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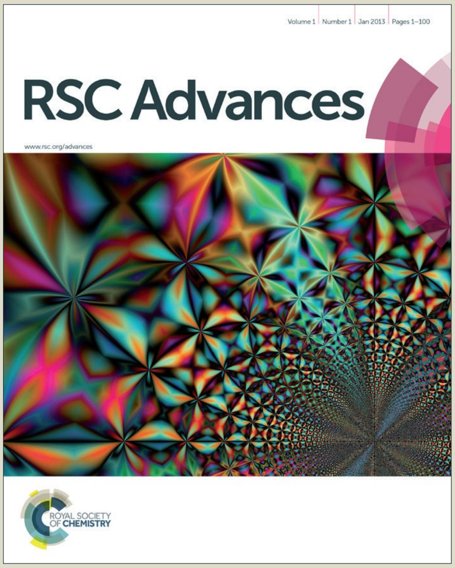

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Increasing toughness and tensile strength of epoxy-diamine system using inorganic ultra-accelerator

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Epoxy resins are a class of thermoset materials with extensive structural and composite applications. A wide range of chemical species, of which room temperature curing agents are most favourable, due to their widespread applications, can easily cure these resins. The major drawback of low temperature curing agents is their relatively high curing time. To overcome this issue, accelerators are usually added to decrease curing time and temperature. In this study, we present a unique type of inorganic accelerator that has immensely reduced curing time and temperature as well as imparting major improvements in mechanical properties including tensile strength, izod impact strength and adhesion strength. The epoxy resins used in the following study were diglycidyl ether of bisphenol A (DGEBA) and epikure F 205 (modified isophorone diamine). Curing kinetic of epoxy resin was determined in the presence of inorganic accelerator with FT-IR spectrum. Resins were cured with and without inorganic accelerator and the thermal properties were investigated using DSC (differential scanning calorimetry). FT-IR spectrum results showed that using 5% of inorganic accelerator, forced 99.68 % of epoxy groups to open, while crosslinks were observed after 18 minutes at 25 °C. Results from DSC analysis showed that using 5% of inorganic accelerator reduce the initial onset curing temperature from 68.3 °C to -2.4 °C, while significantly improve its mechanical properties in the polymer backbone.

Introduction

Due to the combination of excellent chemical and mechanical properties, epoxy resins are one of the most important classes of thermosetting polymers. They are suitable for a wide range of applications such as engineered composites, forming compounds, protective coatings, casting and adhesives[1-5]. Epoxy resins are low molecular weight pre- polymer that have one or more epoxy groups.[6]. These resin form a cross linked network along with curing agents[7]. There are many different types of curing agents including aliphatic and aromatic amines, polyamides, anhydrides and polymercaptans [8]. A number of factors including the type of curing agent and its curing temperature can affect the final properties of an epoxy network[9]. The curing agent can also determine the type of chemical bonds, morphology and crosslink density within a composite structure[8].

Curing agents that cure epoxy resins at room temperature or lower are more demanding due to their widespread applications[10]. Examples of such curing agents include aliphatic and cycloaliphatic amines, polyamides and polymercaptans which can ultimately cure epoxy resins at ambient temperatures[8]. The curing time for aliphatic amines are relatively high, while the density of the formed crosslink at room temperature is low. When such curing agents are used, due to the low degree of conversion, an extra step (post cure) is carried out to complete cure reaction and enhanced chemical and mechanical properties. Due to a stronger nucleophilicity of sulphur in comparison with nitrogen, polymercaptans can cure epoxy resins at much faster rate compared to aliphatic or cycloaliphatic amines[11]. Main disadvantages associated with the usage of polymercaptans are their unpleasant smell and the relatively complex and expensive synthesis procedures of mercaptans[8].

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For the last few years a range of compounds called accelerators are utilised to overcome the issues with temperature and curing time of epoxy systems [10]. Examples of such compounds include substituted urea, N- Methyl secondary amines, transition metal chelates, quaternary phosphonium, substituted thiodiphenol, tertiary amine, imidazoles, Lewis acid and dicyandiamide [8, 10, 12-19]. All of these compounds, accelerate curing of epoxy resin at high temperature and at the room temperature they are almost deactivate. On the other hand, using the aforementioned accelerators could reduce crosslink density of the network and ultimately decrease the mechanical properties of the epoxy system [20].

Amines are one of the main curing agents used in epoxy resin systems[8]. Aliphatic and cycloaliphatic amines are widely used in protective epoxy coating and for adhesive applications [8, 21]. Our results show that Epikure F205 base of isophorene diamine (IPDA) can be used as a curing agent for diglycidyl ether of bisphenol A (DGEBA) epoxy resin, with a novel accelerator precursor to make hardener. This accelerator is based on an inorganic salt (calcium nitrate solution). It has been shown that as the aliphatic and cycloaliphatic diamine react; the accelerator increases the curing rate of the epoxy system immediately. As a result, curing time and curing temperature of epoxy dropped. Also it has been found that curing reaction might take place in low (sub-zero) temperatures. When a high concentration of an inorganic accelerator was used, cure rate was high. It was observed that as the mixture of DGEBA, Epikure F 205 and high concentration of inorganic accelerator began to boil, the curing reaction took place immediately and foam was formed (the boiling point of DGEBA is >260 °C). Previous works conducted by Mark whiter *et al* in 2003, led to the invention of an inorganic accelerator that could only reduce the gel time, but was unable to alter the curing time of the epoxy system[22]. . In different tests that were performed it was found that HNO3 does not affect the curing reaction rate. Although aqueous solution of $Ca(NO_3)_2$ only reduces gel time and it does not affect curing time

Experimental

Materials

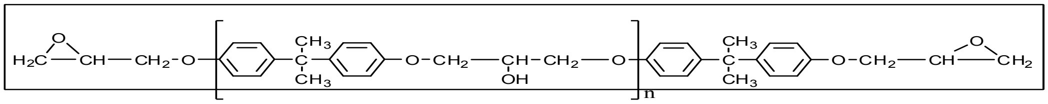
Liquid Diglycidyl Ether of Bisphenol A (Epon 828) (CAS No. 25068-38-6), with an epoxy equivalent weight of 185-192 eq/g was purchased from E.V Roberts. Epikure F205 was supplied by Hexion which was used as the curing agent. This curing agent is based on Isophoron diamine (3- aminomethyl-3, 5, 5-trimethylcyclohexylamine) (CAS No 2855- 13- 2) that was further modified to cure epoxy resin at room temperature. 65 % Nitric acid (CAS No 7697-37-2) and Calcium Carbonate (CAS No 471- 34- 1) was supplied by Sigma Aldrich. The chemical structures of DGEBA and IPDA are shown in Fig 1.

Methods

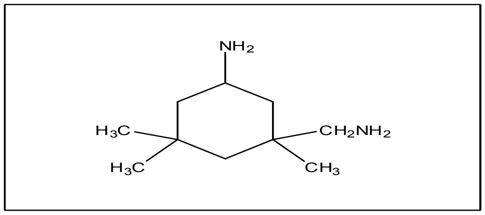
Synthesis of inorganic accelerator. 0.0263 mole (2.63 gr) of calcium carbonate was added to 5.152 ml of 65% nitric acid in room temperature with continuous stirring. After 2 minutes, a green solution was observed. Reaction occurred according to equation (1).

$$CaCO_3 + 4HNO_3 \rightarrow Ca(NO3)_2 + CO_2 + H_2O + 2HNO_3 \qquad (1)$$

Average epoxy molar mass (EMM) of Epon 828, as reported by the manufacturer, was 188.5 g/eq. The amine hydrogen molar mass (AHMM) of Epicure F 205, which was used as the curing agent, was reported to be 105 g/eq. Therefore, each amine hydrogen reacts with one epoxy group and the stoichiometric ratio of the curing agent for reaction with the epoxy resin can be calculated accordingly as AHMM/EMM= 0.557. To prepare the samples, 100g of epoxy resin was mixed with 55.7g of Epicure F 205.



Diglycidyl ether of bisphenol A (DGEBA) (CAS No 25068-38-6)



Isophorone diamine (IPDA) (CAS No 2855-13-2)

Figure 1. Chemical structures of Didlycidyl ether of bisphenol A (DGEBA) and Isophoronediamine (IPDA)

All samples were prepared according to the ratio 1: 0.557 epoxy resin and F 205 respectively and only the amount of inorganic accelerator in the samples was changed. The percentage of inorganic accelerator in the epoxy resin for different samples has been shown in Table1.

Analysis Techniques

Fourier transform infrared spectroscopy (FTIR). The FT-IR analysis was used in FT-IR spectrometer, Tensor 27, Bruker with 40 scan average at a resolution of 4 cm^{-1}. In the present work, FT-IR spectroscopy was applied to determine the degree of conversion for epoxy resins in the isothermal mode at room temperature.

Table 1. Amount of inorganic accelerator in different samples (percentage was considered into to epoxy resin)

Sample Name	Inorganic accelerator content (%)
A	0
B	1.25
C	2.5
D	3.75
E	5

Differential scanning calorimetry (DSC). In this study DSC (Linseis, PT10, and Germany) was used to report the rate and curing condition of the reaction. The DSC machine was calibrated with standard zinc and indium. Samples were prepared by mixing resin, curing agent and accelerator for 30 seconds, according to the recipe shown in Table 1. Then 5 mg of each sample was sealed in an aluminium pan and was placed in DSC machine for curing (because of high curing rate of the system by accelerator, preparation of sample and placing in DSC machine should be fast). A non-isothermal test at a heating rate of 10 °C/min under a nitrogen flow of 40 ml/min was performed at the temperature range of -50 to 170 °C.

The glass transition (Tg). To measure Tg, DCS method (differential scanning calorimetry) was used (DSC, Linseis, PT10, Germany). Mixing, resin, curing agent and accelerator for 30 seconds, samples were prepared according to the recipe shown in Table 1. Prepared samples were left at room temperature for 7 days. Then 5 mg of cured samples were placed in an aluminium pan. For each sample Tg was measured at the temperature range of 0- 150 °C under nitrogen flow of 40 ml/min and heating rate of 10 °C/min by DSC machine.

Tensile strength. Tensile strength test was performed according to ASTM D256 with a Shimadzu 20KN-testing machine. Specimen dimensions were chosen according to type 1 of ASTM (165 × 19 × 3.2 mm). Crosshead speed was 2 mm/min. Specimens were prepared according to Table 1. DGEBA, Epicure F 205 and inorganic accelerator were mixed for 30 seconds and the mixture was then placed into a silicon mould. To allow a complete curing reaction, following 8 hours at room temperature, specimens were removed from the mould and stored at room temperature for 7 days, (the complete curing time for sample A was 7 days). From each sample 5 replicates were prepared and tested.

Pull-off strength. The pull-off strength was measured in accordance with ASTM D4541, by using a Posi Test AT-M from Deflesco. Measurements were carried out following 12 hours of curing process. Each test was repeated 5 times on an aluminium surface.

Izod impact strength. To measure izod impact strength, five replicates from each sample were prepared. Specimens were un-notched with the dimensions of 63.5 × 12.7 × 7.2 mm, according to ASTM D256 using Zwick/Roell 6103 impact tester at room temperature. DGEBA, Epicure F 205 and inorganic accelerator were mixed for 30 seconds with the ratios indicated in Table 1, and then the homogeneous mixture was poured into a silicon mould. After 8 hours, samples were removed from the silicon mould and kept for 7 days at 25 °C to complete curing.

Scanning electron microscopy (SEM). To investigate the effect of inorganic accelerator on the toughness of epoxy samples, fracture surfaces from izod impact strength were observed using Scanning electron microscopy (Tescan, MIRA3 FEG-SEM, Czech Republic). To enhance the resolution, samples were coated with gold vapour. SEM was operated at an accelerating voltage of 5.00 KV.

Results and discussion

The inorganic accelerator reported in the study was synthesized by using inexpensive materials following a simple method of preparation. Unlike existing accelerators, the invented accelerator increases the mechanical properties (adhesion, tensile strength and impact strength) of epoxy resin. Following discussion investigates the influence of inorganic accelerator (Calcium nitrate solution) on the mechanical properties such as adhesion, tensile strength, and izod impact strength on epoxy resins. The time required to completely cure DGEBA and Epikure F 205 at 25 °C was 7 days (from datasheet). In this study, it was found that by using 5% inorganic accelerator in the system, curing time of resin at 25 °C can significantly reduce to only 18 minutes, which is notably lower compared to not using the inorganic accelerator.

The curing mechanism of epoxy resins are different in the presence of an inorganic accelerator, where homopolymerization reaction occurs[8]. In the acidic environment, cationic polymerization takes place which causes an increase in the chain length[23, 24] . In the presence of a curing agent such as Epikure F 205, without the addition of a catalyst and accelerator, the curing reaction is in the form of a polyaddition reaction forming a branched structure [8]. The molecular architecture is directly related to the physical and mechanical properties of the epoxy structure [25], such that as the chain length increases, the tensile strength also increases. This is mainly due to the fact that the elongation and yield points are directly related to the length of polymer chains.

The inorganic accelerator synthesized in this study was acidic in nature, while the curing agent contained amine groups and was basic. Upon reacting the inorganic accelerator with the curing agent, the acid-base reaction caused an increase in the temperature of the system, with the formation of inorganic salts. This increase in the system temperature fastens the curing reaction. The mechanism of curing epoxy in the presence of this inorganic accelerator can be categorised into the following 3 mechanisms: poly addition, cationic polymerization and anionic polymerization. The mentioned mechanisms can take place simultaneously, or occur one after another depending on the system condition. Figure 2 illustrates the 3 mechanisms of curing epoxy as discussed earlier (Polyadditional reaction). Initially, ring opening took place by the attack of primary amine groups to the epoxy ring. The second amine group reacted with another epoxy group and also with the hydroxyl group that was formed during the curing reaction. This resulted in the formation of an etherification reaction, as shown in Figure 2 [8,20].

Cationic and anionic polymerization. Cationic polymerization using Lewis acids and protonic acids has been discussed in many different studies[23, 24]. In this investigation, nitric acid was used as the protonic acid compound. Upon mixing the inorganic accelerator with DGEBA and Epikure F 205, an acid-base reaction took place (inorganic accelerator has an acidic role while Epicure F 205 has an alkaline role) (Figure 3). Due to the exothermic nature of the acid-base reaction, the system temperature and curing rate were increased.

Upon further investigation, it was discovered that the reaction of calcium nitrate with the amines resulted in the formation of protons and nitrate ions. This was important as protons had the ability to lead to cationic polymerization, while the negative nitrate ions could initialise anionic polymerization. The proposed mechanisms of cationic and anionic polymerization are schematically shown in Figure 4 and Figure 5 respectively. Experimentally, it was found that during the anionic polymerization, nitrate ions were attached to the epoxy group, causing the rings to open in the presence of [HA]. The mentioned process could be referred to as a proton supplying curing reaction.

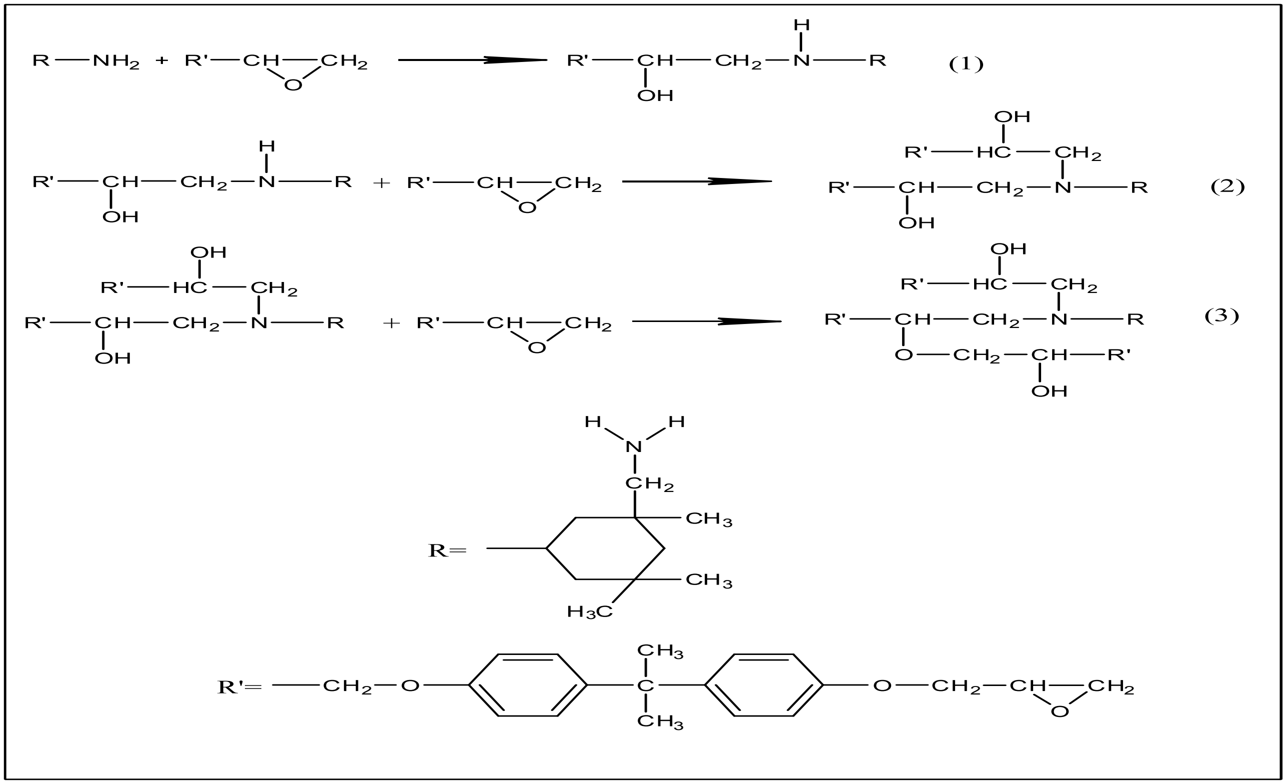


Figure 2. Polyadditional reaction with epoxy resin and polyamine.

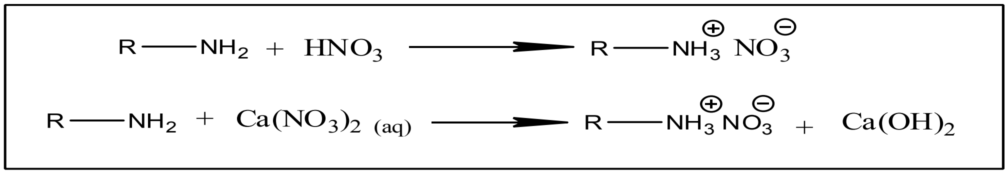


Figure 3. Reaction between amine curing agent and inorganic accelerator (acid- base reaction)

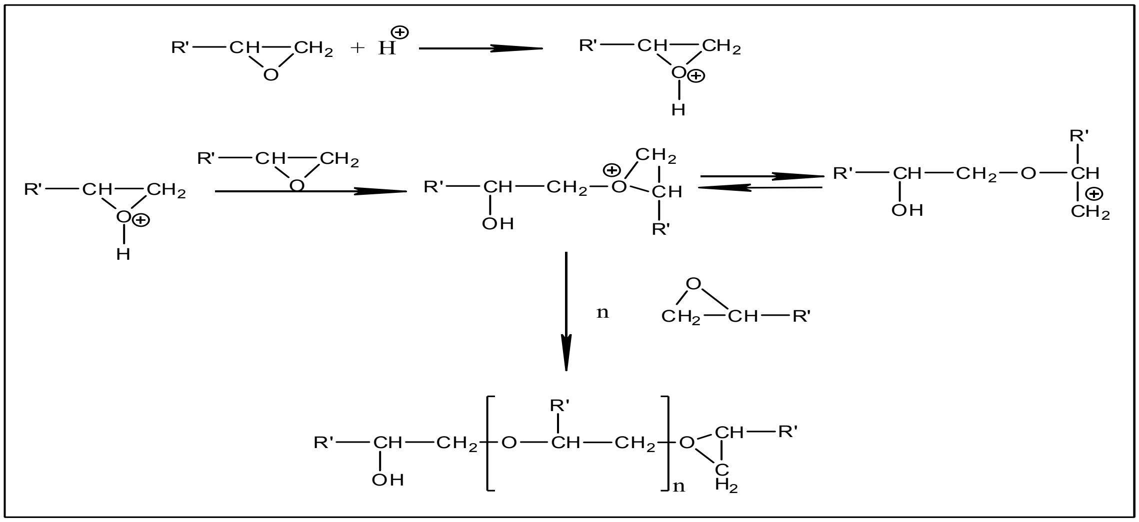


Figure 4. Proposed cationic polymerization of epoxy groups in the presence of inorganic accelerator

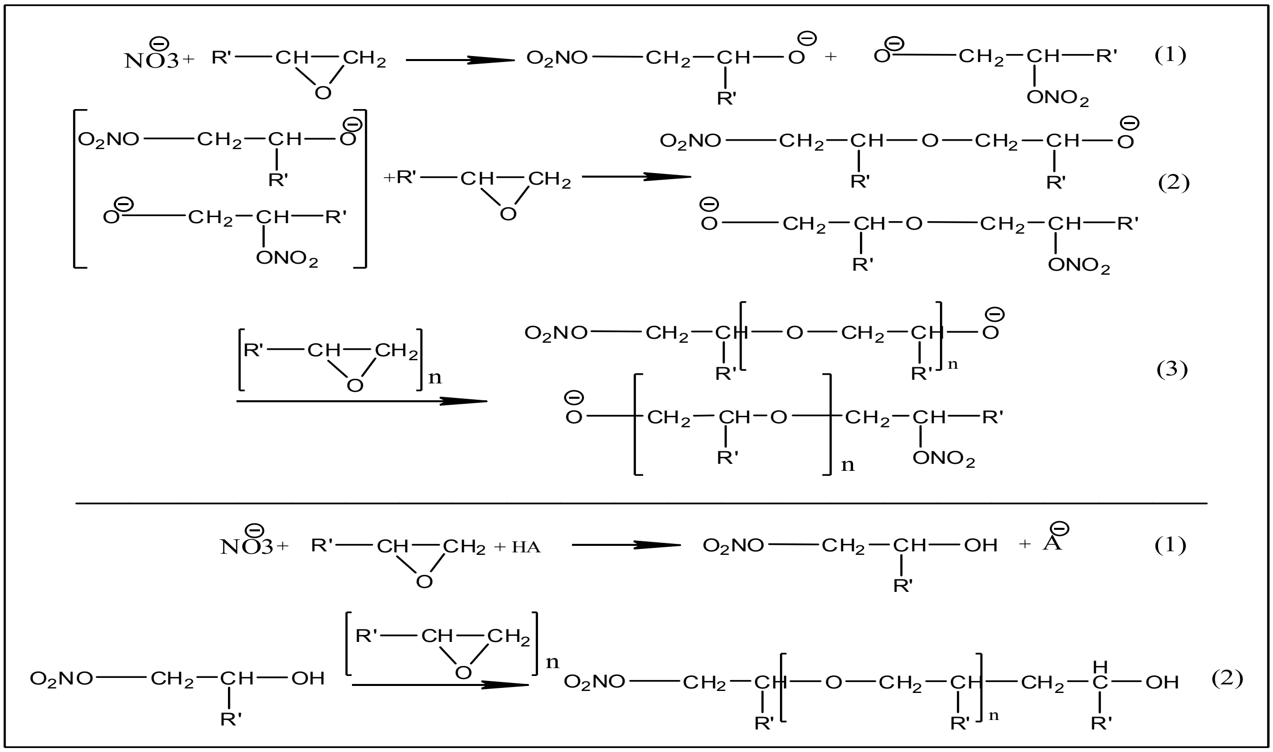


Figure 5. Proposed anionic polymerization of epoxy groups in the presence of inorganic accelerator

The newly formed hydroxyl groups continue the reaction until completion. In this reaction [HA] can be water or nitric acid. Both anionic and cationic polymerizations increase the chain length, and epoxy chains grow more in the length direction[23, 24, 26]. This increase of chain length gives the network of epoxy new properties. The final epoxy structures were assumed to be similar to Figure 6 for both cases of with and without inorganic accelerator. The epoxy network crosslink of DGEBA/ Epicure F 205 after curing, covered all directions and lacked any special orientation, however the in the presence of inorganic accelerator, it was found to have a lengthy orientation, increased chain length and with most of the crosslinks aligned across one line.

FTIR analysis

FT-IR spectroscopy is a sensitive method to determine crosslink density and curing rate of epoxy resin [1, 27-30]. To study the effect of inorganic accelerator on the curing reaction and determining the degree of conversion of epoxy resin, sample No 5 (sample E) was selected. Required amounts of epoxy resin (DGEBA), Epicure F 205 and inorganic accelerator were prepared based on Table 1. The materials were transferred into a container and were mixed for 30 second in 25 °C. Immediately after mixing, a drop of mixture was poured between two KBr tablets for FT- IR spectroscopy and a sample was taken out every two minutes from the spectrum for 18 minutes, preparing a total of 9 spectrums.

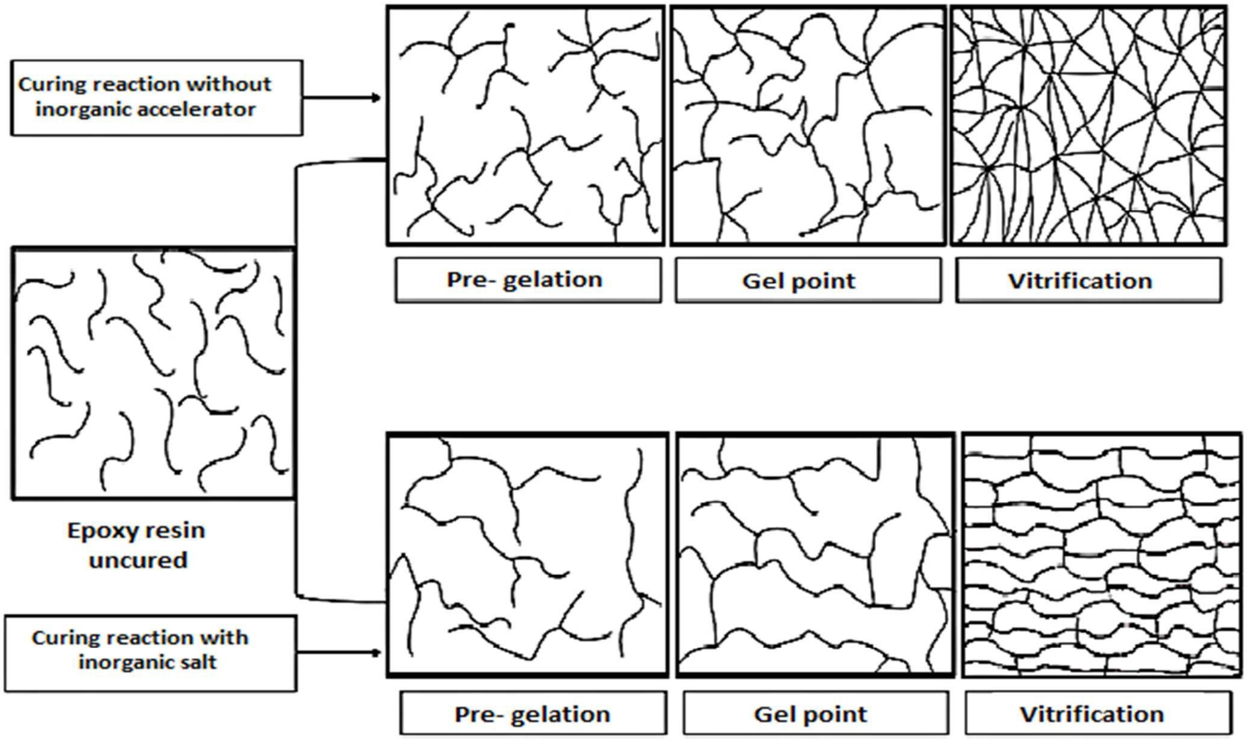


Figure 6. Cure epoxy architect with and without of inorganic accelerator

Spectra with an area of 3800-400 cm^{-1} were obtained, whilst a range of 1300-400 cm^{-1} was used to determine the degree of conversion. The absorption bond of 915 cm^{-1} indicated the presence of epoxy groups. After XXXXX minutes, the intensity of absorbent with a peak of 915 cm^{-1} was reduced due to the opening of the epoxy rings. The peak at 1182 cm^{-1} showed C-O stretching of aromatic ring of the epoxy resin (DGEBA)[30, 31]. As it can be seen from Figure 7, the absorbance intensity of the epoxy group at 915 cm^{-1} was halved after 2 minutes, which indicated a very high curing rate for the reaction. It should be noted that the reaction temperature was 25 °C.

Equation (2) was used to determine the degree of conversion.

$$\alpha = \left\{1 - \left[\frac{(\mathrm{A}\ 915, \mathrm{t})(\mathrm{A}\ 1182, 0)}{(\mathrm{A}\ 915, 0)(\mathrm{A}\ 1182, \mathrm{t})}\right]\right\} \times 100 \qquad (2)$$

In equation (2), A indicates the absorbance of the functional group and the subscripts 0 and t refer to the initial and current reaction time respectively. According to Figure 8, after 2 minutes from the reaction initiation, 49.942% of the epoxy groups were opened and formed crosslink, while after 18 minutes, the percentage of crosslink degree reached 99.6779%, which indicated almost a complete curing reaction of DGEBA and high activity rate of the inorganic accelerator. However in the case of curing the sample without an accelerator (DGEBA, Epicure F205), the curing time required was 7 days at room temperature (25°C). Since the rate curing reaction declined dramatically after 6 minutes, presenting all 9 spectra will cause an overlapping of spectra and ultimately make it impossible to determine. Other results of spectra was used for calculation of degree of crosslink.

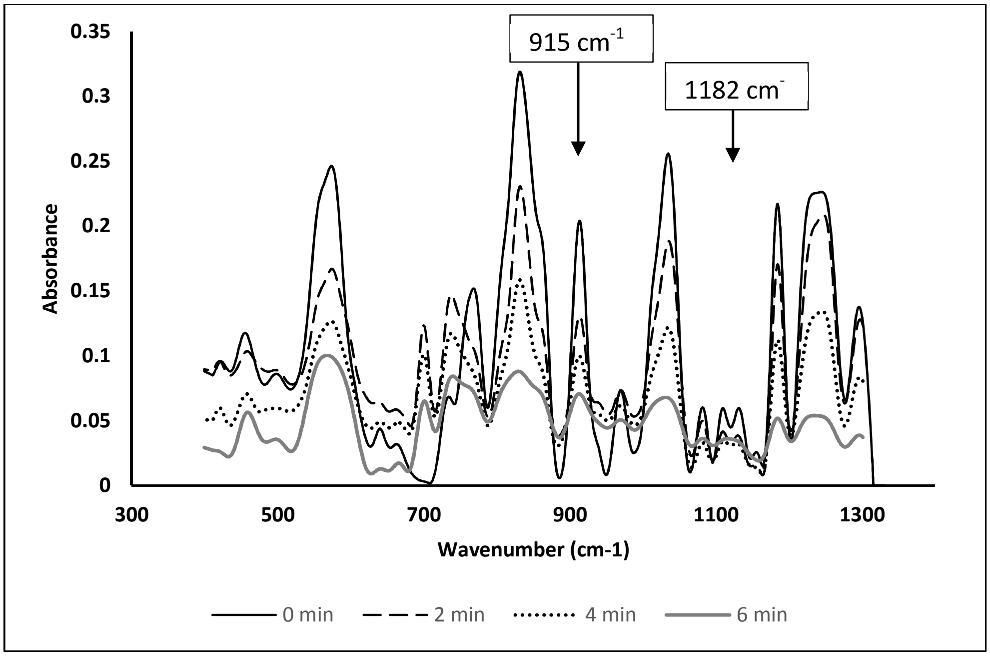


Figure 7. The partial FT-IR spectra of epoxy resin and Epikure F 205 with 5% Inorganic accelerator

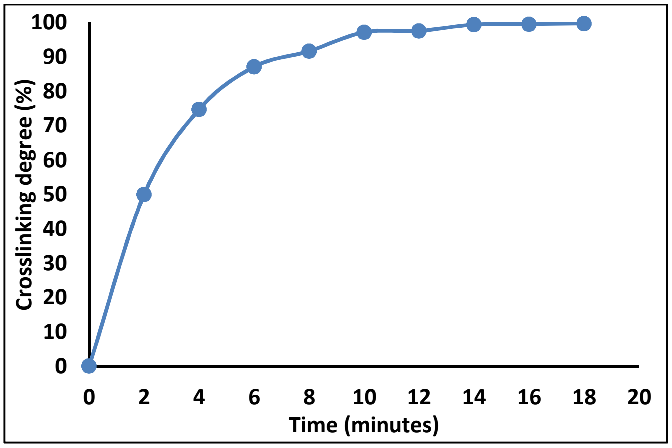


Figure 8. Process of epoxide reaction in DGEBA/ Epikure F 205/ Inorganic accelerator system by FT-IR spectroscopy at 25 °C

DSC analysis

Information obtained from DSC (differential scanning calorimety) can be related to the curing reaction of epoxy system [14]. Many studies have been conducted on the epoxy resin to obtain kinetic data with the DSC[1, 5-7, 12, 13, 32-36].

Non- isothermal DSC curves of sample A (without inorganic accelerator), showed one exothermic peak (Figure 9, a). The initial onset temperature (T_1) and the first peak temperature (T_{p1}) were seen at 68.3 °C and 93.2 °C respectively. By adding inorganic accelerator the initial onset temperature (T_1) and the first peak temperature (T_{p1}) were dramatically reduced and the curing of epoxy resin was divided into two parts: the first part occurred in the lower temperatures and the second part occurred in the higher temperatures. As low concentration of inorganic accelerator was used, curing reaction was divided into two parts. In sample B (with 1.25% inorganic accelerator), the initial onset temperature (T_1) was 11.9 °C compare to the sample without accelerator (sample A) (the second onset temperature (T_2), was 54.7 °C). This curing reaction occurred in two steps, the first step at the presence of inorganic accelerator, which initiated early. Hence the concentration of accelerator was low, curing reaction was partially gone and consequently majority of the accelerator was consumed, causing the curing reaction to stop at the low temperature. The curing reaction was paused until the temperature of the system increased. Consequently at the second onset temperature 54.7 °C (T_2), the remainder of the curing reaction continued. Total heat of curing (ΔH) was raised from 56.9692 J/g (sample A, without accelerator) to 329.2118 J/g (sample 2, with 1.25% accelerator), which shows the release of heat from curing reaction in the presence of accelerator. In sample C (2.5% inorganic accelerator), similar to sample B (1.25% inorganic accelerator), the curing reaction occurred in two steps. By increasing the accelerator concentration, the initial onset temperature (T_1), the first temperature peak (T_{p1}), the second onset temperature (T_2) and the second temperature peak (T_{p2}) were decreased ($T_{1:}$ 0.1 °C, T_{p1}: 8.3 °C, T_2: 37.9 °C, T_{p2}: 52.4 °C). As a result total heat of curing (ΔH) of sample C (2.5% inorganic accelerator) decreased. By increasing inorganic accelerator concentration to 3.75% and 5%, the curing reaction of epoxy system had occurred at negative temperature and in one step. Results of various samples have been summarised in table 2. In general, by using inorganic accelerator in the DGEBA/ Epicure F 205 system, the curing reaction would be faster and would occur at the lower temperature.

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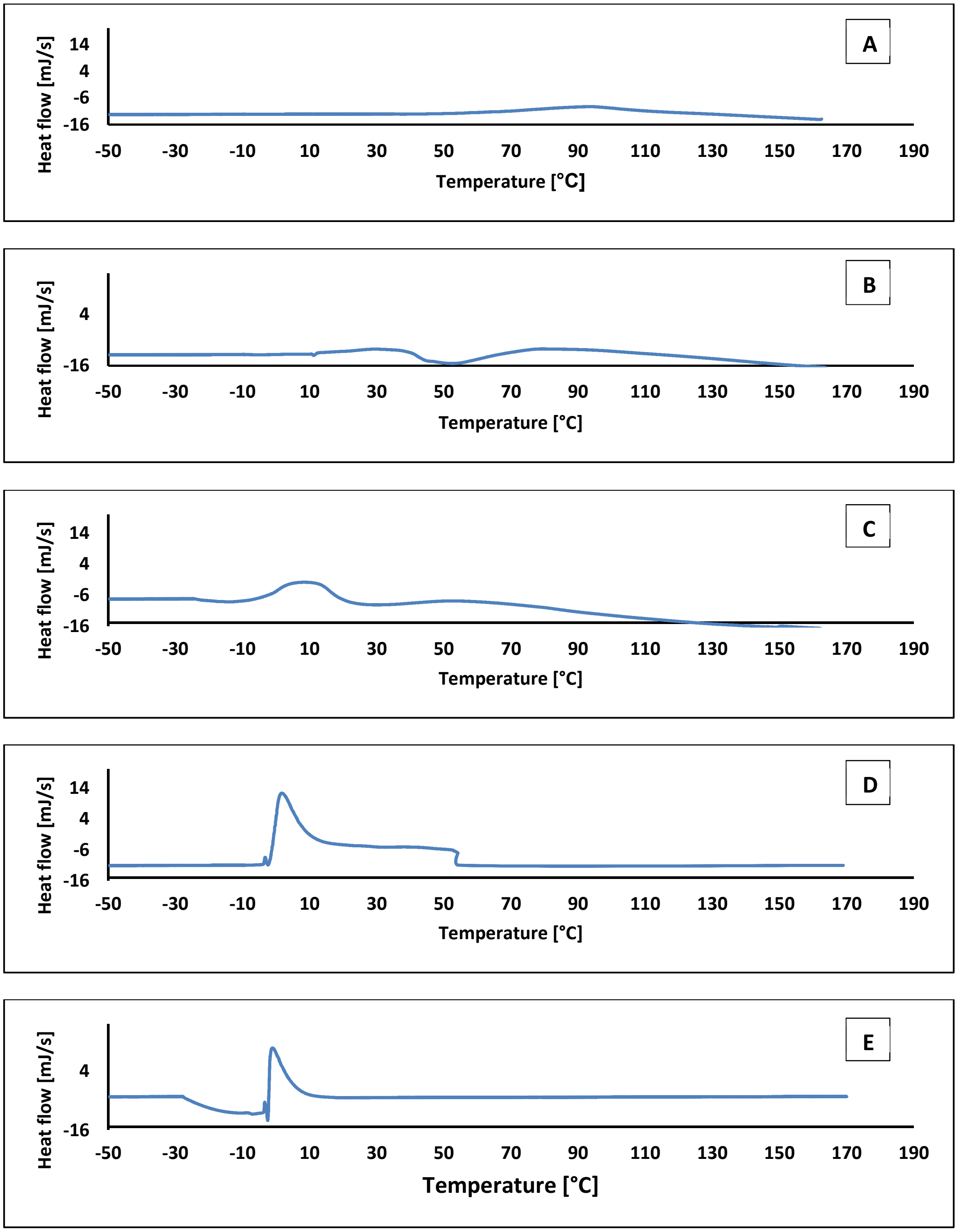

Figure 9. Heat flow versus temperature was obtained by DSC from samples A, B, C, D and E at 10 °C heating rate

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Table 2. DSC information from the epoxy system with and without inorganic accelerator at 10 °C/min heating rate

Sample Name	Inorganic accelerator (%)	T_1 (°C)	T_{p1} (°C)	T_2 (°C)	T_{p2} (°C)	ΔH (J/g)
A	-	68.3	93.2	-	-	56.9692
B	1.25	11.9	30.5	54.7	78.9	329.2118
C	2.5	0.1	8.3	37.9	52.4	165.3273
D	3.75	-1.7	1.7	-	-	123.4849
E	5	-2.4	-0.9	-	-	66.3832

Glass Transition

The glass transition temperature (Tg) relates to chain mobility of a polymer. Findings of the present work show that in the present of an inorganic accelerator, the polymer chain length had increased. When the chains are able to move more freely, Tg is found to be decrease. However it should also be noted that the formation of inorganic salt during the curing reaction decreases mobility of the chains by filling spaces between the chains, causing Tg to increase.

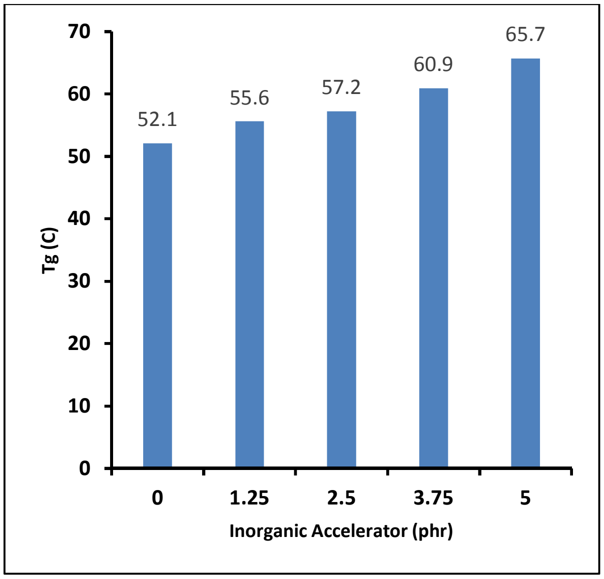

Figure 10. Tg of the samples as a function of inorganic accelerator content

The mentioned process is similar to the experiments where inorganic particles like TiO_2, ZnO and SiO_2 were used[37-40]. Inorganic materials have high melting point, so it is obvious that they increase thermal properties such as Tg. As mentioned, increasing temperature cause formation of the inorganic salts within the gaps of the chains, which prevents polymer chains to move freely.

By increasing the amount of inorganic accelerator, Tg will grow. In the present of inorganic accelerator, inorganic salt will be formed that will make it harder for the polymer chains to move freely. Changes of Tg are shown in figure 10.

Tensile strength

According to table 3 by adding inorganic accelerator, tensile strength, tensile modulus and elongation were increased. This behavior shows that the chains grow in length direction and can be more tensile. When the chains grow in lengthwise direction (in the present of inorganic accelerator), the structure of the cured epoxy can tolerate more loads compared to a chain growth in a disordered direction. Indeed, when the structure is placed under tension, long chains have higher tear resistance, as they are able to stretch further.

All samples in this work were cured at room temperature and without post cure. (Sometimes to increase the mechanical properties, after curing the sample at room temperature, the sample is placed at high temperatures of approximately 90 °C for a duration of 1 hour). For this reason, the tensile strength and modulus of the cured epoxy resin in this work are much lower than the works done by other researches (High temperatures were never used in this work during curing reaction or even after that). This is in strong agreement with one the aims of the proposed work, that is to synthesis an epoxy system independently, without utilizing an external sources of heat.

Pull-off strength

According to the results of pull-of strength (Table 4), adhesion has increased by adding inorganic accelerator. By increasing the amount of inorganic accelerator, the pull-off strength is also increase. It is assumed that formed inorganic salt improves the pull off strength. During the pull off force, the inorganic salt prevents formation of cracks at the surface in initial step. Many studies confirmed this by showing that the fine inorganic particles can indeed increase pull of strength[41-43].

Izod impact strength

Figure 11, shows the impact of izod strength versus inorganic accelerator content. As the concentration of the inorganic accelerator increases, the izod impact strength rises as well. For instance with 5% content of inorganic accelerator, izod impact strength was found to have increases by a multiple of 1.76 times. The inorganic salt present in the gaps of the polymer chains can absorb force. Izod impact strength test was performed to examine fractured surface of samples. It was observed that the fracture surface of the samples that contained inorganic accelerator was rough, indicating that they had higher izod impact strength.

Table 3. Effect of inorganic accelerator on the tensile strength, tensile modulus and elongation at break

Sample Name	Inorganic accelerator (%)	Tensile strength (Mpa)	Tensile modulus (Mpa)	Elongation at break (%)
A	-	29.63 ± 1.3	1770.86 ± 46.4	1.79 ± 0.41
B	1.25	32.12 ± 2.53	1544.63 ± 41.1	2.11 ± 0.62
C	2.5	32.76 ± 4.2	1365.46 ± 52.8	2.25 ± 0.33
D	3.75	33.97 ± 1.6	1294.27 ± 49.5	2.36 ± 0.19
E	5	36.00 ± 1.4	1257.92 ± 44.7	2.88 ± 0.21

Table 4. The pull-off result from epoxy system with different amount of accelerator

Sample name	Inorganic accelerator (%)	Pull-off strength (Mpa)
A	-	4.41 ± 0.3
B	1.25	4.69 ± 0.2
C	2.5	5.03 ± 0.5
D	3.75	5.17 ± 0.4
E	5	5.45 ±0.2

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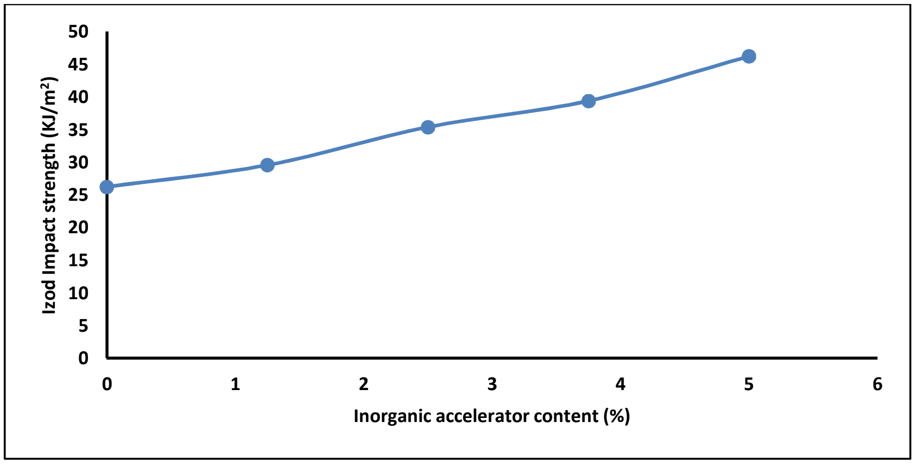


Figure 11. Izod impact strength of epoxy/ Epikure F 205/inorganic accelerator (%)

SEM morphology

Figure 12 shows the surface of sample A (without inorganic accelerator) was completely slick and flat. Upon addition of an inorganic accelerator, the surface specimen changes from its initial flat condition and become rougher. Jagged surface absorbs the energy ultimately and forces the samples to break. As the amount of inorganic accelerator is increased, the surface of sample becomes more jagged. Increase in toughness is shown in figure 12. Holes can be seen clearly in figure 12- D and 12- E. When the two parts (epoxy resin Epicure F 205 and inorganic accelerator) are mixed air bubbles tend to be introduced into the system. In samples that contain greater amounts of inorganic accelerator such as samples D and E, rate of curing is much higher and air bubbles are trapped into the structure of the epoxy. Hence it can be concluded that with the addition of inorganic accelerator, the surface fracture of the sample becomes more rough and rigid.

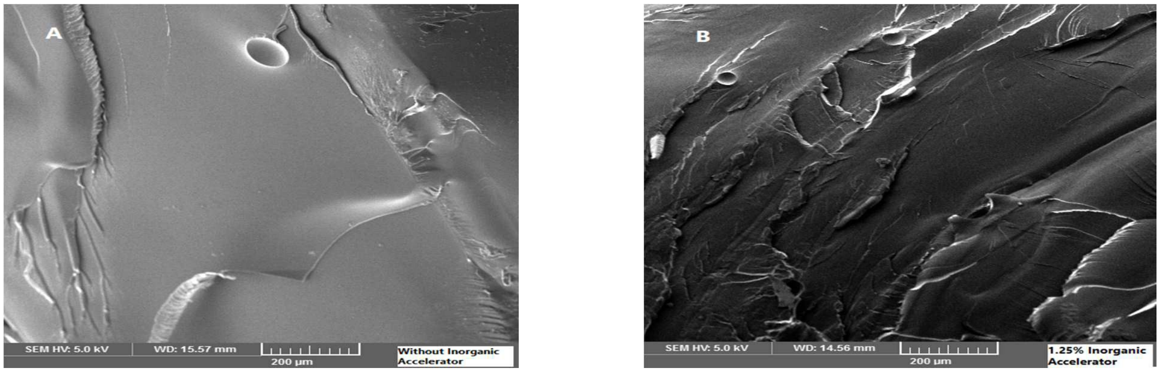
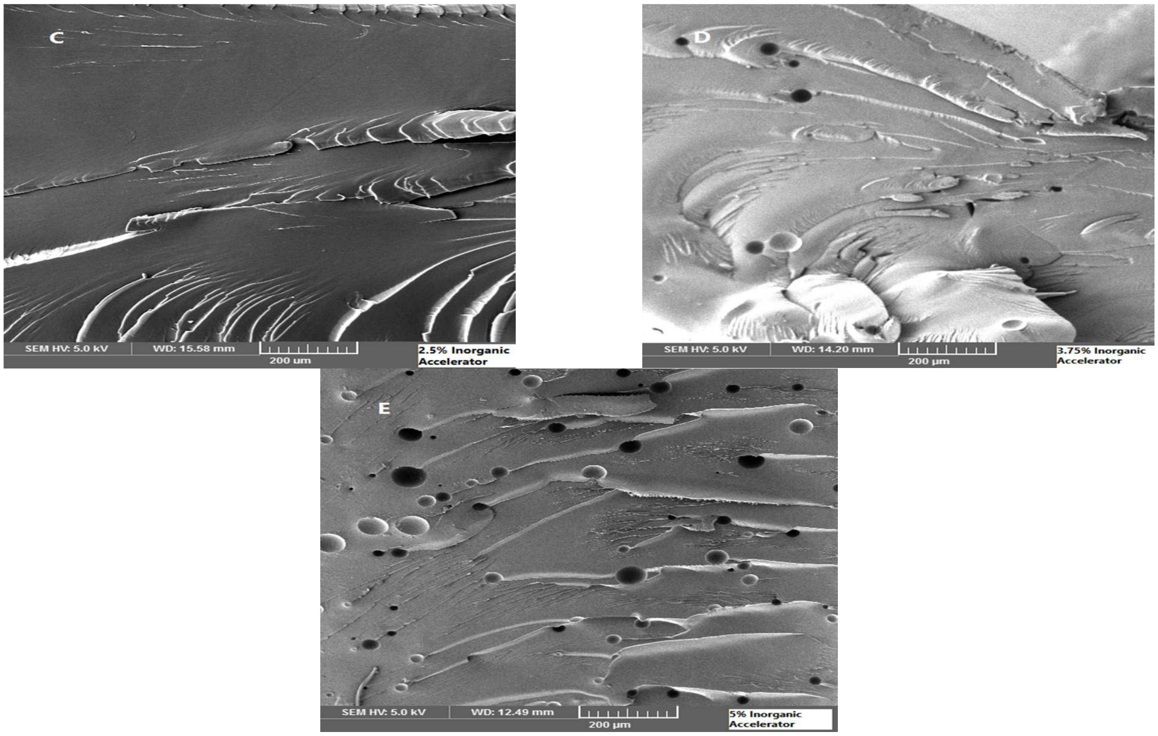


Figure 12. SEM images of the fractured surface (A: without inorganic accelerator, B: 1.25% inorganic accelerator)



Continue of Figure 12. SEM images of the fractured surface for samples (C: 2.5% inorganic accelerator, D: 3.75% inorganic accelerator, E: 5% inorganic accelerator)

The presence of such air bubbles can alter the mechanical properties, but it should be noted that the invented inorganic accelerator changed the architect of the chain polymers, and the formed inorganic salts acted as fender within the chain gaps. Hence, even though air bubbles may be present within the mixture, the mechanical strength is unaltered due to the inorganic accelerator.

Conclusions

In conclusion, results of this study indicate that inorganic accelerator is an effective compound to reduce curing time and curing temperature of epoxy resins. This accelerator can be used at negative temperatures with cycloaliphatic and aliphatic diamine curing agents such as Epicure F 205. The degree of conversion after 18 minutes of curing reaction at 25 °C with 5% inorganic accelerator reached 99.68%. While in the absence of inorganic accelerator, the complete degree of conversion of epoxy system (DGEBA and Epicure F 205) required a period of 7 days at room temperature to cure. It was also found that mechanical properties of the resin (tensile strength, izod impact strength and pull-off) have increases significantly in the presence of accelerator. It is assumed that an inorganic accelerator changes the architecture and structure of the epoxy network in a way that the length of the chains becomes longer. Subsequently the amounts of hydroxyl groups are increased, providing the epoxy resin with the required adhesion property.

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