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Authors: Şevki EREN, Serkan SUBAŞI

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Effect of The Filler Type and Particle Distribution Changes on Polyester Matrix Composites

Şevki EREN*[1], Serkan SUBAŞI[2]

Abstract

In this study, the effect of the usage of additives at different initiator ratios as well as the usage of fillers of different types and different grain distributions on resin consumption and compressive strength of polyester matrix composites were investigated. Orthophthalic unsaturated polyester resin (UP) was used as a matrix. The initiator (methyl ethyl ketone peroxide) at the ratio of 1.0%, 1.5%, and 2.0% and the accelerator (cobalt octoate) at the ratio of 1.0% by weight was used to start the polymerization. Silica, basalt, and quartz sand were used as the filler type. All fillings used in the study were prepared in the grain size distribution of the American Foundry Society (AFS 40-45) and the grain size distribution determined by reference to the Fuller equation (F 1.0). Resin consumption and compressive strength of produced composites were determined, and SEM analyses were carried out. As a result of the study, in all filled composites, the minimum resin consumption was achieved at a starting rate of 1.0%. The highest compressive strength was determined as 130.43 MPa in the basalt filled composite and in the AFS40-45 grain distribution.

Keywords: Polyester matrix composites, filler grain distribution, resin consumption, fuller equation, American foundry society

1. INTRODUCTION

The mechanical properties of the composite are affected by the size, shape, aspect ratio, and distribution of the reinforcing particles [1-3]. It has been reported that the increase of the specific surface area and the contents of the fillers enhance the mechanical and impact properties of the composites. However, when the size of the fillers becomes smaller and the content of the fillers becomes higher, the viscosity of the composite resin will be too high to process. In that case, the interfacial strength will be a more important factor due to the increased surface area of the fillers [4]. Fuller and Thompson [5], emphasized the important effect of aggregate grading on the properties of concrete. The problem of the best possible proportioning of aggregates and their contribution to optimal proportioning for the concrete mixture has been the subject of many experimental and theoretical investigations. One early example is presented by Fuller and Thompson 1907 in a series of curves which are

* Corresponding author: seren@ahievran.edu.tr
[1] Ahi Evran University, Vocational School of Technical Sciences, Kırşehir, Turkey.
ORCID: https://orcid.org/0000-0003-0773-4034
[2] Düzce University, Engineering Faculty, Düzce, Turkey.
E-Mail: serkansubasi@duzce.edu.tr
ORCID: https://orcid.org/0000-0001-7826-1348

currently used for the optimization of concrete and asphalt aggregates [5].

Most foundries in the United States use the AFS grain fineness number as a general indication of sand fineness [6]. This number is calculated from the size distribution, which is determined by standard ASTM sieves. Each fraction is multiplied with a weighting factor, the results are added together and divided by 100. AFS number gets bigger as the average size decreases. The AFS number is considered to be proportional to the number of grains per unit weight [7]. In the industry, AFS 35 (390μm), 40 (340 μm), 45 (300 μm), 50 (280 μm), 55 (240 μm), 60 (220 μm), 65 (210 μm), 70 (195 μm), 80 (170 μm), 90 (150 μm), etc. different AFS grain fineness numbers are used [6].

The primary purpose of fillers is to restrict the movement of the polymer chain, thereby increasing hardness, abrasion resistance, stiffness, and strength but reducing ductility. [1, 8-11]. They can also be used to reduce the cost of the product, control the shrinkage of the product, or increase the formatting of the material [9, 10]. Several analyses have been carried out to study the effects of particulates on the mechanical properties of polymeric composites;

Haddad and Kobaisi [12] determined the pressure strength of the polymer concrete produced by using coarse-grained basalt aggregate, sand, and fly ash with UP resin as 190 MPa. Ateş and Aztekin [13] conducted a study on the densities and mechanical properties of quartz sand filler reinforced (5% -45%) and UP matrix composites and obtained the highest compressive strength value as 131.009 MPa in 10% quartz sand volume ratio. Mahdi et al. [14], studied PET that consisting styrene and obtained from waste bottles using methyl ethyl ketone peroxide (MEKP) as a free radical initiator and cobalt naphthenate (CoNp) as the catalyst, then mixed with inorganic aggregate (10% w/w aggregate/resin). The maximum compressive strength was determined as 42.2 MPa that is higher (28.5 MPa) than the compressive strength of the neat polymer composites. Ateş and Barnes [15], prepared polymer concrete composite specimens by using polyester and quartz as binder and filler material, in different aggregate diagrams and binder ratio. The highest compression strength was obtained at 95.8 MPa. Ateş [16], examined the change of the compressive strength properties of the polyester and epoxy resin-based composite material having quartz sand as a filler which has different grain distributions and produced by using binder material at different ratios. The highest compressive strength value was obtained as 62.8 MPa in a mixture of 18% resin + 82% quartz filler. Akıncı [17], in his study, reported that the content of basalt filler added to the raw resin affected the structural integrity and mechanical properties of the composite. Moloney et al. [18], studied the effects of several parameters on the mechanical properties of particulate composites. The results showed that the tensile strength, tensile modulus, flexural strength, flexural modulus, compressive yield strength, and fracture toughness improved with an increasing volume fraction of silica particulates.

The main objectives of this study are to investigate the usage of additives at different initiator ratios and how particle size distribution and type of filler influence the resin consumption and compressive strength of polyester matrix composites.

This study was supported by TUBITAK (The Scientific and Technological Research Council of Turkey), University-Industry Cooperation Project (Project Number: 5140058) in Turkey.

2. MATERIALS and METHODS

2.1. Matrix Materials

Unsaturated orthophthalic polyester resin (Polipol 3562-SR) was used as a matrix structure. The initiator (methyl ethyl ketone peroxide; MEKP; Akperox ER 59) at the ratio of 1.0%, 1.5%, and 2.0% and the accelerator (Cobalt Octoate; Akkobalt RC88) at the ratio of 1.0% by weight were used to start the polymerization. Resins and additives used in the study are given in Figure 1 and the mechanical properties of raw polyester resin types are given in Table 1. Some physical properties of polyester resin mixtures used in different initiator ratios are given in Table 2.

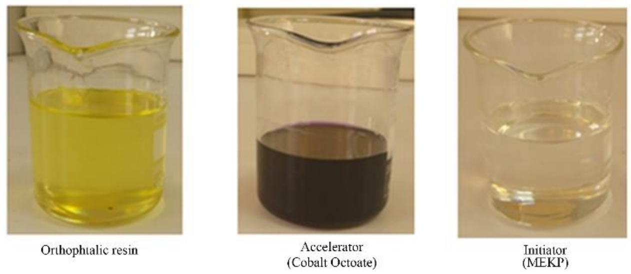

Figure 1 Visual of raw resin, initiator and accelerator

Table 1 Mechanical properties of orthophtalic resin

Property	**Orthophtalic Resin**
Tensile modülüs (MPa)	3550
Tensile strength (MPa)	74
Flexural strength (MPa)	125
Flexural modülüs (MPa)	3800
Elongation at break (tensile) (%)	3.15

Table 2 Some physical properties of polyester resin mixtures used in different initiator ratios

Initiator ratio (%)	Orthophtalic Resin		
	Gelling time (min)	Gelling temperature (0C)	Peak exotherm temperature (0C)
1.0	15	28.5	189.8
1.5	13	29	177
2.0	10	32.8	182.6

2.2. Filling Materials

Three types of fillers were used in the study: silica sand, basalt sand, and quartz sand, in the range of 0-1000 μm, with different grain distributions. A picture of the filling materials used is shown in Figure 2. The chemical and physical analyses of the filling materials used are given in Table 3 and Table 4 respectively.

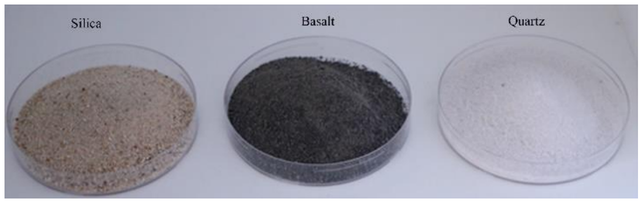

Figure 2 Filling materials

Table 3 Chemical compositions of filling materials

Chemical Composition	Filling materials (%)		
	Quartz Sand	Silica Sand	Basalt Sand
SiO2	99.18	98.94	61.21
Al2O3	0	0.08	13.61
Fe2O3	0.02	0.1	5.72
MgO	0	0	3.90
CaO	0.16	0.12	6.20
Na2O	0	0	2.63
K2O	0.03	0.05	2.83
TiO2	0.04	0.12	0.76
MnO	-	-	0.13
SO2	0.02	0.3	-
P2O5	0.01	0.01	-
Cr2O3	0.004	0.053	-
Mn2O3	0.0017	0.0035	-

Table 4 Physical properties of filling materials

Physical parameters	Filling materials		
	Quartz Sand	Silica Sand	Basalt Sand
Moisture content (%)	0.002	0.6	0.5
Relative density (g/cm^3)	2.55	2.74	2.57
Loose unit weight (g/cm^3)	1.616	1.537	1.541
Compact unit weight (g/cm^3)	1.791	1.723	1.764
Water absorption ratio (%)	2.03	2.73	2.29
Specific surface area (m^2/kg)	12.07	11.3	10.42
The average grain size (μ)	237	245	254

2.3. Mixture Design of Filled Composites

The polymerization reaction was carried out at room temperature. Firstly, the orthophtalic resin, methyl ethyl ketone peroxide, and cobalt octoate were added to the beher, and immediately after, the former mixture stirred 2 min at 400 rpm in the magnetic stirrer. Before the curing, the former prepared solution was transferred to ultrasonic homogenizer (Bandelin, RK 100 H, Germany) and sonicated for 1 min at 35 kHz to improve the homogeneity. Then the obtained mixture was transferred and the fillers that prepared according to predetermined particle distributions were added and mixed at 1000 rpm for 1 min in the ultraturrex disperser (Heidolph, Hei TORQUE 100, Germany) before the resin reached the gelling time. Finally, the obtained suspension poured for molding to a steel cylinder (35mm * 70mm) with controlled leveling and kept at room temperature for 1 h and afterward was transferred to an oven (Utest, UTD 1035, Turkey) in order to keep at 80 °C for 24h. Pre-mold, release agent (Poliya, Polivaks SV-6, Turkey) were applied to the molds to remove easily after curing. The preparation process of filled composites is shown in Figure 3.

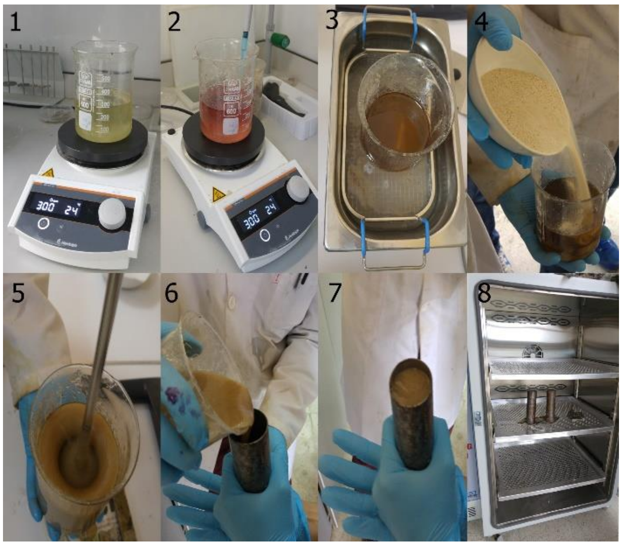

Figure 3 The preparation process of filled composites

Filling grain distributions have been prepared by referencing the Fuller equation. The reference Fuller equation is given Eq. (1) [5].

$$P = 100 \text{ x } (\frac{d}{D})^n \qquad (1)$$

In the referenced Fuller equation, n coefficient value was chosen as 1.0 and after determining the amount of material required to be taken between each sieve, these materials were mixed to form a blend and made ready for mixing with resins.

F 1.0: The grain distribution generated by taking n=1.0 in the Fuller equation

P is the total percent of particles passing through (or finer than) sieve, %

D is the maximum size of aggregate, mm

d is the diameter of the current sieve (1000μ), mm

n is the exponent of the equation

Also, fillings used in the study were prepared in the grain size distribution of the AFS 40-45. AFS 40-45 and Fuller 1.0 grain distribution curves used in the production of filled samples are given in Figure 4.

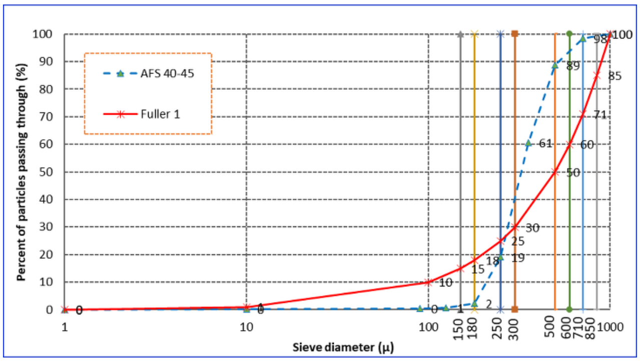

Figure 4 AFS 40-45 and Fuller 1.0 grain distribution curves

2.4. Compressive Strength Test

The compressive strength was performed according to the ASTM C 579-01 [19] standard. The loading speed is 41 MPa/min. For the pressure test, 3 samples were produced from each mixture. A visual representation of the cylindrical specimens extracted from the mold and the cutting operations of these samples is given in Figure 5, a visual representation of the cut cylinder samples for pressure testing is given in Figure 6 and a visual representation of the pressure test is given in Figure 7.

Figure 5 Cylinder samples and cutting process

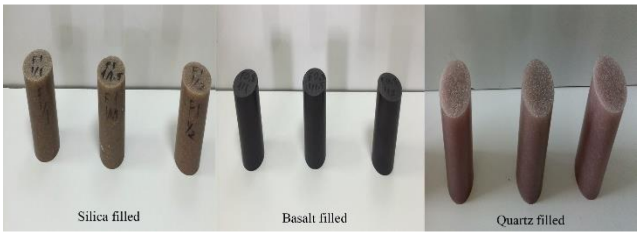

Figure 6 The pressure testing samples

Figure 7 The pressure test of the filled composites

2.5. Resin Consumption

The weights of the cut samples were weighed to determine the resin consumption quantities of the filled resin mixtures. After the samples were placed in the ash furnace, the temperature of the ash furnace was set to approximately 650 ^{0}C in order to completely blow out the polyester and the samples were kept for 2 hours at this temperature. After the samples removed from the ash furnace were kept until they reached the ambient temperature, their weights were weighed, and their resin consumption was determined. A visual representation of cut samples to determine the resin consumption is shown in Figure 8 and a visual representation of resin consumption testing of filled composites is shown in Figure 9.

Figure 8 The resin consumption samples

Figure 9 Resin consumption testing of filled composites

2.6. SEM Analysis

For SEM analysis, cross-section images were taken of the samples at 20 kV acceleration voltage (FEI, Quanta 250, Netherland). The specimens were mounted with tweezers onto a substrate with carbon tape and coated with a thin layer of gold/palladium mixture.

3. RESULTS and DISCUSSION

3.1. Compressive Strength Test Results and Evaluation

The combined representation of the average compressive strength of the filled resin mixtures in different grain distributions due to the change in the initiator ratio is given in Table 5.

Table 5 The average compressive strength values of the filled resin mixtures

Initiator ratio (%)	Compressive strength values (MPa)					
	AFS 40-45			FULLER 1.0		
	Silica sand	Basalt sand	Quartz sand	Silica sand	Basalt sand	Quartz sand
1.0	116	130	130	128	129	117
1.5	118	124	112	122	124	121
2.0	124	116	115	112	121	122

Changes in the average compressive strength of silica, basalt, and quartz-filled resin mixtures with a grain distribution of AFS40-45 and F1.0 due to the change in the initiator ratio are given in Figure 10, Figure 11, Figure 12, Figure 13 and Figure 14 respectively. The graph showing the compressive strength in the first three rows among all filled composites is shown in Figure 15.

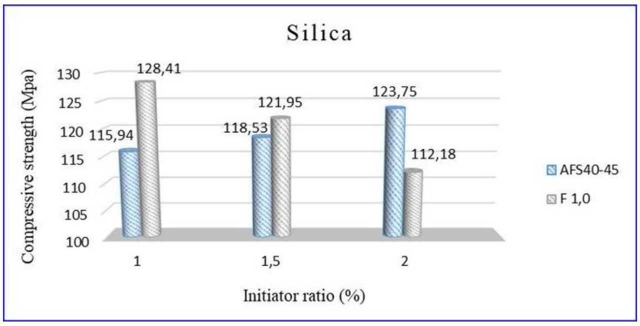

Figure 10 Compressive strengths in silica filled composites

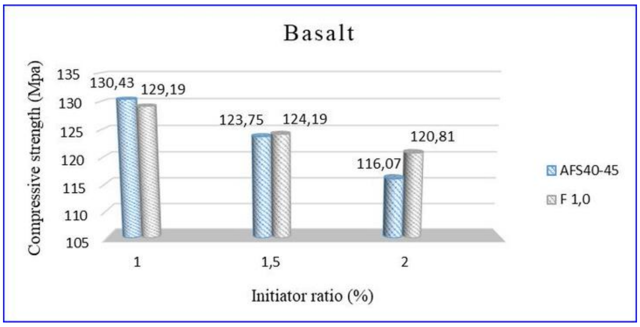

Figure 11 Compressive strengths in basalt filled composites

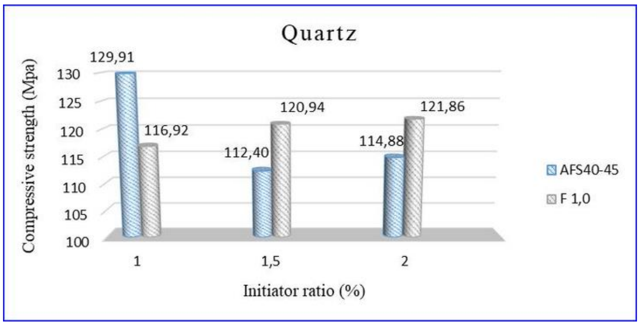

Figure 12 Compressive strengths in quartz filled composites

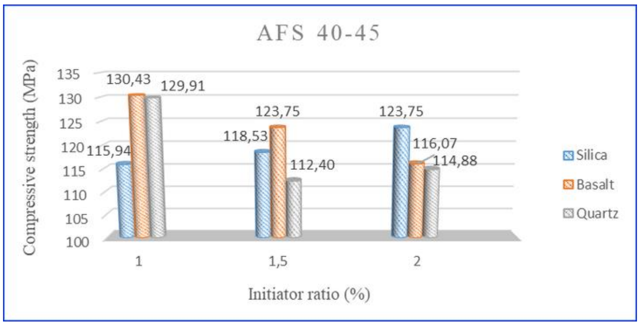

Figure 13 Compressive strengths of AFS 40-45 grain distributed fillers

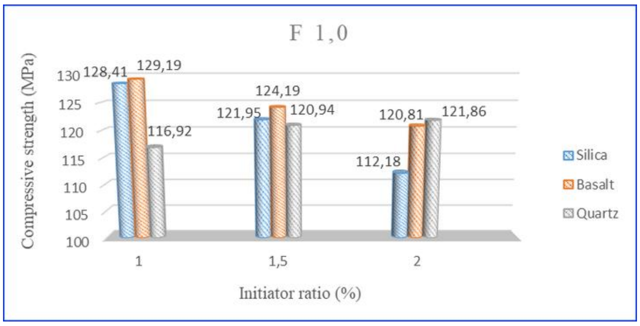

Figure 14 Compressive strengths of F 1,0 grain distributed fillers

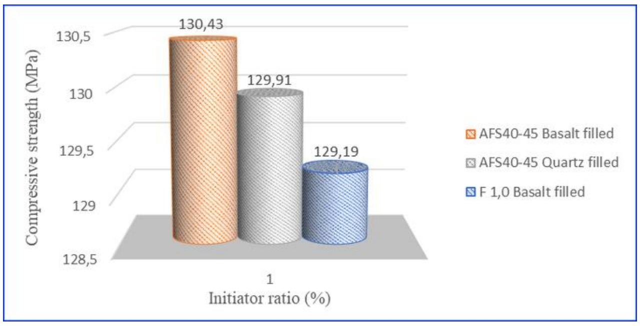

Figure 15 Compressive strengths in the first three rows

Pressure tests on silica-filled resin mixtures with AFS 40-45 grain distribution showed that their compressive strength increased as the initiator rate increased. The lowest compressive strength value was obtained at a 1.0% initiator rate and this value was determined as 115.94 MPa. The compressive strength increased to 118.37 MPa in a 1.5% initiator ratio and reached the maximum compressive strength value of 123.75 MPa in a 2.0% initiator ratio. The percentage of increases was 2.09% and 6.73% respectively. However, it is thought that by increasing the rate of initiator, the strength will decrease as the heat value of the mixture will increase.

The highest compressive strength value of silica-filled resin mixtures in F 1.0 grain distributed was determined as 128.41 MPa at the rate of 1.0% initiator. However, by increasing the rate of initiator, compressive strength also decreased. As a result, the highest compressive strength of all silica-filled resin mixtures was obtained in the F 1.0 grain distribution and the initiator ratio of 1.0%, and this strength value was obtained as 10.75% higher than the silica-filled composite in the AFS 40-45 grain distribution. When these results were evaluated together, AFS 40-45 grain distribution has uniform grain distribution, while F 1.0 grain distribution has a better-rated grain distribution, so it was observed that strength values increased only with the improvement made in grain distribution.

In basalt-filled resin mixtures, the highest compressive strength values for each different grain class were obtained at the initiator ratio of 1.0%. As the initiator ratio increased, the compressive strength values of all grain distributed mixtures decreased. The highest compressive strength value was determined as 130.43 MPa in the “AFS40-45” grain distributed mixture. The second highest strength was determined as 129.19 MPa in the F 1.0 grain distributed mixture.

The highest compressive strength value in quartz filled resin mixtures was obtained as 129.91 MPa in AFS 40-45 grain distribution and 1.0% initiator ratio. As the initiator ratio increased, the compressive strength values of AFS 40-45 grain distributed mixtures decreased, while F 1.0 grain distributed mixtures increased.

In AFS 40-45 grain distributed filled resin mixtures, the highest compressive strength was obtained in basalt and quartz fillings and a 1.0% initiator ratio. The strength value of basalt-filled composites with a 1.0 % initiator ratio was determined as 12.49% higher than silica-filled composites, while the strength value of quartz-filled composites was determined as 12.05% higher than silica-filled composites. When these results were evaluated together, it was observed that in composites with grain distribution of AFS 40-45 the filler type had a significant effect on compressive strength, and higher strengths were obtained when basalt and quartz fillings were used.

In F 1.0 grain distributed filled resin mixtures, the highest compressive strength was obtained in basalt and silica fillings and a 1.0% initiator ratio. In quartz-filled composites with the same initiator ratio, a lower strength value was obtained. The strength value of basalt and silica-filled composites with a 1% initiator ratio was determined as 10.26% and 9.4% higher than quartz-filled composites with the same initiator ratio, respectively. In addition, it was observed that the strength values of silica and basalt filled composites decreased with increasing initiator ratio, while quartz filled composites increased. When these results were evaluated together, it was observed that in composites with grain distribution of F 1.0, the filler type had a significant effect on compressive strength, and higher strengths were obtained when basalt and silica fillings were used.

- When the results of filled composites are evaluated;

Among all filled composites, the highest 3 compressive strengths were obtained as a 1.0% initiator ratio.

The highest compressive strength value was determined as 130.43 MPa in the AFS40-45 grain class basalt filled resin mixture. This strength value was found to be approximately 1.0% higher than basalt filled composites with F 1.0 grain distribution. The second highest strength value was determined as 129.91 MPa in AFS40-45 grain distributed quartz filled resin mixture. This strength value was determined as 0.55% higher than basalt filled composites with F 1.0 grain distributed.

The compressive strength value of polymer concrete, determined by Haddad and Kobaisi [12] as 190 MPa, was 45.67% higher than the maximum strength value determined in this study (130.43 MPa). When these data are evaluated, the strength values of the fillings in the composite increase as the grain diameter grows larger.

The highest compressive strength values determined in the scope of the study and the strength values determined by Ateş and Aztekin [13] on quartz-filled composites were close together.

Ateş [16] determined that the change in resin and filling ratios and grain distribution curves affected the compressive strength of polymer concrete. These results were found to support the results obtained in the study.

Akıncı [17], in his study, reported that the content of basalt filler added to the raw resin affected the structural integrity and mechanical properties of the composite.

3.2. Resin Consumption Amounts of Filled Resin Mixtures

Resin consumption amounts of filled resin mixtures in different grain distributions are given in Table 6.

Table 6 Resin consumption amounts of filled resin mixtures in different grain distributions

Initiator ratio (%)	Average resin consumption amounts (%)					
	Silica sand		Basalt sand		Quartz sand	
	AFS 40-45	F 1.0	AFS 40-45	F 1.0	AFS 40-45	F 1.0
1.0	52	49	53	51	59	53
1.5	51	50	51	51	55	55
2.0	54	52	52	52	55	55

Among all filled resin mixtures, the least resin consuming mixtures were obtained at the grain distribution of F 1.0 and the initiator rate of 1.0%. In these mixtures, the minimum resin consumption was obtained in silica-filled mixtures and this ratio was determined as 49%. In basalt filled mixtures, this ratio was 51% and in quartz filled mixtures it was 53%. Among all filled resin mixtures, the most resin consuming mixtures were obtained in composites with a grain distribution of AFS 40-45. In silica-filled composites, the amount of resin consumption obtained in F 1.0 grain distribution and 1.0% initiator ratio was 6.12% less than the amount of resin consumption in AFS 40-45 grain distribution with the same initiator ratio. In the same grain distribution comparison, 3.92% less resin consumption was achieved in basalt-filled composites, while 11.32% less resin consumption was achieved in quartz-filled composites. The reason for this is considered to be that composites in the grain class AFS 40-45 have uniform grain distribution, whereas composites in the grain class F1.0 have a better-graded distribution. It is believed that good grading of the particles allows the composite to have a less porous structure, thereby causing the composite to consume less resin.

3.3. SEM Images and Morphological Analysis of Filled Resin Mixtures

SEM images of filled composites with the highest 3 compressive strengths are shown in Figure 16, Figure 17, and Figure 18 respectively.

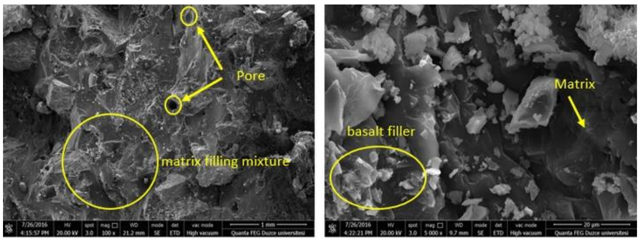

Figure 16 SEM image of AFS 40-45 distributed basalt filled composite

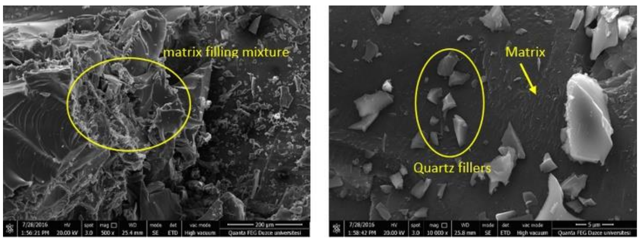

Figure 17 SEM image of AFS 40-45 distributed quartz filled composite

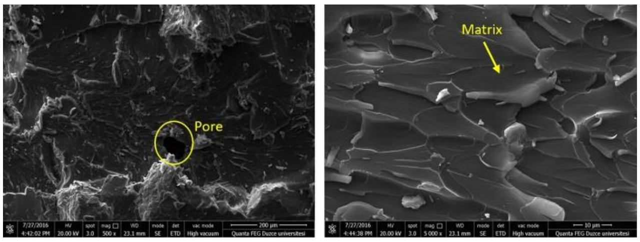

Figure 18 SEM image of F 1.0 distributed basalt filled composite

When the SEM images of the AFS40-45 and F 1.0 grain distributed basalt filled composites were examined, it was seen that the basalt fillings were homogeneously distributed, the matrix structure completely wetted the filling material and the adherence of the resin and fillings was very good. It is thought that the failure is not due to the loss of adherence because of the adhesion between the filling material and the matrix interface is very good.

When the SEM images of quartz-filled composites in the AFS 40-45 grain distribution were examined, it was observed that the quartz fillers in the polyester matrix were largely uniformly distributed within the matrix structure and that the matrix structure completely wetted the filling material.

4. CONCLUSION

Among all filled composites, the highest 3 compressive strengths were obtained as a 1.0% initiator ratio.

Among all filled resin mixtures, the three highest compressive strength was determined as 130.43 MPa in basalt filled resin mixture in AFS 40-45 grain distribution, 129.91 MPa in quartz filled resin mixture in AFS 40-45 grain distribution and 129.19 MPa in basalt filled resin mixture in F 1.0 grain distribution.

Among all filled resin mixtures, the least resin consuming mixtures were obtained at the grain distribution of F 1.0 and the initiator rate of 1.0%. Composites produced in F 1.0 grain distribution and 1.0% initiator ratio consumed less resin than composites in AFS 40-45 grain distribution. In silica-filled composites, the amount of resin consumption obtained in F 1.0 grain distribution and 1.0% initiator ratio was 6.12% less than the amount of resin consumption in AFS 40-45 grain distribution with the same initiator ratio. In the same grain distribution comparison, 3.92% less resin consumption was achieved in basalt-filled composites, while 11.32% less resin consumption was achieved in quartz-filled composites. The reason for this is considered to be that composites in the grain class AFS 40-45 have uniform grain distribution, whereas composites in the grain class F1.0 have a better-graded distribution. It is believed that good grading of the particles allows the composite to have a less porous structure, thereby causing the composite to consume less resin.

As a result of the study, it was concluded that the filler type and grain distribution used affected the mechanical properties and resin consumption of the composites.

Acknowledgments

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Research and Publication Ethics

This paper has been prepared within the scope of international research and publication ethics.

Ethics Committee Approval

This paper does not require any ethics committee permission or special permission.

Conflict of Interests

The author declared no potential conflicts of interest concerning the research, authorship, and/or publication of this paper.

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