P_4, is very similar, the main slope in the weight loss curve occurs at different temperatures, possibly due to the increased number of aromatic rings of the polymer P_1 relative to the polymer P_3 and also relative to the polymers P_2 and P_4 which led to an increase in the hardness of the polymer. Copolymers exhibited three distinct stages of weight loss, with the initial stage beginning at approximately 100ºC and resulting in a 6% weight loss. The most significant weight loss occurred at 310°C for copolymer P_6 and at 300°C for copolymer P_7. It was concluded that copolymerization has an effect on the thermal stability of the polymers. It was found that the onset temperature for degradation of the copolymers is lower due to a decrease in crystallization and an increase in the amorphous structure for copolymers [39, 40]. Thermogravimetric analysis shows that the copolymer samples P_7, P_6 have larger residues at 600 °C, that may be related to the presence of the increase of phenyl rings in the one structural repeating unit of the polymer, which gave greater thermal stability to polymer P_6 and P_7 compared to other polymers, this result agrees well with those given by Z. Yang and et al. [11] The Fig.8 shows the thermalgravimetric analysis of the polymers P_1 - P_4 and copolymers P_6, P_7, the result are listed in Table 4.



Polymer(P_1) Polymer(P_2)

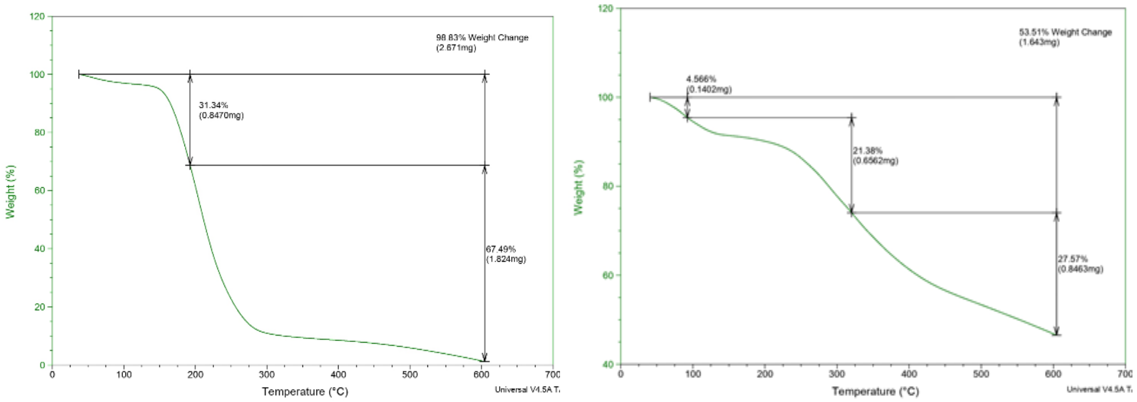


Polymer(P_3) Polymer(P_4)

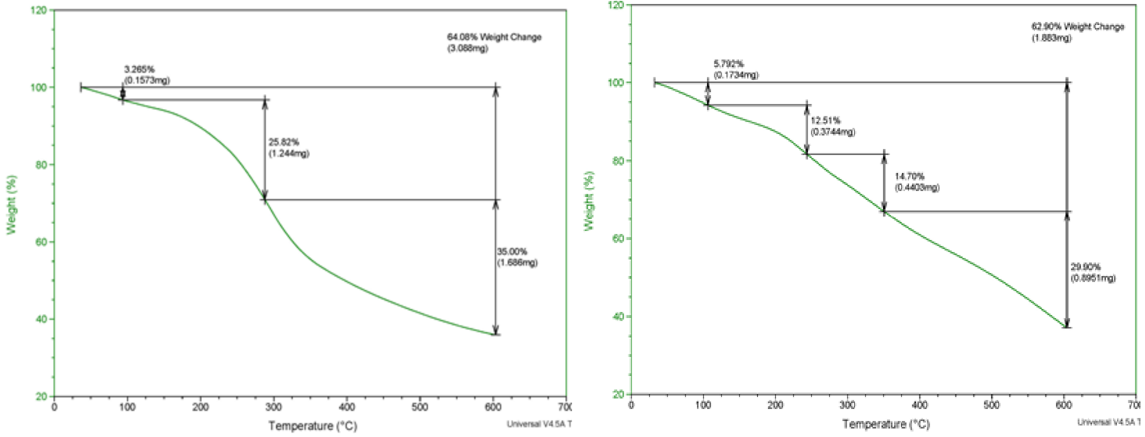


Copolymer(P_6) Copolymer(P_7)

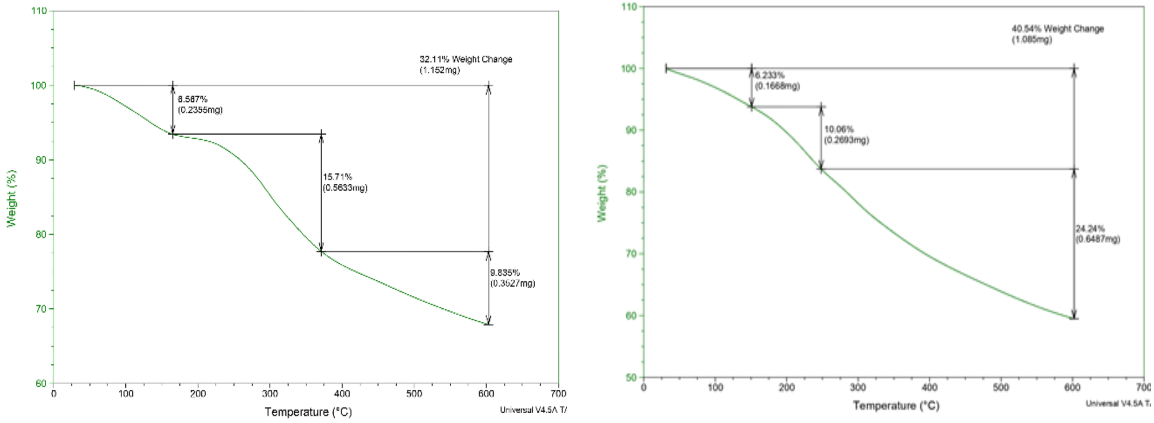
Figure 7: The DSC curves of the silicone polymers P_1-P_4 and copolymers P_6, P_7



Polymer (P_1) Polymer (P_2)



Polymer(P_3) Polymer(P_4)



Copolymer (P_6) Copolymer (P_7)

Figure 8: The TGA curves of the silicone polymers P_1-P_4, and copolymers P_6, P_7

Table 4: The DSC and TGA results of the silicone polymers P_1-P_4 and co polymers P_6, P_7 (T_g: glass transition temperature, T_c: Crystallization temperature, T_m: Melting temperature, T_d: Degradation temperature T_i: initial temperature of degradation, T_f : final temperature of decomposition, LW: Percentage of weight loss at600℃

compound	DSC				TGA			
	T_g °C	T_c °C	T_m °C	T_d °C	step	T_i °C	T_f °C	LW%
P_1	211.93	286	312	550	1	50	205	-98.83
					2	205	600	
P_2	251.74	305	325	550	1	55	100	-53.51
					2	100	350	
					3	350	600	
P_3	187.54	264.50	289.06	550	1	50	110	-64.06
					2	110	285	
					3	285	600	
P_4	160.58	187.42	212.97	550	1	45	112	-62.90
					2	112	175	
					3	175	600	
P_6	110	230	325	550	1	40	67	-32.11
					2	67	370	
					3	370	600	
P_7	125	250	310	550	1	35	150	-40.54
					2	150	250	
					3	250	600	

Viscosity measurement and molecular weight

Different concentrations of P_1-P_7 were prepared in DMS solvent as shown in Table 5 which was used to measure various viscosities including relative viscosity (ηrel), specific viscosity (ηsp), and reduced viscosity (ηred) using the following equations 1, 2, and 3, respectively. Where (t) is the flow time of the polymer solution, (t_0) is the flow time of the pure solvent and C is the concentration of polymer [32]. In order to ensure the accuracy of the results, the experiment was repeated 3 times for each solution.

$$\eta rel = t/t_{\circ} \tag{1}$$

$$\eta sp = t/t_{\circ} - 1 = \eta rel - 1 \tag{2}$$

$$\eta red = \eta sp\ /C \tag{3}$$

by drawing the relationship between the reduced viscosity versus the concentration, we obtain a straight line whose intersection represents the intrinsic viscosity [η] and using the Mark–Houwink equation the viscous molecular weight of the prepared polymers can be calculated.

$[\eta] = KM^a$................ Mark–Houwink equation

Where K, a are constants, their values differ by the types of the polymer and solvent, M= molecular weight. The molecular weights of polymers were also measured using a gel chromatographic device and were found that they ranged from 18484 g/mol to 630589 g/mol. The copolymer P_7 showed the highest viscosity, while the polymer P_5 had a lower viscosity. The results may be related to the presence and number of substituted groups [30], as some of the repeating units contain terminal groups (CH_3) and (OCH_3) that hinder the agglutination of the polymer chains and thus lead to a decrease in viscosity. Also, the presence of a larger number of aromatic rings within the repeating unit increases the agglutination of the polymer chains and thus increases the viscosity and molecular weight and this result is in good agreement with those previous reports [31]. Table 5 shows the values for the types of measured viscosities.

Table 5: Summary of relative viscosity (ηrel), specific viscosity (ηsp), and reduced viscosity for polymersP_1-P_7

Concen-tration ppm	**P_1**			**P_2**			**P_3**		
	ηrel	**ηsep**	**ηred cm^3g^{-1}**	**ηrel**	**ηsep**	**ηred cm^3g^{-1}**	**ηrel**	**ηsep**	**ηred cm^3g^{-1}**
0.1	1.1611	0.1611	1.6113	1.1421	0.1421	1.4218	1.0663	0.0663	0.6635
0.3	1.7914	0.7914	2.6382	1.7630	0.7630	2.5434	1.2843	0.2843	0.9478
0.5	2.7440	1.7440	3.4881	2.6682	1.6682	3.3364	1.6113	0.6113	1.2227
0.7	4.0047	3.0047	4.2924	3.9763	2.9763	4.2518	1.9905	0.9905	1.4150

concen-tration ppm	**P_4**			**P5**			**P_6**		
	ηrel	**ηsep**	**ηred cm^3g^{-1}**	**ηrel**	**ηsep**	**ηred cm^3g^{-1}**	**ηrel**	**ηsep**	**ηred cm^3g^{-1}**
0.1	1.1848	0.1848	1.8483	1.04265	0.0426	0.4265	1.2796	0.2796	2.7962
0.3	1.8009	0.8009	2.6698	1.2322	0.2322	0.7740	1.9431	0.9431	3.1437
0.5	2.7488	1.7488	3.4976	1.5165	0.5165	1.0331	2.938389	1.9383	3.8767
0.7	4.0284	3.0284	4.3263	1.9431	0.9431	1.3473	4.1706	3.1706	4.5294

concen-tration ppm	**P_7**		
	ηrel	**ηsep**	**ηred cm^3g^{-1}**
0.1	1.3696	0.3696	3.6966
0.3	2.1563	1.1563	3.8546
0.5	3.1090	2.1090	4.2180
0.7	4.2891	3.2891	4.6987

Corrosion inhibition

Fig.9 shows the corrosion of aluminium at a concentration (0.1 M NaOH) with and without polymer concentrations (0.05, 0.1, and 0.15) w/v%, respectively. The results obtained indicate that the weight loss of samples immersed in 0.1 M NaOH solutions increased with increasing immersion hours to 4 hours then the loss weight began to fade gradually, this can be attributed to the fact that the chemical reaction rate increases with increasing hours of immersion. Fig. 9 reveals that the polymers prevented the corrosion of aluminium in NaOH solutions, as there was a general decrease in weight loss with increasing polymer concentration.

The corrosion rate of aluminium metal in different concentration solutions was determined by using the formula (1) [41]:

$$\text{Corrosion Rate (mdd)} = w/\,A\,t \qquad (1)$$

Where W= weight loss of aluminium metal (mg) in the presence and absence of inhibitor (polymer) respectively in a solution 0.1M of NaOH under the same conditions
A = area of sample (dcm^2)
t = exposure time (hrs.)

The % inhibition efficiency was calculated by using following formula (2) [34,41]:

$$I.E\% = \{1- CR_1/ CR_2\} *100 \tag{2}$$

Where CR_1 and CR_2 =Corrosion rate of aluminium in the presence and absence of an inhibitors. It was found that the maximum inhibition efficiency (84%) was obtained from inhibitor P_5 at a concentration of 0.15, this is due to the small size of the repeating unit in polymer P_5 compared to other polymers, which makes it possible to adsorb more inhibitor molecules on the metal surface. The percentages of inhibition efficiency were higher at a concentration of 0.15 than at 0.05% for all inhibitor polymers, moreover, the corrosion rates were much lower than without the inhibitor, this minimum corrosion rate is due to the adsorbent layer (inhibitor) on the surface of the sample, which protects against corrosion, the efficiency of an organic compound inhibitor is mainly dependent on the functional groups and π electrons in their structure, the charge density around nitrogen and oxygen atoms ,Schiff bases are compounds that contain (–C double bond N–) groups in their own structures. These groups are donors and can form stable complexes on the metal surface [42]. This result agrees well with those given by J. Arjomandi and et al. [43] Scanning electron microscopy test (SEM) was performed to study samples surface topography, which immersed in solutions with and without silicone polymers. Fig.10 (a, b) shows sample after immersion test in 0.1 M NaOH solution without inhibitor. It seems clearly the dense pitting corrosion with different sizes due to surface corrosion and the effects of hydroxyl ions. Whereas Fig. 11(1-7) shows the samples surface after immersed in 0.15 wt./ vol. % silicon polymers with 0.1 M NaOH. It is obvious the large differences in both cases due to the activity of adsorbed inhibitor layer (silicone polymer) at the sample surface which protected against corrosion. Table 6 shows the weight loss, corrosion rate CR and inhibition ratio efficiency in 0.1M NaOH solutions in the absence and presence of polymers P_1-P_7 at concentration values (0.05, 0.1, and 0.15) for all polymers at room temperature. SEM was employed to study the surface topography of the samples, which were immersed in solutions with and without silicone polymers. The image demonstrated that the high corrosion inhibition efficiency of polymers in NaOH solution (0.1 M), this may be interpreted due to the adsorption of polymer on alloys surface in order to block the active site present on the alloys surface resulting in a decrease in the contact between the aluminium and the alkaline medium and sequentially exhibited excellent inhibition effect.

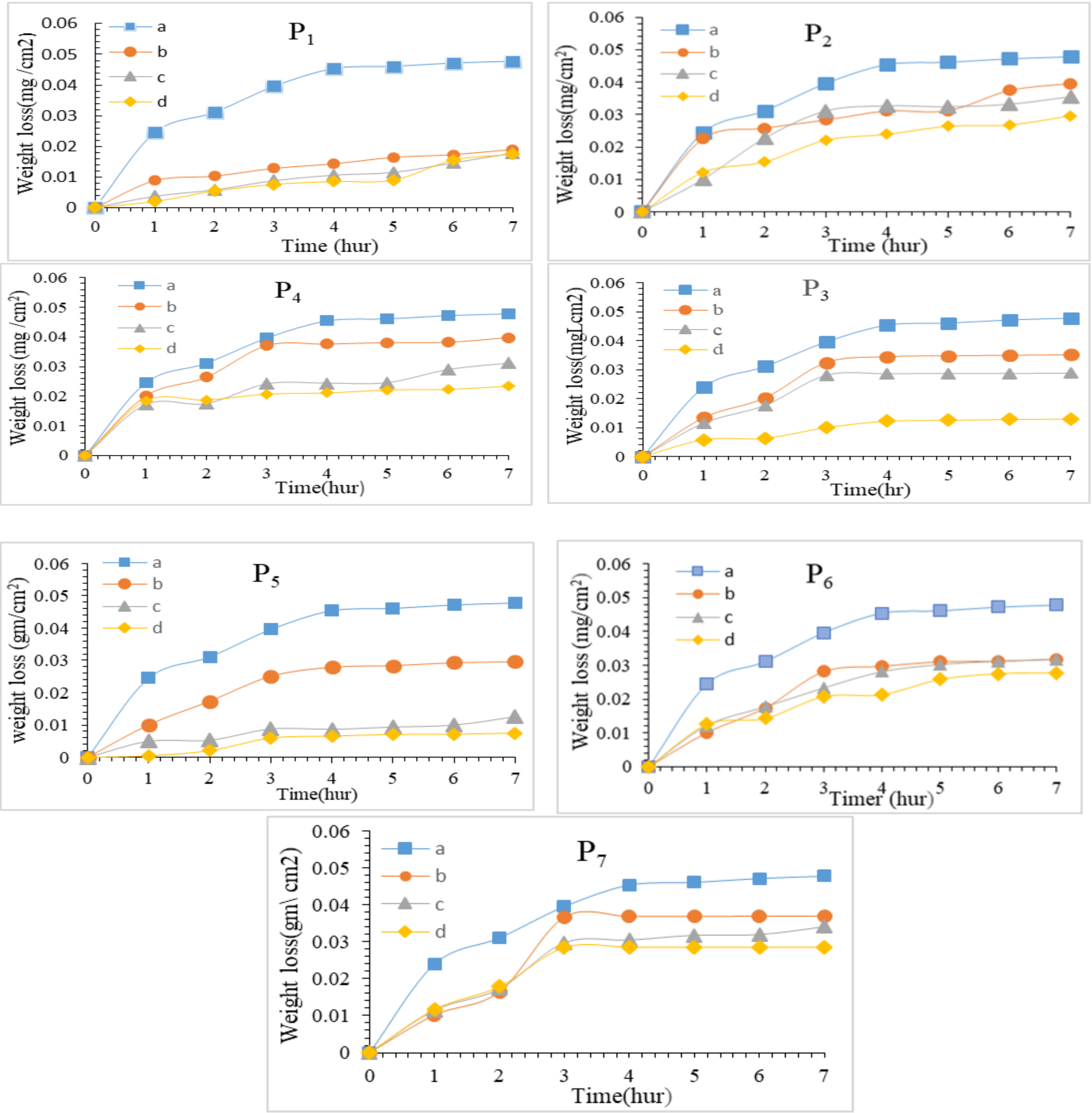
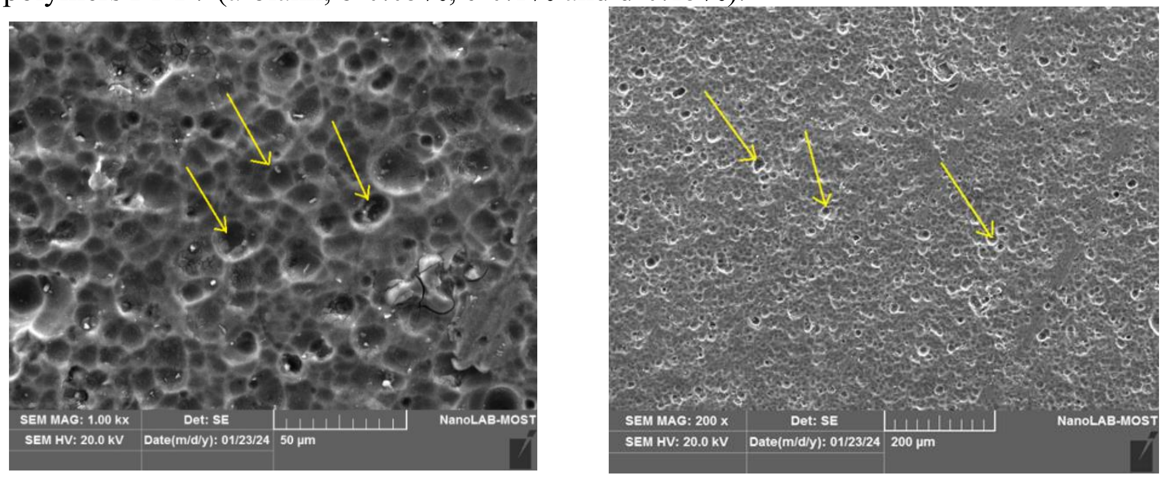
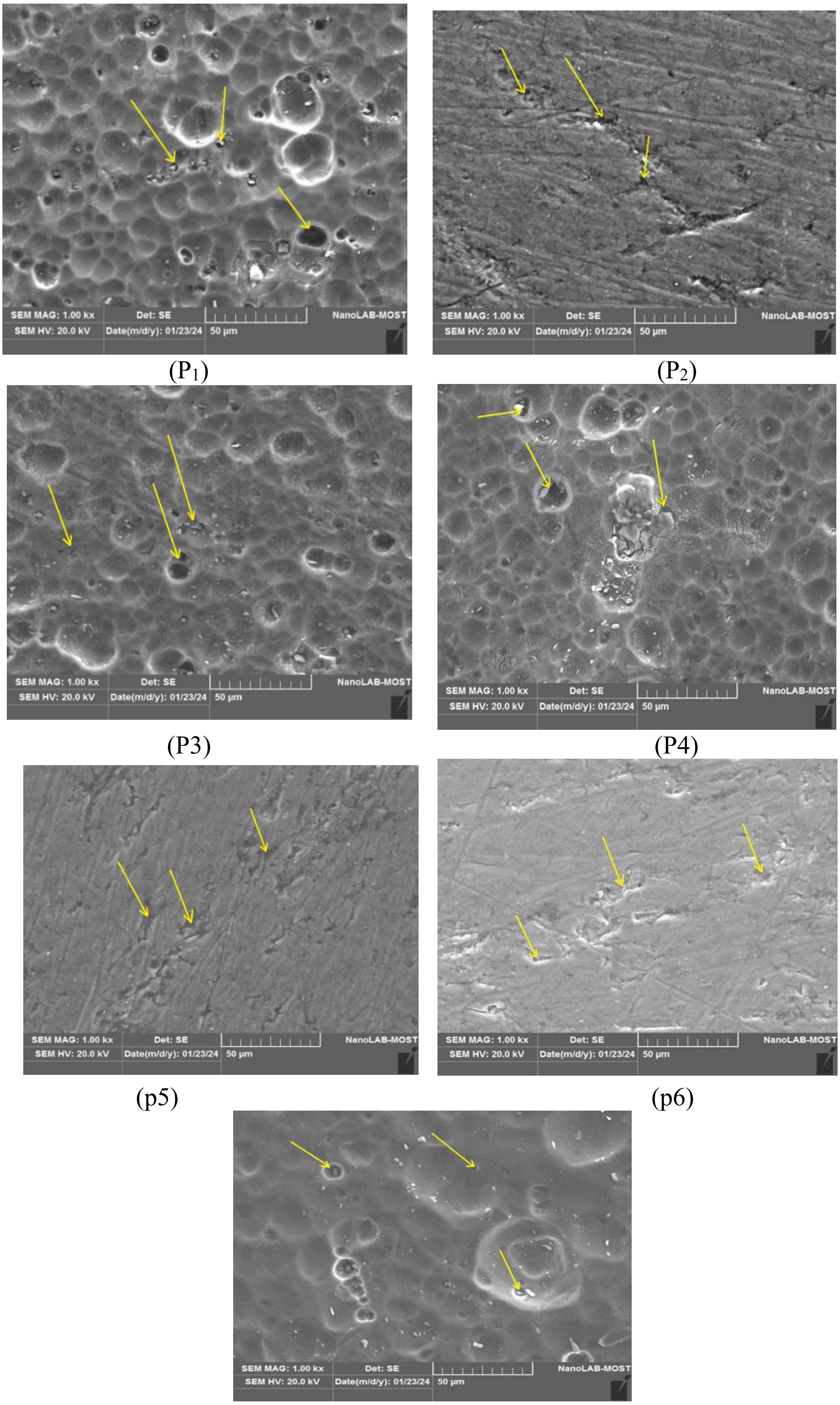


Figure 9: Variation of weight loss with time in 0.1M NaOH with different concentrations of polymers P1-P7 (a-blank, b-0.05%, c-0.1% and d-0.15%).



(a) (b)

Figure 10: a and b the SEM images of aluminum sample after immersion in alkaline medium without inhibitor after 7 hours showed pits in the form of spherical cavities



(P$_1$) (P$_2$)

(P3) (P4)

(p5) (p6)

(P$_7$)

Figure 11: SEM images of aluminum immersed in NaOH solutions (0.1M NaOH + 0.15% Inhibitor polymers P$_1$-P$_7$) for 7 hours, showed a smaller number of pits in the form pale depressions to nearly cylindrical or spherical cavities

Table 6: Calculated values of Weight loss and % Inhibition efficiency, Corrosion rate (mdd) for corrosion of P in 0.1 M NaOH at 25 ºC.

compound	% Inhibitor concentration of	Loss in weight (g)	Corrosion rate CR(mdd)	%Inhibitor Efficiency I.E (%)
blank	0	0.0478	261202.18	—
P_1	0.05	0.019	103825.13	60.26
	0.1	0.0178	97267.70	62.76
	0.15	0.0174	95081.96	63.6
P_2	0.05	0.0396	216393.40	17.16
	0.1	0.0355	193989.07	25.74
	0.15	0.0296	161748.60	38.07
P_3	0.05	0.0352	192349.70	26.36
	0.1	0.0288	157377.04	39.75
	0.15	0.0129	70491.80	73.02
P_4	0.05	0.0398	217486.40	16.74
	0.1	0.0312	170491.80	34.73
	0.15	0.0234	127868.80	51.05
P_5	0.05	0.0296	161748.60	38.08
	0.1	0.0126	68852.45	35.36
	0.15	0.0075	40983.60	84.31
P_6	0.05	0.0331	180874.30	30.76
	0.1	0.0318	173770.40	33.48
	0.15	0.0278	151912.50	41.85
P_7	0.05	0.037	202185.79	22.6
	0.1	0.0341	186338.79	28.67
	0.15	0.0286	156284.10	40.017

Dissolution and molecular interventions

Polymers differ from small molecules because of the large molecular weight of the polymers because the dimensions of the solvent and polymer molecules differ greatly. The solution process takes place in two stages. The first stage involves the penetration of solvent molecules through the polymer matrix to form a solvent mass called a gel. In the second stage, the gel disintegrates and the molecules disperse into a true solution. Dissolution is often a slow process. While some polymers dissolve in specific solvents, others require a long period of heating at a temperature close to the melting point of the polymer, this is due to the fact that some polymers have a very high cohesion energy density, which requires high energy to remove the molecule from its nearest neighbours [44]. The dissolution behaviors of the prepared polymers, P1 through P7, are presented in Table 6. It is noted that besides the influence of molecular weight, the dissolution process can be affected by the chain chemistry, composition and stereochemistry. Therefore, the nature of the polymers and differences in free volume and segmental stiffness are responsible for behavior variations of different polymers [45].

Table 6: Solubility of silicone polymers and copolymers in different solvents at room temperature (r.t) and 50 ^{0}C

Compound	**H_2Odistled water at r.t./50ºC**	**1%Aciticacid at r.t./50ºC**	**Ethanol at r.t./50ºC**	**Ether at r.t./50ºC**	**Dioxane at r.t./50ºC**	**DMF at r.t./50ºC**	**DMSO at r.t./50 ºC**	**1,2-dichloro Ethane at r.t./50 ºC**	**NaOH r.t./50 ºC**
P_1	-/-	-/-	+	-/-	-/-	±/+	+	-/±	-/+
P_2	-/-	-/-	-/±	±/+	-/±	+	±/+	-/±	±/+
P_3	-/+	-/-	±/+	-/-	±/+	+	±/+	-/±	+
P_4	-/-	-/-	-/-	-/-	-/±	±/+	±/+	-/±	±/+
P_5	-/+	-/-	±/+	-/-	+	+	+	+	+
P_6	-/-	-/-	-/-	-/±	-/-	±/+	+	-/-	±/+
P_7	-/±	-/-	-/-	-/-	-/-	±/+	±/+	-/-	±/+

"+ "means soluble, "-" means insoluble", ± poorly solubility

Conclusion

From the present work, the following conclusions can be drawn: silicone polymers P1-P7and two of them newly copolymers P_6 and P_7, obtained through condensation polymerization between aromatic imine compounds (monomers) with dichloro dimethylsilane. Spectrometric measurements (FTIR and 1HNMR) showed the expected data, demonstrating the successful synthesis of polymers (P_1-P_5) and copolymers (P_6, P_7). This study compared the influence of p-phenylene units on the thermal stability of the homopolymers and copolymers by DSC test and TGA, these materials presented interesting thermal properties. The variation of the substituents in both the homopolymers and the copolymers did not significantly affect thermal stability, but they demonstrated enhanced thermal stability with increasing phenylene units. Viscosity types of synthesized polymers have been studied, including relative viscosity (ηrel), specific viscosity (ηsp), and reduced viscosity (ηred). The results showed that viscosity of the polymeric solutions rose with higher polymer concentration, larger molecular weight of the silicone polymers and copolymers, and increased size of the polymer chains. On the other hand, the corrosion inhibition of aluminum in 0.1 M NaOH was investigated using the synthesized polymers, and showed the rate of corrosion of the aluminium in NaOH solutions is a function of the concentration of silicone polymers, the results showed increased inhibition with increasing polymers concentration, inhibition generally was obvious at concentration 0.15 %. This may be due to protonation of the hydrogen evolution process or the formation of an insoluble compound on the surface aluminum by polymer molecules. Also, the polymer P_5 at concentration 0.15% showed decreased in the corrosion rate to 40983.0 and the inhibitor efficiency reaches 84% compared with uninhibited solution for 7 hours. In general, silicone polymers and copolymers have been good for designing active corrosion protection systems with superior performance

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"The authors declare that they have no conflicts of interest."

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