



Research Paper / Makale

Microwave curing process parameters on epoxy-based polymer composites

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Abstract: Microwave rapid curing processing has great potential for improving composite manufacturing such as reduction of curing time, energy requirements and operational costs. In this study, pre-waiting time and curing time effects on epoxy-based polymer composites with and without fillers were investigated during a microwave curing process. Silica sand was used as filler material. After prefabrication of polymer samples with and without fillers, the samples were left waiting for pre-curing for 0, 30, 60 and 90 min. Besides, the power of microwave curing was altered to be either 100W, 300W, 450W, 600W or 800W. Other polymer samples were cured at the laboratory for 7 days for comparison. Tensile strength, energy to break, hardness, water absorption, and microstructure investigations were performed on all samples. Results show that subjecting polymer materials to microwave treatment instead of keeping them at room temperature for 7 days yielded similar or higher tensile strength, hardness and impact toughness values in a shorter time.

Keywords: Microwave; curing; polymer; pre-waiting time.

Epoksi Esaslı Polimer Kompozitlerde Mikrodalga Kürlenme İşlemi Parametreleri

Öz: Mikrodalga hızlı kürlenme işlemi, kürlenme süresinin azaltılması, düşük enerji gereksinimleri ve işletme maliyetleri ile kompozit malzeme üretimini geliştirmek için büyük bir potansiyele sahiptir. Bu çalışmada, mikrodalga kürlenme işlemi sırasında dolgu maddeleri içeren ve içermeyen epoksi esaslı polimer kompozitler üzerine işlem öncesi ve sonrası mikrodalgada farklı bekleme sürelerinin, enerji miktarlarının ve kürlenme süresinin malzemenin mekanik özelliklerine etkisi araştırılmıştır. Dolgu malzemesi olarak silis kumu kullanılmıştır. Polimer örneklerinin hem dolgu maddesi ile hem de dolgu maddesi olmadan prefabrikasyonundan sonra, numuneler ön kürlenme için 0, 30, 60 ve 90 dakika bekletilmiştir. Ek olarak, mikrodalga kürlenme gücü 100W, 300W, 450W, 600W veya 800W farklı güç değerleri uygulanmıştır. Diğer polimer numuneleri karşılaştırma için 7 gün boyunca laboratuvarında kürlenmiştir. Bütün numunelerde çekme mukavemeti, kopma enerjisi, sertlik, su emme ve mikroyapı incelemeleri yapılmıştır. Sonuçlar, polimer materyallerini 7 gün boyunca oda sıcaklığında tutmak yerine mikrodalga işlemine tabi tutmanın, daha kısa sürede benzer veya daha yüksek gerilme mukavemeti, sertlik ve darbe tokluğu değerleri verdiğini göstermektedir.

Anahtar Kelimeler: Mikrodalga; kürlenme; polimer; ön bekleme süresi.

Bu makaleye atf yapmak için

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1. Introduction

Epoxy resins are gaining popularity in various fields of engineering, for example in the electrical industry, and structural applications in both the commercial and military aircraft industries. Epoxy resin is classified as an industrial thermosetting resin and it has relatively good mechanical and physical properties. At present, the most common curing technique for epoxy resin is conventional oven curing, which is time-consuming. This has led to the study of alternate curing techniques using microwave heating, which is expected to shorten the curing period [1-3].

The use of microwave processing could potentially revolutionize the preparation of composites. Typical cure times are reduced from hours to minutes through the use of microwave rather than conventional curing. There is thus the potential for savings to be made in the commercial manufacture, through shorter cure cycles, and since the microwave energy is largely concentrated on the sample, greater efficiency. This, in turn, could lead to further savings. For example, the higher throughput reduces the number of facilities required, thus leading to a reduction in capital equipment costs. If the microwave processing of resins is to be a useful commercial technique, it is important to understand the structure and properties of cured resins. Changes in the network structure and detailed chemistry are possible and this could affect moisture uptake and resin degradation properties. This is an area where there has been controversy in the literature [4-6].

Tanrattanakul and Tiaw [7] compared the mechanical properties of epoxy resins cured by thermal heating and microwave heating. They reported that equivalent or better mechanical properties were obtained by microwave curing, in comparison with those obtained by thermal curing. Microwave curing also provided a shorter cure time and an equivalent degree of conversion. Berg et al. [8] investigated the curing process of thin layers of epoxy resins using microwave radiation as an alternative technique. The results showed that the current achievable curing temperature and time required to cure fully the tested mixtures are about 90°C and 75 sec. The powder additions did not change significantly the hardening process. Yusoff et al. [9] compared the difference between microwave heating and conventional thermal heating in fabricating carbon/epoxy composites. The experiments resulted in both conventional and microwave heating yielded similar glass transition temperatures. Microwave cured composites had higher void contents than conventionally cured composites. Maity et al. [10] investigated the curing of epoxy resin by new aromatic amine functional curing agents along with mechanical and thermal evaluation. Mechanical properties study of cured epoxy networks reflects the greater rigidity of amine functional aniline formaldehyde condensates cured epoxy matrix than amine functional chloroaniline formaldehyde condensates cured matrix. Yarlagadda and Hsu [11] studied the glass transition temperature of alternative mold materials by using both microwave heating and conventional oven heating. Results indicate that microwave curing can improve the strength of the samples better than the samples cured through conventional heating. Finally, the results for the glass transition temperature for microwave-cured samples showed an increase in glass transition temperature when compared to conventional-cured samples. Wallace et al. [12] used microwave and conventional oven heating for a cure a resin system, PR500 (3M). It was found that the epoxy-amine reaction occurs to a greater extent than the epoxy-hydroxyl reaction in the microwave cured resin compared to the thermally cured resin. Boey et al. [13] investigated the effect of different curing agents in microwave curing for an epoxy system (epoxy Araldite). They reported that the curing was strongly dependent on the curing agent used.

2. Experimental Studies

2.1. Materials and Sample Preparation

Commercially available Teknobond 300 epoxy resin along with hardener was used as matrix material in the fabrication of different specimens. Epoxy resin has a modulus of 3.42 GPa and

possesses a density of 1100 kg/m^3 . For processing, the mix ratio (by weight) of epoxy resin (2 parts) and hardener (1 part) were used as specified. The required mixture of resin & hardener (Table 1) was made by mixing them in (2:1) parts in a beaker, stirring the mixture using a rod taking care that no air was entrapped inside the solution.

Table 1. Composition of epoxy-based polymer composites

| Mixture code | Epoxy resin*, kg/m^3 | Quartz, kg/m^3 |
|-----------------------|-------------------------------|-------------------------|
| Q0 (epoxy resin) | 100 | - |
| Q50 (epoxy composite) | 50 | 50 |

*Epoxy resin was used with hardener (2:1)

Table 2 shows the performed experimental parameters (A and B group). The pure epoxy (Q0) and the epoxy composite (Q50) samples were kept at room temperature for 0, 30, 60 and 90 min and then treated in a microwave oven at 800W for 20 sec (A group). Group B samples were kept at room temperature for 30 min and then subjected to microwave treatment at 800W for 20 sec.

Table 2. Experimental parameters

| Samples | Pre-waiting time, (Minute) | | | | Applied power values, (Watt) | | | | |
|-----------------|----------------------------|----|----|----|------------------------------|-----|-----|-----|-----|
| | 0 | 30 | 60 | 90 | 100 | 300 | 450 | 600 | 800 |
| Q0 and Q50 | 0 | 30 | 60 | 90 | 100 | 300 | 450 | 600 | 800 |
| Processing Time | 800W- 20 sec. | | | | 20 sec. | | | | |
| A Group | | | | | B Group | | | | |

Shore D hardness of the specimens was measured at 8 different locations at the same distance from the surface and averages were calculated. Hardness, toughness, tensile strength values and the SEM microstructures of the epoxy resin and quartz specimens were measured depending on the pre-waiting time and microwave power.

3. Results and discussions

3.1. Hardness

Figures 1 and 2 show the hardness values of the Q0 and Q50 samples (groups A and B). The hardness values of the Q0 (reference sample) which was untreated and kept at room temperature for 7 days was 78.5 SD and that of the Q50 samples was 89 SD (Figure 1). The pre-waiting time of the hardness values of the Q0 and Q50 samples was observed to have increased slightly. A 90 min pre-waiting time reduced the hardness value of the Q50 sample slightly (Figure 1). Figure 2 shows the hardness values of the Q0 and Q50 samples subjected to different microwave power values. The hardness values of both the Q0 and the Q50 samples were observed to increase with an increase in the pre-waiting times. The hardness values of the samples subjected to the pre-waiting process and then to microwave treatment at 800W were close to those of the Q0 and Q50 samples kept at room temperature for 7 days. As a result of the 60 min pre-waiting time, the hardness value of the Q0 was 84 and that of the Q50 was 93 SD (Figure 1). By conducting a microwave treatment process, the hardness values obtained were similar to or higher than those of the Q0 and Q50 samples subjected to a 1 hour pre-waiting time and then kept at room temperature for 7 days (Figure 1). The microwave treatment process enabled us to save time by reducing the 7-day pre-waiting time to 1 hour (Figure 1). As can be seen in Figure 2, similar results were observed for the samples subjected to microwave treatment at 5 different power values. The hardness value of the Q0 sample subjected

to a 30 min pre-waiting time and then to the microwave treatment at 600W was 75 SD, while that of the Q50 sample was 85 SD. The hardness value of the Q0 sample at 800W was 78 SD, while that of the Q50 sample was 90 SD. Consequently, when the microwave treatment process is compared with the 7-day pre-waiting time at room temperature, it can be stated that materials with the same hardness values can be achieved in a shorter time.

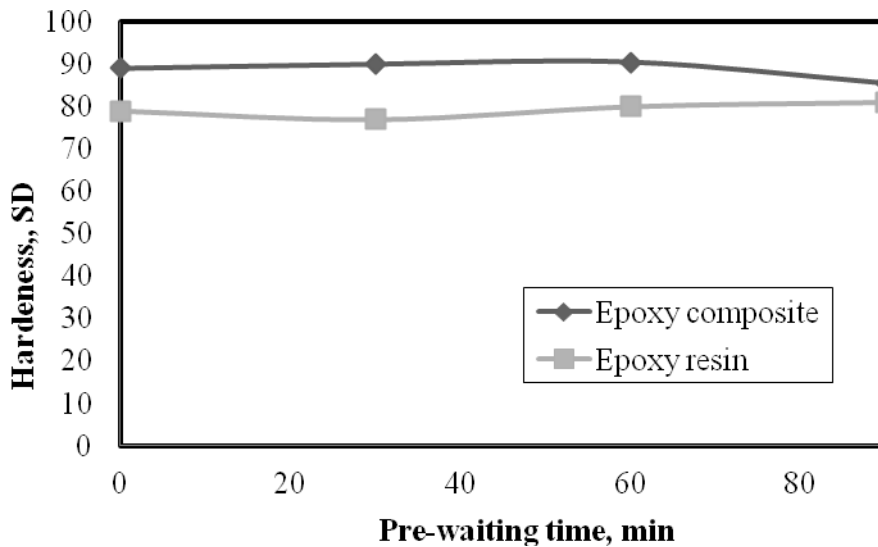


Figure 1. The hardness of epoxy resin after microwave depending on pre-waiting time

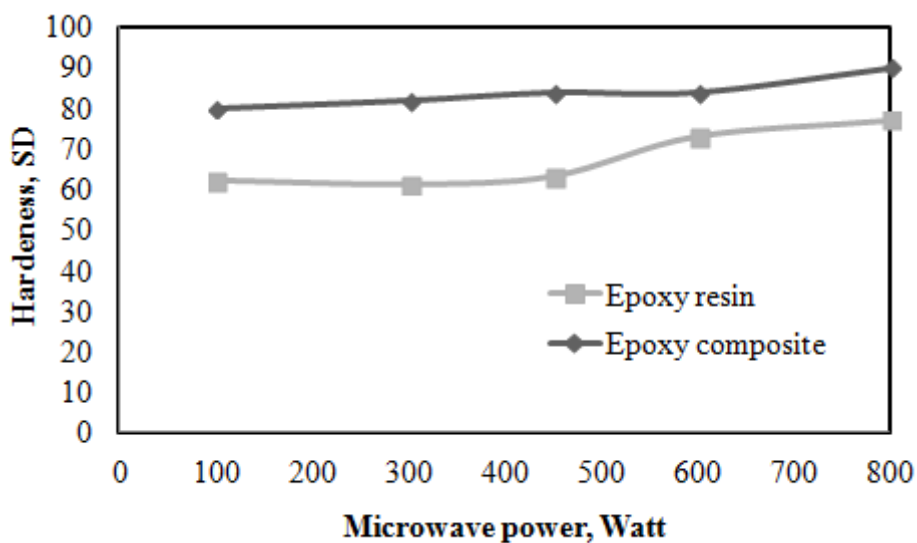


Figure 2. The hardness of epoxy composite with silica sand after microwave depending on pre-waiting time

3.2. Impact and Tensile

Figures 3 and 4 show the impact toughness values of the Q0 and Q50 samples (groups A and B). The impact toughness values of the Q0 (reference sample), which was untreated and kept at room temperature for 7 days was 37J and that of the Q50 sample was 23J (Figure 3). The impact toughness values of the Q0 and Q50 samples subjected to a pre-waiting process for 30, 60 and 90 minutes were detected to be 23.5J, 25J, 27J and 28J, 32J and 35J, respectively. The impact toughness values of the Q0 and Q50 samples increased with an increase in the pre-waiting time. The impact toughness values of the reference samples which were untreated and kept at room

temperature for 7 days decreased with exposure to pre-waiting, while those of the Q50 sample increased with an increase in the pre-waiting time.

Comparison of the impact toughness values of the samples subjected to pre-waiting and then to microwave treatment at different power values with those of the reference samples; Q0 (37J) and Q50 (23J), shows that the impact toughness values of the Q0 sample were 43J, 75J, 33J, 37J and 27J for 100W, 300W, 450W, 600W, and 800W, respectively while they were 30J, 29J, 28J, 30J and 29J for 100W, 300W, 450W, 600W, and 800W, respectively (Figure 4). The impact toughness value of the reference Q0 sample doubled with the exposure to the microwave process at 300W. The impact toughness values of the samples subjected to the microwave treatment at 5 different power values were observed to be higher than those of the reference Q50 sample. These results indicate that subjecting polymer materials to microwave treatment instead of keeping them at room temperature for 7 days yields similar or higher impact toughness values in a shorter time. The impact toughness values of the Q0 sample were observed to decrease when the microwave treatment was carried out above 300W. This was because the hardening reactions of the mixtures accelerated with an increase in the applied power values and as they accelerated, a small amount of bubble formation was observed. These bubbles caused a decrease in fracture toughness.

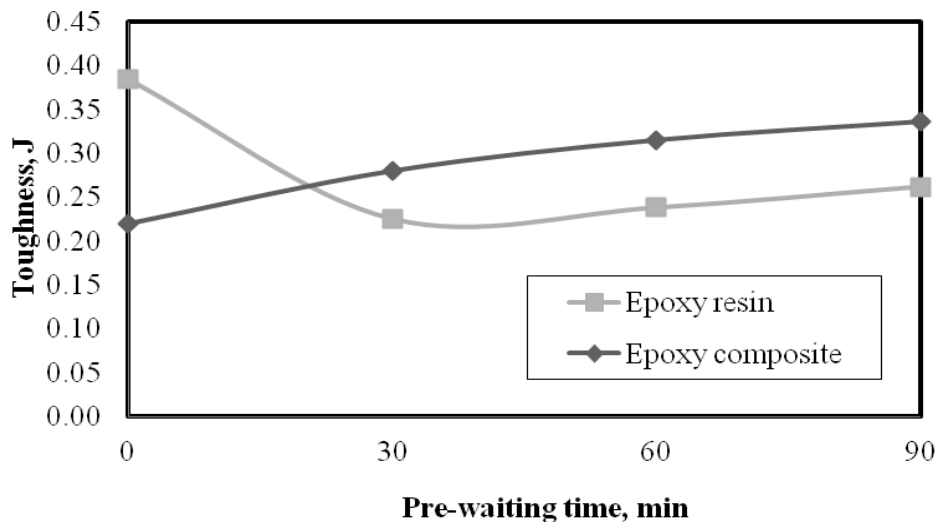


Figure 3. The toughness of epoxy resin after microwave depending on pre-waiting time

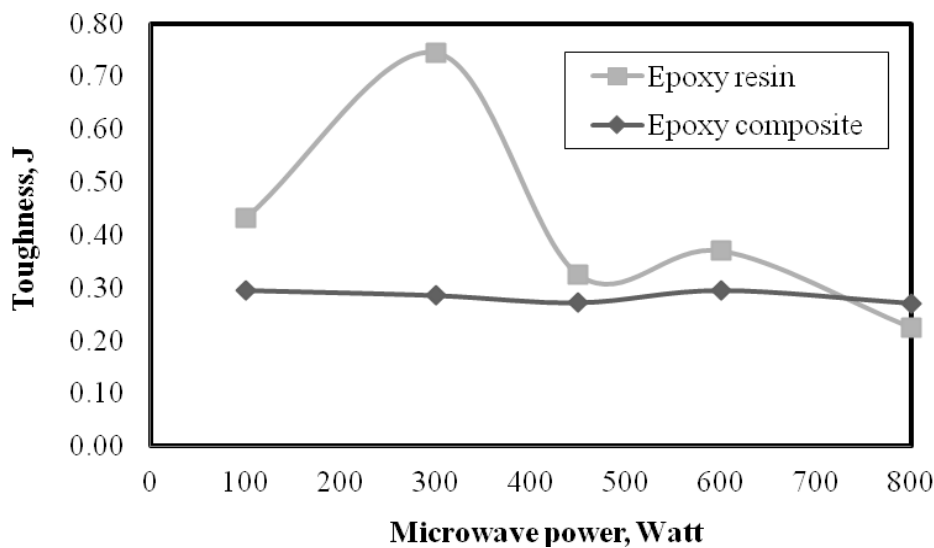


Figure 4. The toughness of epoxy composite with silica sand after microwave depending on pre-waiting time

Figures 5 and 6 show the tensile strength values of the Q0 and Q50 samples (groups A and B). The tensile strength values of the Q0 and Q50 (reference samples) which were untreated and kept at room temperature for 7 days were 23.5 MPa and 7 MPa, respectively (Figures 5 and 6). The tensile strength values of the Q0 and Q50 samples subjected to the pre-waiting process for 30, 60 and 90 minutes were detected to be 3MPa, 21.5 MPa, 21 MPa, and 8 MPa, 7.5 MPa, and 7 MPa, respectively. The tensile strength values of both samples subjected to pre-waiting and the reference samples were close to each other. The tensile strength values of the Q0 and Q50 samples slightly decreased with an increase in the pre-waiting time. By carrying out pre-waiting and then microwave treatment instead of keeping the samples for 7 days at room temperature, materials with similar tensile strength values were obtained in a shorter time. These results indicate that by accelerating polymeric reactions, microwave treatment enables us to obtain samples with similar tensile strength values in a shorter time, without having to keep them at room temperature for 7 days.

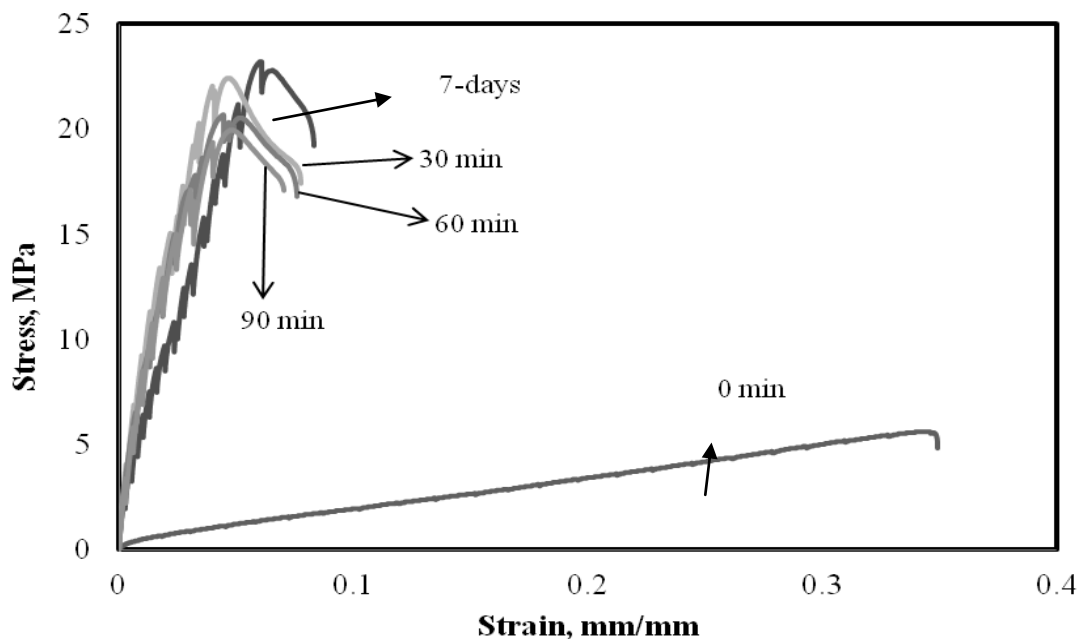


Figure 5. Tensile strength of epoxy resin after microwave depending on pre-waiting time

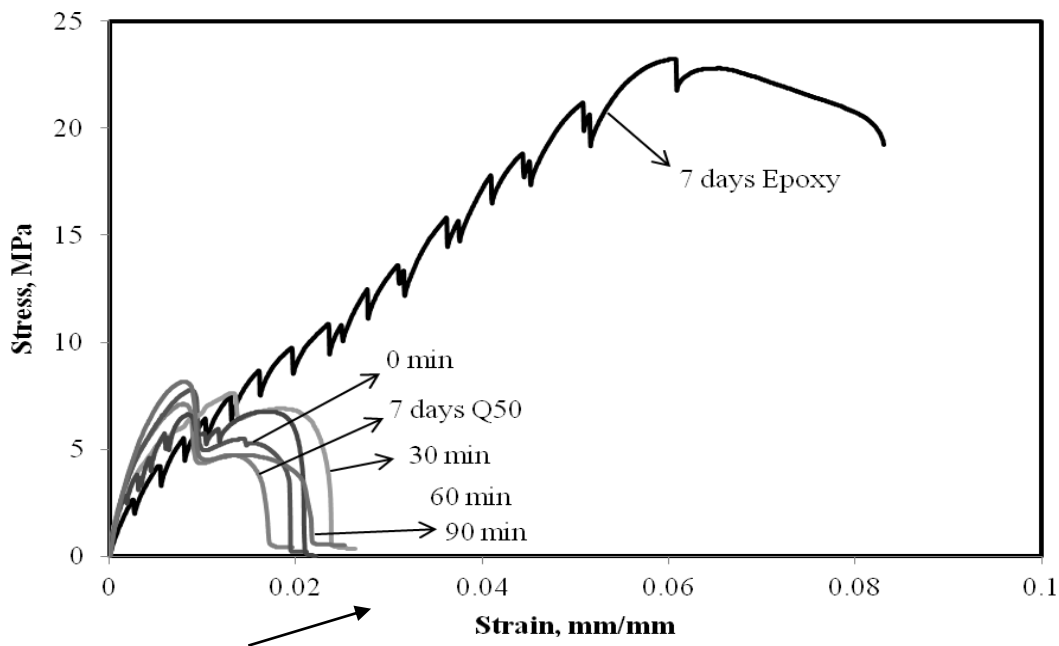


Figure 6 Tensile strength of epoxy composite with silica sand after microwave depending on pre-waiting time

Comparison of the tensile strength values of the samples subjected to pre-waiting and then to microwave treatment at different power values, with those of the reference samples; Q0 (23.5MPa) and Q50 (7MPa), shows that the tensile strength values for the Q0 sample were 7.5 MPa, 8 MPa, 8.5 MPa, 9MPa, and 23MPa at 100W, 300W, 450W, 600W, and 800W, respectively (Figure 7), while 4 MPa, 5 MPa, 6.5 MPa, 8 MPa, and 7.5 MPa for the Q50 sample at 100W, 300W, 450W, 600W, and 800W, respectively (Figure 8). Tensile strength values were observed to increase with the increase in microwave power values (Figure 7). Although tensile strength values were low at low power values, the strain values were found to be higher than those of the reference Q0 and Q50 samples.

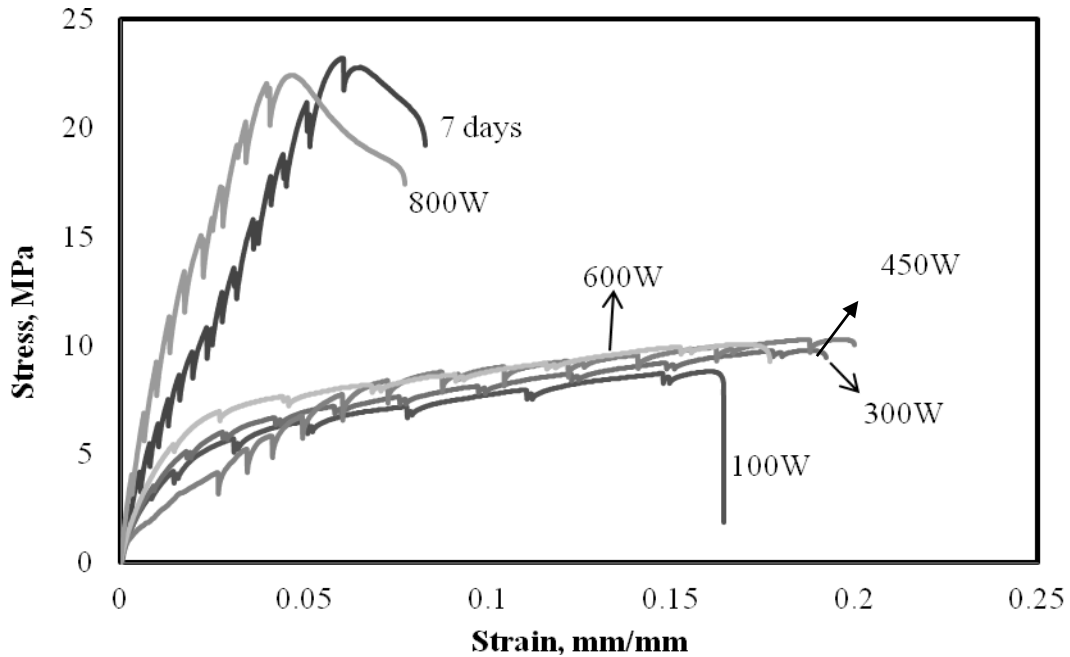


Figure 7. Tensile strength of epoxy without filler after microwave depending on the power

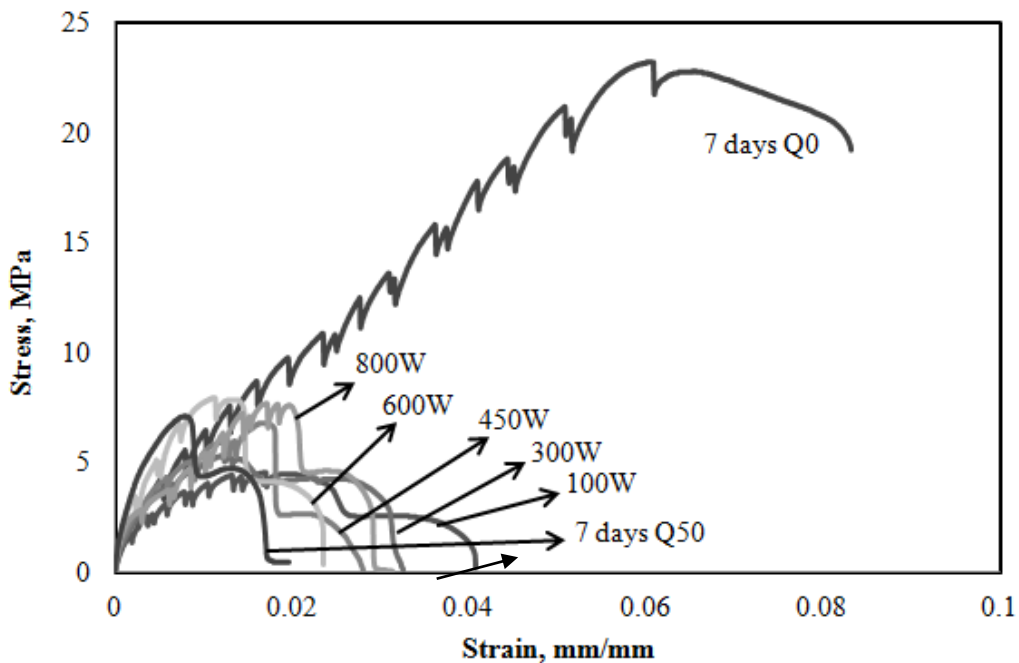


Figure 8. Tensile strength of epoxy composite with silica sand after microwave depending on the power

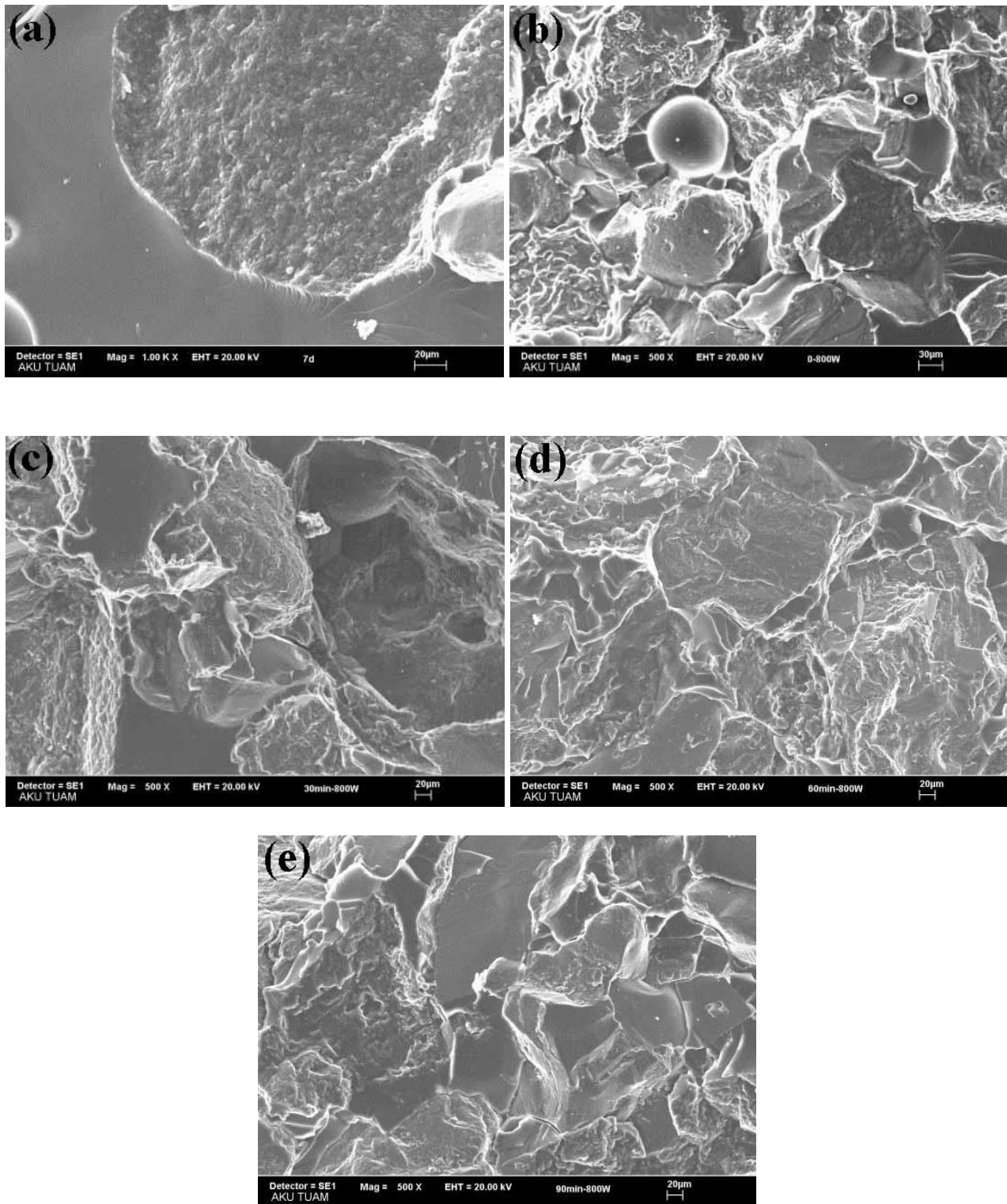


Figure 9. SEM image of epoxy composite with silica sand after microwave depending on power; a)7 days, b) 0min-800W, c) 30min-800W, d) 60min-800W, e) 90min-800W

The unit elongation values were observed to decrease with an increase in the microwave power values (Figures 7 and 8). Similar tensile strength values were obtained from the pure epoxy sample kept at room temperature for 7 days and the sample subjected to the microwave treatment at 800W (7 days; 23.5MPa, 800W; 23MPa). Similar tensile strength values were obtained from the Q50 sample subjected to the microwave treatment and the reference Q50 sample.

3.3 Microstructure Findings

Figure 9 shows the fracture surface SEM microstructures of the Q0 samples subjected to microwave treatment at different power values. The figure indicates that the SEM microstructures of the pure epoxy sample (Reference Q0) kept at room temperature for 7 days and the samples subjected to the microwave treatment at 800W were similar to each other.

Figure 10 shows the SEM microstructures of the Q50 samples subjected to microwave treatment at 800W at different pre-waiting times. It is quite evident from all the samples that the epoxy wrapped around the sand particles intensively and bound them together. Air bubbles entrained during chemical reactions were observed in the microwave treated sample which was not subjected to pre-waiting (Figure 10b). However, no air bubbles were observed in the samples subjected to pre-waiting (Figures 10c-10e).

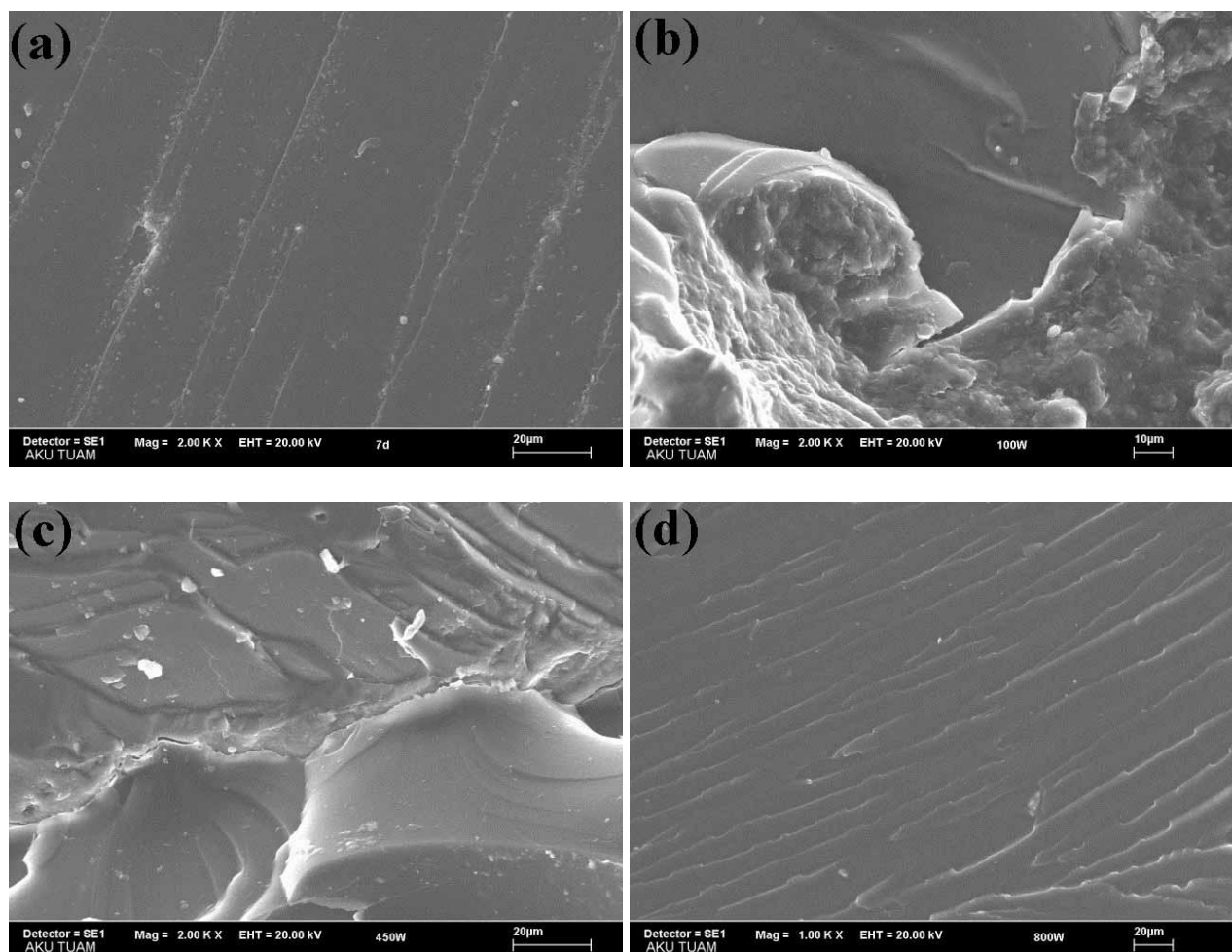


Figure 10. SEM image of epoxy without filler after microwave depending on power; a)7 days, b) 100W, c) 450W, d) 800W

4. Conclusions

This study examined the effect of different pre-waiting times and different microwave power values on some mechanical properties of pure epoxy (Q0) and epoxy composite (Q50) samples to improve their mechanical properties and shorten the time in which they gain strength. The results are as follows:

- The hardness value of the untreated Q0 (reference sample) which was kept at room temperature for 7 days was 78.5 SD and that of the Q50 sample was 89 SD.
- As a result of a 60 min pre-waiting time, the hardness value of the Q0 was 84 and that of the Q50 was 93 SD. The hardness values of both the Q0 and the Q50 samples were observed to increase with an increase in the pre-waiting time.
- The hardness value of the Q0 sample was 78 SD while that of the Q50 sample was 90 SD, as a result of microwave treatment at 800W. Comparing/contrasting the microwave treatment process with the 7-day curing time at room temperature reveals that materials with the same hardness values can be achieved in a shorter time.
- The impact toughness values of the reference Q0 and Q50 samples were determined as 37J and 23J.
- The impact toughness values of the Q0 and Q50 samples subjected to a pre-waiting process for 30, 60 and 90 minutes were detected to be 23.5J, 25J, 27J and 28J, 32J and 35J, respectively
- The impact toughness values of the Q0 and Q50 samples increased with an increase in the pre-waiting time.
- The impact toughness values of the reference Q0 sample decreased with exposure to pre-waiting, while those of the Q50 sample increased with an increase in the pre-waiting time.
- Comparison of the impact toughness values of the samples subjected to microwave treatment at different power values with those of the reference samples; Q0 (37J) and Q50 (23J), shows that the impact toughness values of the Q0 sample were 43J, 75J, 33J, 37J and 27J for 100W, 300W, 450W, 600W, and 800W, respectively while 30J, 29J, 28J, 30J and 29J for 100W, 300W, 450W, 600W, and 800W, respectively
- The impact toughness value of the reference Q0 sample rose from 37J to 75J after it was subjected to microwave treatment at 300W.
- The impact toughness values of the samples subjected to microwave treatment at 5 different power values were observed to be higher than those of the reference Q50 sample.
- Subjecting polymer materials to microwave treatment instead of keeping them at room temperature for 7 days yielded similar or higher impact toughness values in a shorter time.
- The tensile strength values of the reference Q0 and Q50 samples were 23.5 MPa and 7 MPa, respectively.
- The tensile strength values of the Q0 and Q50 samples subjected to a pre-waiting process for 30, 60 and 90 minutes were detected to be 3MPa, 21.5 MPa, 21 MPa, and 8 MPa, 7.5 MPa and 7 MPa, respectively.
- Similar tensile strength values were obtained from both the samples subjected to pre-waiting and the reference samples.
- Microwave treatment accelerated the polymeric reactions and enabled us to obtain samples with similar tensile strength values in a shorter time without having to keep them at room temperature for 7 days.
- The tensile strength values of the Q0 and Q50 samples were 7.5 MPa, 8 MPa, 8.5 MPa, 9MPa, and 23MP at 100W, 300W, 450W, 600W, and 800W, respectively while those of the Q50 sample were 4 MPa, 5 MPa, 6.5 MPa, 8 MPa, and 7.5 MPa at 100W, 300W, 450W, 600W, and 800W, respectively.
- The tensile strength values were observed to decrease with an increase in microwave power values.
- Although tensile strength values were low at low power values, the strain values were found to be higher than those of the reference Q0 and Q50 samples.
- Similar tensile strength values were obtained from the pure epoxy sample kept at room temperature for 7 days and the sample subjected to the microwave treatment at 800W (7 days; 23.5MPa, 800W; 23MPa).

- Given the fact that the maximum mechanical properties of polymer materials were reached in 7 days, the mechanical properties of the samples subjected to microwave treatment were found to be close to the hardness values of the reference samples as a result of a 1-day pre-waiting time.

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