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Ultrasonic Characterization of Polymer Based Sille Stone Powder Composite Mortars

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ABSTRACT

The Sille stone mined in the Sille Region of Konya province in Turkiye, is an andesitic rock. This stone is a material used in the construction of traditional and modern buildings. The Sille stone is cut for use in buildings. The Sille stone powder (SSP) is formed during this cutting process. The SSPs cause environmental pollution. This study was carried out to produce durable and eco-friendly new restoration mortars from the SSP that can be used in the restoration of historical buildings. The SSP composites were prepared by contributing SSP into epoxy and polyester resins in varied ratios such as 60–75 wt.%. The effect of resin types and SSP contribution ratios on the elastic properties of epoxy resin (ER)/SSP and polyester resin (PR)/SSP composites was investigated by the ultrasonic pulse-echo method. Additionally, the morphology of these composites was investigated by scanning electron microscopy (SEM). Experimental results indicated that both the longitudinal and shear wave velocity values of the PR/SSP composites were higher than those of the ER/SSP composites. Furthermore, a linear increase in the elastic properties of the obtained composites was observed with increasing amounts of SSP.

Keywords: Polymer materials, Sille stone powder, ultrasonic, nondestructive testing, restoration.

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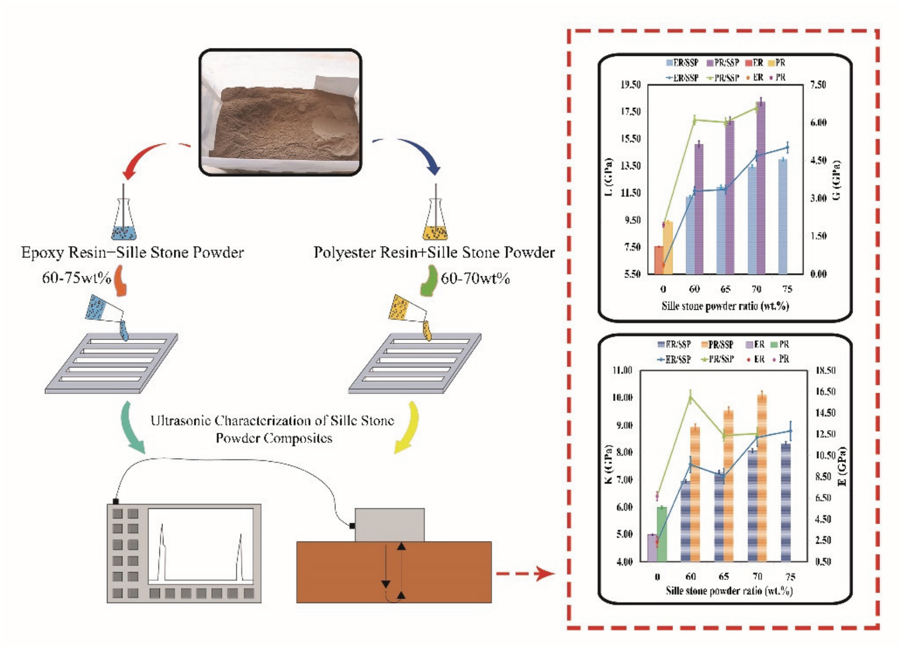

Graphical Abstract

1. INTRODUCTION

Polymers have been preferred over traditional materials due to their application in various fields. In the preference for polymers over traditional materials; the fact that they can be processed, have high strength, and are economical has been effective [1]. Additionally, the many properties of materials could be improved by adding fillers into polymer resins such as polyester, vinyl ester, and epoxy. Fillers provide volume to the material, reduce cost, and provide aesthetic properties [2]. Composite materials developed with polymer resins have been used in different fields such as aviation, maritime, automobile industries, and construction sectors. Nowadays, the potential of polymer materials has been recognized in the construction industry for the reinforcement of structures and their use as repair materials [3]. For example, by mixing polymer resins with concrete, high-performance concrete and modified mortars with high tensile strength, compressive strength, and corrosion protection have been developed [4, 5]. Polymer-modified mortars are used as repair materials in the construction industry and public works because they have a longer life, provide good insulation against moisture on the wall, have good adhesion properties, and are resistant to the negative effects of the environment [6]. Since the aggregates used in mortars affect the durability of the mortar, the demand for aggregates is increasing with the rapid development of the construction industry. The inadequacy of aggregates such as river sand used in buildings in the construction sector also damages the natural environment of the land in aggregate mining. For this reason, recycled aggregates from waste materials have had an important place in meeting the increasing demand for aggregates in the construction industry [7]. The use of waste materials in the development of new materials allows both to protect the environment and to produce more economical materials. Therefore, recycling waste materials contributes to the reduction of environmental pollution caused by the construction industry to the whole world and to sustainable development. Also, using the waste materials in polymer mortars helps improve the many properties of materials [8-11].

Sille stone is mined in the Sille region of Konya in Turkey. Sille stone is known as "Sulutas Volcanic". However, this stone contains dacite, rhyodacite, and andesite [12]. The stones in the Sille region are gray, brown, and pink [13, 14]. The different colors and high strength of this stone are used in the construction of buildings in and around Konya and the restoration of historical buildings. However, for this stone to be used in buildings, it must be cut to certain dimensions. In Sille quarries, stones are cut into blocks with different methods. These stones, processed for decorative purposes, are made economically valuable. However, a large amount of stone powder is formed during the cutting process of Sille stones. The use of Sille stone powder, which is formed during the cutting of the stone, as a repair mortar in the repair and strengthening of structures is important both in terms of environmental protection and economically.

The durability of building materials affects the lifetime of the buildings. Deteriorations and damage occur due to external effects such as corrosion, humidity, and earthquakes during the usage period of the buildings. The material used in the construction of the structures is important in providing resistance to these external effects. The use of polymers in the construction industry has become increasingly common recent years. Epoxy and polyester resins draw attention due to their good mechanical properties, low viscosity, and fast curing [15, 16]. Therefore, many epoxy and polyester resin-based composites have been extensively studied in engineering applications recently [15-21].

One of the most common non-destructive testing methods used to determine the material's properties is the ultrasonic testing (UT) technique [22, 23]. One of the most important advantages of the UT method is the non-destructive determination of the width of the cracks formed by the wear of the materials, the location of the cracks, and the compressive strength of the materials. Thus, the UT method has proven useful in determining the structural behavior, microstructure, and mechanical properties of polymer composites [24-26]. UT method is used to evaluate composites' elastic properties and microstructures [27]. For example, it has been used to estimate the strength of rock [28], concrete [29], brick [30], and steel [31] by the UT method. Also, the UT method enables the determination of the defects and damaged areas of the composite materials as well [32]. For instance, Sun and Zhu [33], used the UT technique to detect internal defects of steel plates in thick concrete walls. They determined that the UT technique can be used to evaluate the early and late age conditions of three connecting rods with different bonding conditions in concrete. Additionally, the UT method has been used to determine the mechanical properties of composite mortars used in historical and ancient buildings as well [34-38]. For example, in the study conducted by Branco et al. [35], the binding properties of lime mortars, masonry structures, and wall coverings in historical buildings were investigated. In addition, the compressive and flexural strengths and ultrasonic velocities of the mortars were also measured. Since it is not possible to take core samples from historical buildings, the mechanical properties of the materials used in the repair of historical buildings should be known. Therefore, it is extremely important to determine the mechanical properties of the materials used in the repair of historical buildings with the UT method [22, 39]. In the literature, many studies have been published on the use of the stone, such as the mechanical properties of Sille stone [40-42], the effect of salt crystallization on historical buildings and monuments [43], the effects of the freeze-thaw (F–T) cycle of rocks [44], the sulfate effect of building stones [45], on the use of stone in floor tiles [46] and the physicochemical properties of heat-treated Sille stone [47]. However, the development of repair mortar to be used in the repair of Sille stone structures and cultural assets in Konya has not been mentioned in the literature. Moreover, as far as we know, no studies have been conducted on the use of Sille stone powder (SSP) with epoxy and polyester resins and the ultrasonic characterization of the epoxy resin (ER)/SSP and polyester resin (PR)/SSP composites. Therefore, this study was carried out to produce durable and environmentally friendly new restoration mortars from SSP that can be used in the restoration of traditional structures (for example: houses, mansions, etc.) built with Sille stone in Konya and determine the elastic properties of these composite mortars by the UT method. Moreover, this study was conducted to reduce the cost of raw materials in the preparation of mortars and evaluate the waste products generated during the processing of natural stones.

2. MATERIALS AND METHODS

2.1. Materials

Commercial bisphenol-A type epoxy resin (BRTR Kimya A.Ş., Turkiye), and polyester resin containing 0.2% by weight of Cobalt octoate 6% accelerator (Kompozit Pazarı, Turkiye) were used to produce composite materials in the study. Cycloaliphatic polyamine (BRTR Kimya A.Ş., Turkiye) and methyl ethyl ketone peroxide (MEK-P) (Kompozit Pazarı, Turkiye) were used as hardeners. The properties of epoxy resin, polyester resin, and SSP used in this research are given in Table 1. In addition, Sille stone powder (SSP), which was used as a filling material in the study, was obtained from the quarry in the Sille region (Konya, Turkiye). The chemical content of the SSP is given in Table 2.

Table 1 - Some features of matrix systems and SSP used in this research [40, 48-50].

Used Materials	Features of Matrices and SSP	
Epoxy resin (ER)	Color and appearance	: Transparent and Glossy
	Density (g.cm^{-3} at 25°C)	: 1.10
	Mixing Ratio	: 5/3
	Mixture Life at 25°C (Minutes)	: 30
	Drying Time at 25°C (Hours)	: 12
Polyester resin (PR)	Color	: Yellowish
	Density (g.cm^{-3} at 20 °C)	: 1.12 – 1.14
	Viscosity (cps, at 20 °C)	: 550 – 650
	Gelation time (Minutes)	: 4 – 8
	Exothermic warming (°C)	: 160 – 200
	Boiling point (°C)	: 145.2 °C

Table 1 - Some features of matrix systems and SSP used in this research [40, 48-50]. (continue)

Sille stone powder (SSP)	Color	: Pink
	Density (g.cm^{-3} at 20 °C)	: 2.26 – 2.35
	Water absorption (%, at 23 °C)	: 3.9
	Melting point(°C)	: 2000
	Fineness (μm)	: < 150

Table 2 - Chemical content of the SSP.

Content of the SSP	SiO_2	Al_2O_3	K_2O	Na_2O	MgO	CaO	Fe_2O_3	TiO_2	ZrO_2
%	66.67	13.24	4.11	4.94	1.09	5.07	3.66	0.52	0.39

2.2. Preparation of the ER/SSP, and the PR/SSP Composites

Within the scope of this research, two types of composite series were produced. For the first type of composite series, a cycloaliphatic polyamine hardener was added to the neat ER at a ratio of 5:3 by weight to obtain a hardened neat ER sample. Then, the SSP was added to neat ER in 75, 70, 65, and 60 wt.% ratios. Each mixture of the ER/SSP obtained was mixed for 3

Figure 1 - Production stage of composite mortars.

minutes, and after that, cycloaliphatic polyamine hardener was added to each of the ER/SSP mixtures at a ratio of 5:3 by weight. Finally, each of the ER/SSP composite mortar mixtures obtained was mixed for 3 minutes as well. For the second type of composite series, a methyl ethyl ketone peroxide hardener was added to the polyester resin containing cobalt octoate accelerator in 1.5 % by weight and mixed for 3 minutes to obtain the hardened neat PR sample. The SSP has added to neat PR in 75, 70, 65, and 60 wt. % ratios for obtaining the PR/SSP composite mortars. Then each mixture of the PR/SSP obtained was mixed for 3 minutes. Then, methyl ethyl ketone peroxide hardener was added to each of the PR/SSP composite mortar mixtures at 1.5% by weight and mixed for 3 minutes as well (Figure 1 and Table 3). The ER/SSP and PR/SSP composite mortar mixtures were poured into 20x20x20 mm molds made of polymethyl methacrylate (PMMA) by ASTM D638-14[51] standard, using the traditional hand lay-up process. Finally, all the obtained composite mortars were dried at 25±1°C for 24 hours. After completing the mechanical strength of the composite mortars in 7 days, ultrasonic measurements of the samples were carried out. However, unlike the ER/SSP composites, when the PR and SSP were mixed at a ratio of 25:75 by weight, it was observed that a suitable structure was not formed between them.

Table 3 - The abbreviations and ingredients of composite mortars.

Composite mortars' abbreviations	Combination ratio of resin type/SSP
ER	100:0
ER/SSP-1	40:60
ER/SSP-2	35:65
ER/SSP-3	30:70
ER/SSP-4	25:75
PR	100:0
PR/SSP-1	40:60
PR/SSP-2	35:65
PR/SSP-3	30:70

2.3. Density and Ultrasonic Wave Velocity Measurements

Density measurements were carried out by an analytical balance (Radwag AS220/C/2, capacity 220 g, readability 0.1 mg, Poland) and a density kit (Radwag 220, Poland) at room temperature (25°C).

Ultrasonic wave velocity measurements of composite samples were carried out by ultrasonic pulse-echo method using the flaw detector given in Figure 2a (Epoch-XT-Panametrics Olympus).

Before measuring the ultrasonic velocities of the composite samples, the thicknesses of the samples were measured using an analog micrometer (Somet, Czechoslovakia).

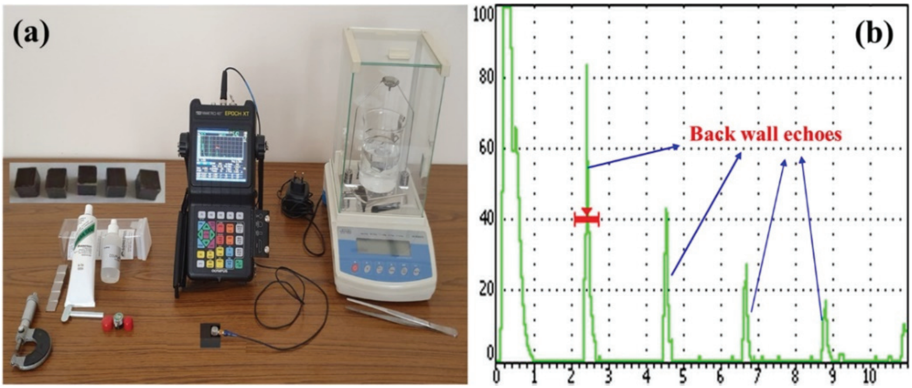

Figure 2 - a: Tools used for density and ultrasonic wave velocity measurements, b: A-scan signals of back wall echoes.

The appropriate frequency was determined as 5 MHz for both longitudinal and shear wave velocity measurements. Therefore, both the longitudinal wave velocity and shear wave velocity measurements were carried out at 5 MHz frequencies. Longitudinal wave velocity measurements were carried out using a longitudinal mode transducer (5 MHz, V116-Panametrics Olympus, USA), while shear wave velocity measurements were carried out using a shear mode transducer (V155-Panametrics Olympus, USA). At the interface between two environments with different acoustic impedances, most of the energy of the sound waves coming from the first environment is reflected before they pass to the second environment due to the impedance difference. In this case, clear peaks cannot be obtained on the oscilloscope screen since the sound wave energy coming back from the second environment is very low. This not only makes ultrasonic measurement difficult but also prevents sensitive measurements from being taken. Thus, coupling fluids are used as impedance matches [52]. The coupling fluids used nowadays can pass only 10-15% of the sound wave energy sent from the first medium to the second medium. Glycerin is one of the best coupling fluids, and it can pass up to 15% of the sound wave energy coming into it to the second medium [53]. Therefore, glycerin (BQ-Panametrics Olympus, USA) and shear wave coupling (SWC-Panametrics Olympus, USA) were used in ultrasonic longitudinal and shear wave measurements, respectively. Additionally, a constant force was applied to the ultrasonic transducer to have a stable layer at the interface of the sample. Ultrasonic wave velocity measurements were repeated 10 times to ensure the accuracy of the results.

During the ultrasonic longitudinal and shear wave velocities measurements, errors such as micro voids in the examined material can be detected by the extra peaks appearing between the peaks of two consecutive back wall echoes. Normally, in the ultrasonic pulse-echo method, the time (Δt) between two consecutive back wall echoes (for example, the 1st and 2nd, 2nd and 3rd or 3rd and 4th back wall echoes) is determined on the oscilloscope screen (Figure 2b). Ultrasonic waves pass through the material thickness twice in the time between two consecutive back wall echoes in the ultrasonic pulse-echo method. The velocity is measured by substituting these obtained values into Equation (1) below. Meanwhile, in this research, velocity measurements were carried out at points that did not give error peaks between back wall echoes as much as possible.

$$V = \frac{2\Delta d}{\Delta t} \tag{1}$$

where *V, Δd,* and *Δt* are the velocity of sound, the thickness of the sample, and the time-of-flight between subsequent backwall signals on the oscilloscope, respectively.

2.4. Determination of Elastic Constants

The elastic modulus is related to the interatomic forces. Therefore, the elastic modulus of the materials expresses the maximum strength that can be achieved. There is a direct mathematical relationship between the elastic modulus, ultrasonic longitudinal, and shear wave velocities [54]. The elastic properties of composites obtained were determined using Equations (2-8) which are used for isotropic composite materials [55].

$$L = \rho V_L^2 \tag{2}$$

$$G = \rho V_S^2 \tag{3}$$

$$K = L - \frac{4}{3}G \tag{4}$$

$$\mu = \frac{L - 2G}{2\left(L - G\right)} \tag{5}$$

$$E = 2G\left(1 + \mu\right) \tag{6}$$

$$H = \frac{\left(1 - 2\mu\right)E}{6\left(1 + \mu\right)} \tag{7}$$

$$Z = \rho V_L \tag{8}$$

Where ρ, V_L, V_s, *L, G, K, E,* μ, *H,* and *Z* are the density, longitudinal wave velocity, shear wave velocity, longitudinal modulus, shear modulus, bulk modulus, Young’s modulus, Poisson’s ratio, ultrasonic micro-hardness, and the characteristic acoustic impedance of composites samples, respectively.

3. RESULTS AND DISCUSSIONS

The ultrasonic testing technique is widely used in the construction industry, as it provides a non-destructive evaluation of the changes in the microstructure of various materials, the locations of damages, and their mechanical properties [56]. Particularly in the construction industry, in the characterization of building materials, the ultrasonic testing technique gives reliable results in the evaluation of mechanical properties without damaging the material compared to destructive methods. For example, many studies have been conducted in the characterization of traditional materials such as wood [57], stone [58], and brick [59] and in the characterization of today's materials such as cement mortars [60], concrete [61] and steel [62] using UT methods. Ultrasonic studies on SSP and polymer material are insufficient in the literature. Therefore, the elastic properties of ER/SSP and PR/SSP composite mortars were determined using the ultrasonic pulse-echo method. The experimental results were given in Tables (4-6) and plotted in Figures (3-10).

3.1. Morphological Results

SEM micrographs of the ER/SSP and PR/SSP composites and the morphology of the composites are shown in Figure 3 and Figure 4. As shown in Figures 3 and 4, SSP particles

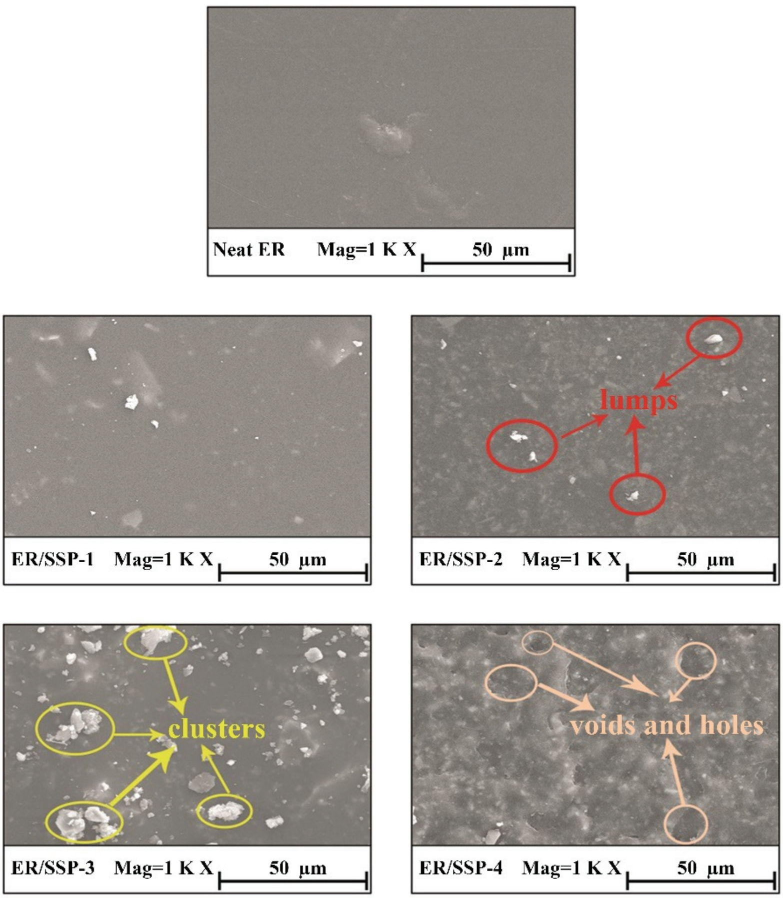

Figure 3 - SEM images of the ER/SSP composites (Magnification of 1.00 K X, 50 µm).

are homogeneously dispersed in the ER and PR matrix. However, lumps occur in composite samples due to the increase in the amount of SSP filler. As stated in studies in the literature, polyester, and epoxy resins have a glassy, brittle, and smooth surface. Therefore, the addition of SSP fillers to the polymer matrix enables the composite to form rough surfaces and the material to have a more durable structure.

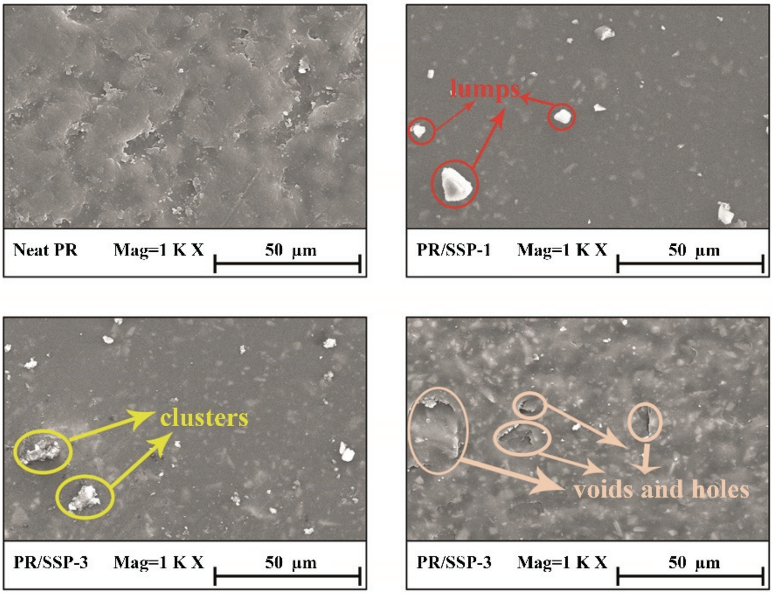

Figure 4 - SEM images of the PR/SSP composites (Magnification of 1.00 K X, 50 µm).

In the ER/SSP-2 and PR/SSP-2 composites, SSP particles agglomerate when interacting with the matrix, these composites do not form a good bond with the polymer matrix. Additionally, in all composites, the number of voids and holes in the material increases due to the increase in the amount of SSP filler.

In the ER/SSP-4 and PR/SSP-3 composites, it is seen that there is a better adhesion in the composites with the homogeneous distribution of the SSP particles with the matrix (See Figures 3 and 4).

3.2. Densities and Ultrasonic Wave Velocities

As can be seen in Table 4 and Figure 5, the density values of the ER/SSP and PR/SSP composites ranged from 1607.12 to 1757.16 kg.m^{-3}, and 1784.24 to 1873.24 kg.m^{-3}, respectively.

The density values measured for obtained the ER/SSP and PR/SSP composite mortars are in good agreement with related literature [36, 38, 63]. For example, Aggelakopoulou et al. [63]

produced restoration mortars to be used on historical walls by mixing metakaolin and hydrated lime with sand, and the density values of the restoration mortars they obtained were measured between 1850–1900 kg.m^{-3}. The density values of all the ER/SSP and PR/SSP composites are found higher than the density values of neat ER and PR, respectively. Additionally, the highest density value among all composite mortars was determined as 1873.24 kg.m^{-3} for the PR/SSP-3 sample. When PR/SSP and ER/SSP composites were compared, it was determined that PR/SSP composites had higher densities than ER/SSP composites. As a result, the density values of ER/SSP and PR/SSP composites increase linearly with the addition of SSP.

Table 4 - Density (ρ) and ultrasonic wave velocities (V_L and V_S) values of materials obtained in this research.

Samples' ID	Composition ratio (wt.%)	ρ (kg.m^{-3})	V_L (m.s^{-1})	V_S (m.s^{-1})
ER	100:0	1109.84±7.59	2360.80±12.04	991.20±7.99
ER/SSP-1	40:60	1607.12±5.22	2692.20±8.73	1381.00±5.48
ER/SSP-2	35:65	1653.72±11.08	2666.20±4.12	1448.00±5.87
ER/SSP-3	30:70	1742.32±23.47	2858.80±64.53	1564.80±8.38
ER/SSP-4	25:75	1757.16±7.98	2907.40±88.20	1610.20±13.42
PR	100:0	1216.32±8.34	2743.40±38.19	1303.60±21.08
PR/SSP-1	40:60	1784.24±5.90	3078.00±9.80	1698.60±3.14
PR/SSP-2	35:65	1843.16±4.87	3012.40±13.16	1815.60±19.62
PR/SSP-3	30:70	1873.24±9.57	3081.60±45.92	1909.20±29.29

According to the results given in Table 4 and Figure 6, both ultrasonic longitudinal and shear wave velocities of the composites are higher than that of ER. The longitudinal wave velocity values of the ER/SSP composites vary between 2666.20 and 2907.40 m.s^{-1} while the shear wave velocity values vary between 1381.00 and 1610.20 m.s^{-1}. The longitudinal ultrasonic wave velocity value of all ER/SSP was increased with the addition of SSP compared to neat ER. However, as can be seen from Table 4 and Figure 6, when the amount of SSP was increased from 60% to 65% by weight, the longitudinal wave velocity in ER/SSP-2 was decreased compared to the longitudinal wave value of ER/SSP-1. The uneven distribution of the micro-filler causes the scattering that increases the transition time of longitudinal waves passing through the composite sample [64]. Thus, the decrease in the velocity of longitudinal ultrasonic waves for the ER/SSP-2 composite can be attributed to this agglomeration. On the other hand, it was determined that the longitudinal wave velocity values of the ER/SSP composite mortars increased about 12.96–23.17 % compared to neat ER.

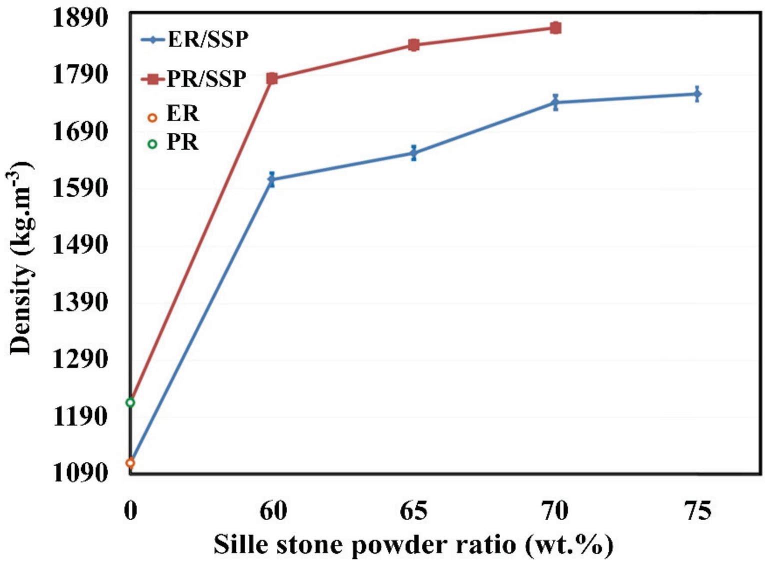

Figure 5 - Variation in densities of the ER/SSP and PR/SSP composites depending on the SSP amount.

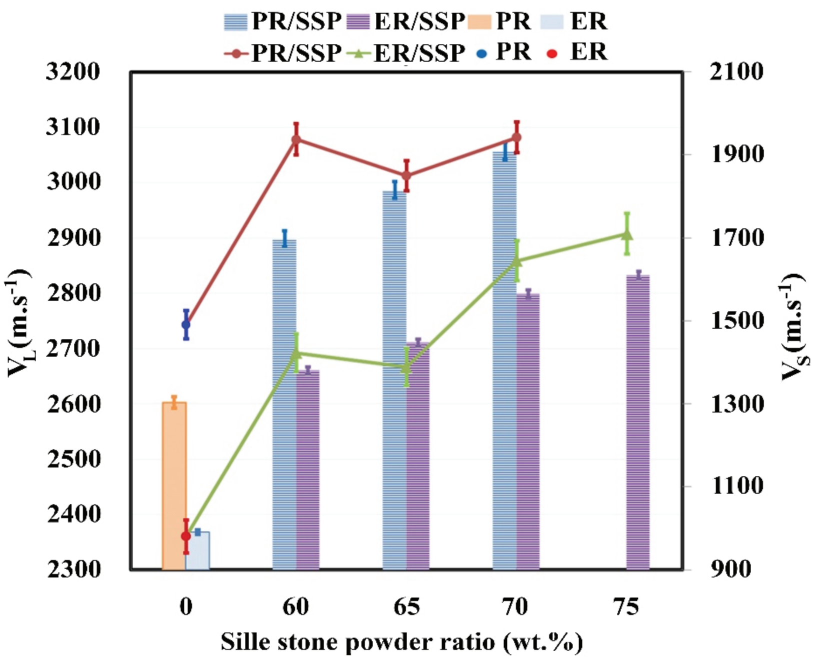

Figure 6 - Variation in ultrasonic wave velocities (V_L and V_S) of the ER/SSP and PR/SSP composites depending on the SSP amount.

Like longitudinal wave velocity values obtained for the ER/SSP composites, a linear increase in shear wave velocity was observed with the increasing amount of SSP in the ER/SSP composites. It was determined that the shear wave velocity values of the ER/SSP composite mortars increased by about 39.35–62.46 % compared to neat ER. This result shows that the contribution of SSP to neat ER increases the shear wave velocity values much more than the longitudinal wave velocity values. Since longitudinal wave velocity values are related to covalent bonds and shear wave velocity values are related to transverse interactions such as Van der Waals, these data show that the contribution of SSP to neat ER creates strong transverse bonds [65]. The higher shear wave values indicate that the outer electron clouds of adjacent atoms of the epoxy resin and SSP are quite close to each other.

Similar behavior of longitudinal ultrasonic wave velocity value of the ER/SSP composites was observed for the PR/SSP composites. The longitudinal wave velocity values of the PR/SSP composites vary between 3012.40 and 3081.60 $m.s^{-1}$, while the shear wave velocity values vary between 1698.60 and 1909.20 $m.s^{-1}$. The longitudinal wave velocity values of all PR/SSP composites were higher than those of neat PR. On the other hand, it was determined that the longitudinal wave velocity values of the PR/SSP composite mortars increased by about 9.81–12.32 % compared to neat PR. Also, a linear increase was observed in the shear wave velocity values of the PR/SSP composites with the increasing amount of SSP (see Table 4; Figure 6). The shear wave velocity values of the PR/SSP composite mortars increased by about 30.31 to 45.51% compared to neat PR. This significant increase in shear wave velocity values of the PR/SSP composites shows the effect of SSP on the shear wave velocity values of the PR/SSP composites.

The longitudinal wave velocity values measured for different kinds of composite mortars have been determined in the range of 800 $m.s^{-1}$ to 3400 $m.s^{-1}$ [34-36, 66, 67]. For example, Gupta and Vyas [68] prepared mortar mixtures (6FS, 6CS-30 and 6CS-40) using granite powder (GP), coarse river sand (CS) and fine river sand (FS) and various properties of obtained mortars were examined both by mechanical tests and ultrasonic velocity measurements. In the research conducted by Gupta and Vyas [68], it was determined that the ultrasonic velocity values of 6CS-30 and 6CS-40 mortars were 13% and 27% higher, respectively, than the control mortar (FS). The reason for this difference was shown to be that granite powders have better packaging properties than the control mortar. Thus, the measured values of ultrasonic longitudinal wave velocity are in good agreement with the related literature. To our knowledge, there is no study that measured the ultrasonic shear wave velocity of composite mortars in the related literature. In this consideration, the determination of the shear wave velocity values of the ER/SSP and PR/SSP composite mortars for the first time is quite important for the literature.

3.3. Elastic Modulus of the ER/SSP and PR/SSP Composites

The elastic properties of ER and PR at different amounts of SSP were calculated using the equations (2, 3, 4, and 6) by the measured density and ultrasonic velocity values. The results obtained are given in Table 5 and Figures 7-8.

The L, G, K, and E values of the neat ER were determined as 6.19 GPa, 1.09 GPa, 4.73 GPa, and 3.04 GPa, respectively. On the other hand, all values of the L, G, K, and E of the ER/SSP composites were determined bigger than those of the neat ER. The L, G, K, and E values of the ER/SSP composites were determined between 11.65 and 14.87 GPa, 3.07 and 4.56 GPa, 7.56 and 8.79 GPa, and 8.10 and 11.64 GPa, respectively. Also, compared to the L, G, K, and E values of the neat ER, the L, G, K, and E values of the ER/SSP composite mortars increased about 88.21-140.23%, 181.65-318.35%, 50.74-85.84 %, and 166.45-282.89 %, respectively.

Table 5 - The elastic properties of materials obtained in this research.

Samples' ID	L(GPa)	G(GPa)	K(GPa)	E(GPa)
ER	06.19±0.06	1.09±0.01	4.73±0.05	3.04±0.04
ER/SSP-1	11.65±0.11	3.07±0.03	7.56±0.10	8.10±0.07
ER/SSP-2	11.76±0.06	3.47±0.02	7.13±0.06	8.95±0.04
ER/SSP-3	14.24±0.63	4.27±0.07	8.56±0.57	10.97±0.23
ER/SSP-4	14.87±0.94	4.56±0.09	8.79±0.88	11.64±0.30
PR	09.16±0.30	2.07±0.08	6.40±0.20	5.60±0.21
PR/SSP-1	16.90±0.09	5.15±0.03	10.04±0.12	13.19±0.06
PR/SSP-2	16.73±0.19	6.08±0.14	8.62±0.19	14.76±0.24
PR/SSP-3	17.79±0.50	6.83±0.19	8.69±0.31	16.23±0.43

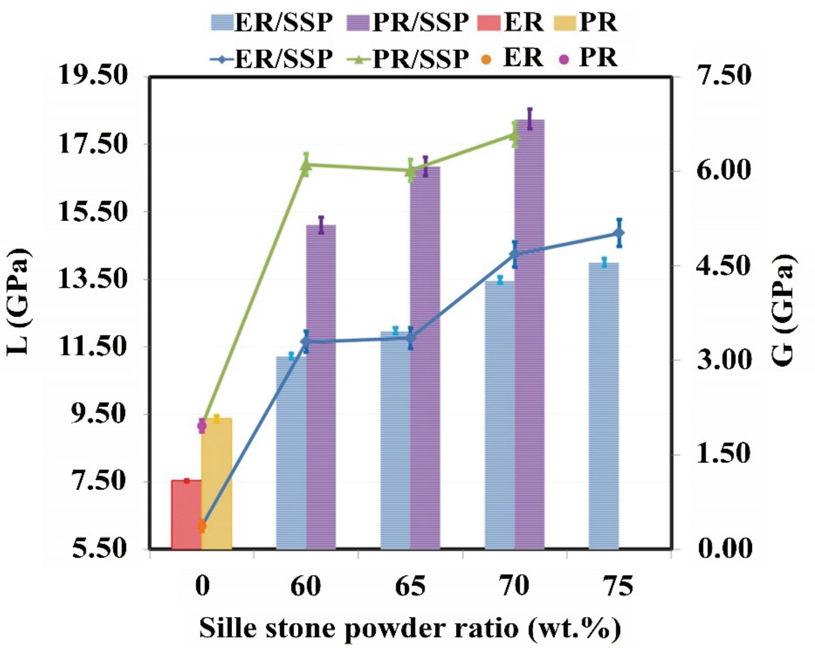

Figure 7 - L, G modulus values of the ER/SSP and PR/SSP composites depending on the SSP amount.

The L, G, K, and E values of the neat PR were determined as 9.16 GPa, 2.07 GPa, 6.40 GPa, and 5.60 GPa, respectively. On the other hand, all values of the L, G, K, and E of the PR/SSP composites were determined bigger than those of the neat PR as well. The L, G, K, and E values of the PR/SSP composites were determined between 16.90 and 17.79 GPa, 5.15 and 6.83 GPa, 8.62 and 10.04 GPa, and 13.19 and 16.23 GPa, respectively. Also, compared to the L, G, K, and E values of the neat PR, the L, G, K, and E values of the PR/SSP composite mortars increased about 84.50 to 94.21%, 148.79 to 229.95%, 34.69 to 56.88%, and 135.54 to189.82%, respectively.

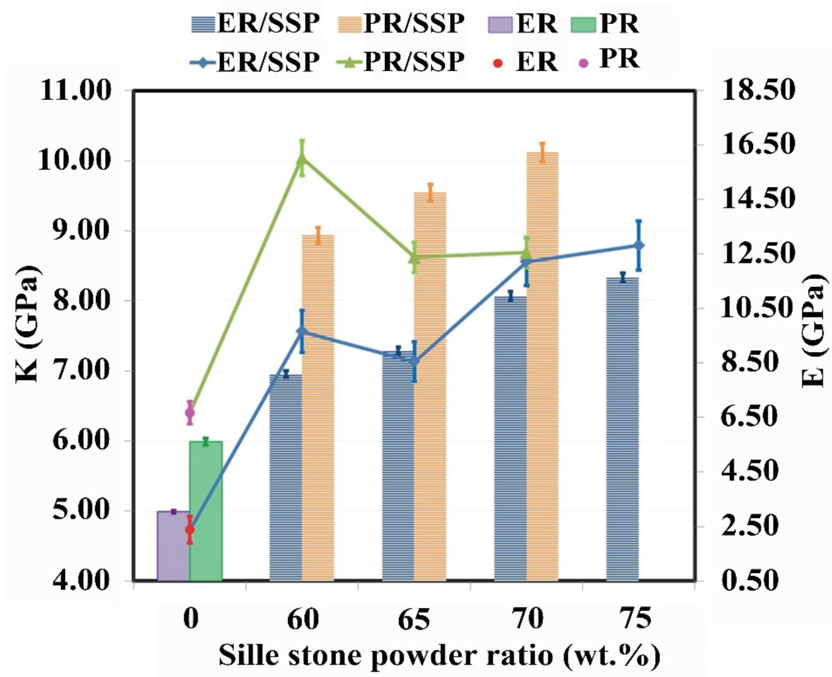

Figure 8 - K, E modulus values of the ER/SSP and PR/SSP composites depending on the SSP amount.

When the literature on restoration mortars is examined, it is seen that there are some studies that measured the ultrasonic longitudinal velocity to determine the durability of produced mortars[34-36, 66, 67]. For instance, Gupta and Vyas [68] determined that the dynamic elasticity modulus of mortars (CS-30 and CS-40) obtained with granite powder and coarse river sand at 1:4 ratios using ultrasonic velocity values and they reported an increase about 29% and 64% in the dynamic elasticity modulus of mortars, respectively, compared to the control mortar (FS). Wu, Liu, Sun and Zhang [69] added peach shell (PS) and apricot shell (AS) to concrete instead of normal weight aggregate (NWA) and sand to obtain environmentally friendly concrete. As a result of the research, it was determined that the elasticity modulus (10.91 GPa to 15.93 GPa) of concretes obtained with PS and AS additives were lower than those of NWA concretes. The low elastic modulus values of the new concretes are attributed to the fact that the hardness of PS and AS is lower than that of NWA. Also, it is seen that the mechanical properties of the mortars were generally realized by destructive methods such as tensile and compression tests as well [36-38, 63, 70-74]. The elastic modulus values of these restoration mortars measured by mechanical methods are between 0.3 and 18.2 GPa. It is seen that the highest elastic modulus values were determined for the cement-based restoration mortars.

The ratio of fillers in the matrix is effective on the mechanical properties of the composites. The durability of the composites increases with the increasing amount of fillers in the matrix at a certain rate. When the ratio of fillers in the matrix exceeds this certain ratio, the mechanical properties of the composites decrease. Because the increase in the amount of filler after a certain ratio causes the interfacial area that weakens the strength of the composite [75]. Also, adding more than a certain amount of filler to the matrix of a composite decreases the mechanical properties of composites due to agglomeration in the composite [76, 77]. On the other hand, studies show that as the fineness of the aggregate increases, the bond between brick and mortar weakens [78], and therefore the mortar prepared with coarse sand will have greater tensile strength than the mortar prepared with fine sand. It has been reported that the tensile bond strength between brick and mortar decreases as fineness of aggregate increases [79].

3.4. Poisson's Ratio, Ultrasonic Micro-Hardness, and Acoustic Impedance

In this part of the research, Poisson's ratio (μ), ultrasonic micro-hardness (H), and acoustic impedance (Z) values of the neat ER, ER/SSP, neat PR, and PR/SSP composites were calculated using Equations (5, 7, and 8). The results obtained are given in Table 6, and Figures 9-10. As seen in Table 6 and Figure 9, the Poisson's ratio values of the neat ER and PR were measured as 0.393 and 0.354, respectively. Additionally, the Poisson ratio values of the ER/SSP and PR/SSP composites were obtained from 0.278 to 0.321, and 0.188 to 0.281, respectively.

Table 6 - Poisson's ratio (μ), ultrasonic micro-hardness (H), and acoustic impedance (Z) values of materials obtained in this research.

Samples' ID	μ	H (GPa)	Z (MRayl)
ER	0.393±0.001	0.15±0.00	2.62±0.02
ER/SSP-1	0.321±0.003	0.64±0.01	4.33±0.03
ER/SSP-2	0.291±0.002	0.81±0.01	4.41±0.03
ER/SSP-3	0.286±0.011	1.01±0.03	4.98±0.12
ER/SSP-4	0.278±0.017	1.10±0.06	5.11±0.16
PR	0.354±0.002	0.37±0.02	3.34±0.06
PR/SSP-1	0.281±0.003	1.23±0.02	5.49±0.02
PR/SSP-2	0.215±0.009	1.71±0.06	5.55±0.04
PR/SSP-3	0.188±0.007	2.00±0.06	5.77±0.08

As it is seen from these results, Poisson's ratio values of the ER/SSP and PR/SSP composites are smaller than those of the neat ER and PR. It was determined that the Poisson's ratios of the ER/SSP composites obtained because of the incorporation of SSP into the neat ER at increasing rates, were lower than those of the neat ER by about 18.32% to 29.26%. A similar decrease of about 20.62% to 46.89% in Poisson's ratio of the neat PR was determined by adding increasing amounts of SSP to neat PR. Since the smaller Poisson's ratio values indicate the enhancement in the mechanical properties of materials, it can be stated that all composite mortars have better mechanical properties than the neat ER and PR resins.

According to the data given in Table 6 and Figure 9, a linear increase was determined in the ultrasonic micro-hardness values of the ER/SSP and PR/SSP composites obtained by increasing the amount of SSP in neat ER and PR resins compared to the micro-hardness value of the neat ER, and PR resins. The ultrasonic micro-hardness values of the ER/SSP and PR/SSP composites were measured between 0.64 GPa to 1.10 GPa and 1.23 GPa to 2.00 GPa, respectively. On the other hand, compared to the H values of the neat ER and PR matrices, an increase of 326.67 % to 633.33% was observed in the H values of the ER/SSP composite mortars while an increase of 232.43% to 440.54% was observed in the H values of the PR/SSP composite mortars. These results are like the results of ultrasonic wave velocities and the elastic properties of ER/SSP and PR/SSP composites. When the ultrasonic micro-hardness values of the obtained ER/SSP and PR/SSP composites are compared, it is seen that the bonds formed because of the interaction of the SSP filler with the neat PR matrix are stronger than those of the bonds formed due to the interaction between the SSP filler and the neat ER. Because the increase in the ultrasonic micro-hardness values in the composite is due to the strong atomic or molecular bonds between the matrix and the fillers.

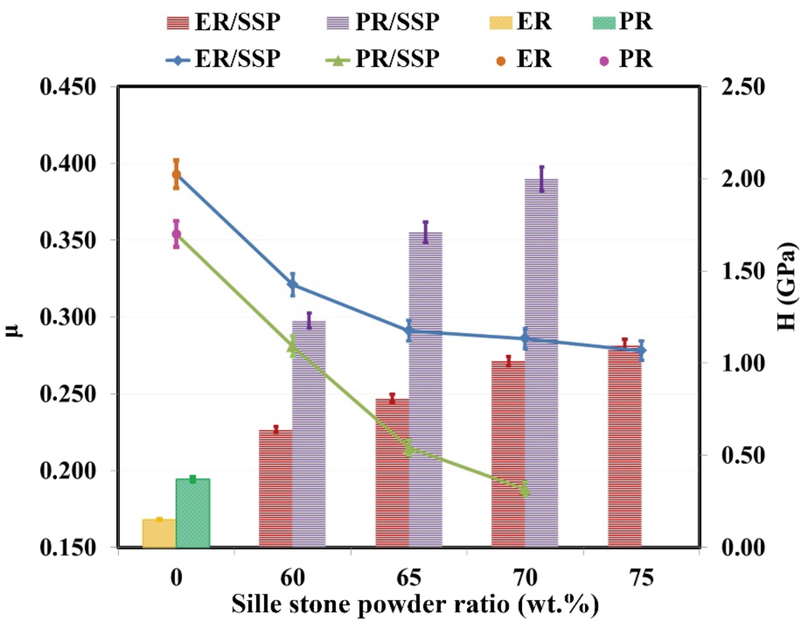

Figure 9 - Change in ER and PR different amounts of SSP Poisson's ratio (μ), and ultrasonic micro-hardness (H).

On the other hand, the acoustic impedance (Z) values of the neat ER and PR were measured as 2.62 MRayl and 3.34 MRayl, respectively. The acoustic impedance (Z) values of the ER/SSP and PR/SSP composites were determined in a range of 4.33 MRayl to 5.11 MRayl and 5.49 MRayl to 5.77 MRayl, respectively (see Table 6; Figure 10). Thus, a significant increase was observed for Z values of neat ER, and PR. Because compared to neat ER, and PR matrices, an increase was seen in a range of 65.27% to 95.04% and 64.37% to 72.75% was observed in the Z values of the ER/SSP, and PR/SSP composites, respectively. As seen in Figure 10, a linear increase was observed in Z values of both neat ER and PR matrices by increasing the amount of SSP. As with all other elastic properties, the Z values of the neat PR matrix, and PR/SSP composites were determined higher than those of the neat ER matrix, and ER/SSP composites. Therefore, it can be stated that adding the SSP filler to the PR matrix provides composites that have higher Z values compared to the ER/SSP composites obtained by adding the SSP filler to the ER matrix.

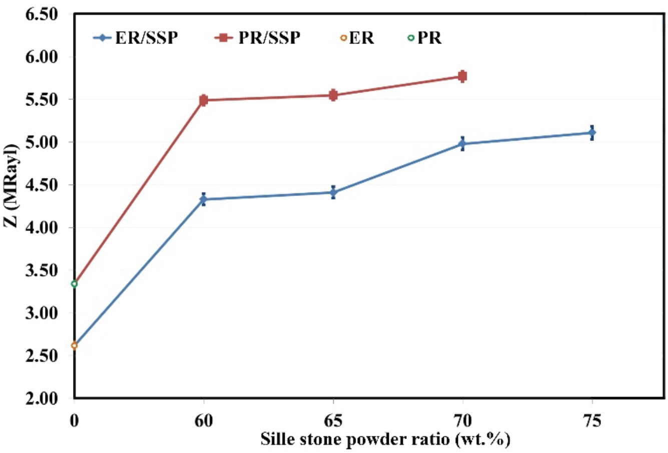

Figure 10 - Change in ER and PR different amounts of SSP acoustic impedance (Z) values.

Although material density is an important factor affecting ultrasonic wave velocity [80, 81], it is not the only factor affecting ultrasonic wave velocity in composite materials. Because moisture content, porosity degree of the material and discontinuities within the material can also affect the materials properties such as ultrasonic wave velocity in composites [70, 82-85]. Ultrasonic wave velocity is also directly related to the density and elastic properties of the material [86]. There are various reasons why the density of materials affects the ultrasonic wave velocity in composites (Elastic modulus, acoustic impedance, packing of particles, homogeneity). For example, ultrasonic wave velocity is frequently directly proportional to the materials' Young's modulus and hardness, both of which depend on the material density [87]. Therefore, if the Young's modulus and hardness value of a material are high, the ultrasonic wave velocity in that material may also be high. On the other hand, acoustic impedance, which is a measure of the ability to transfer ultrasonic wave energy from one medium to another, also affects the ultrasonic wave velocity. Because acoustic impedance is a physical material property that depends on both density and ultrasonic velocity, and a high acoustic impedance causes an increase in ultrasonic velocity. The tight packing of particles such as sand, cement and aggregate added into the matrix in mortar may cause fewer voids in the resulting composite, which causes an increase in both the density of the resulting composite and the ultrasonic wave velocity. Because there are fewer gaps in the resulting composite, it causes ultrasonic waves to travel in the medium in a shorter time.

Since higher density can be achieved in materials with more homogeneous distribution, ultrasonic wave velocity may also be higher in a more homogeneous environment. Achieving the desired homogeneity in composites depends on the composite components and the applications in the production process. Proper mixing time and intensity are very important to ensure homogeneity in mechanical mixing. It is possible to ensure homogeneous distribution of the components by breaking up the lumps by applying appropriate mechanical force during mixing. Sometimes, homogeneous distribution can be achieved by improving the compatibility between these fillings and the matrix material by treating the surfaces of the filling materials with coupling agents or chemical treatments. In some cases, the homogeneity of the matrix and components of the mixture can be supported by the addition of surfactants or dispersants. Making composite mixtures at appropriate temperature and pressure, reducing the size of the particles added to the composite, and adding them to the mixture in the appropriate order can also help in the homogeneous distribution of the composite components. On the other hand, one of the best ways to improve the homogeneity of composite mortars at the production process is to use ultrasonic homogenizer which uses ultrasonic waves.

3.5. Cost Analysis

A cost analysis was conducted to evaluate the feasibility of using the composite mortars produced in this study in the restoration of historical buildings. In the cost analysis, prices of epoxy and polyester resin materials are based on data available on commercial websites [49, 50, 88]. In calculating the cost analysis of these materials, 20% goods and services tax was added to the market price. Additionally, for imported goods, a shipping fee of $65 per ton has been added. Average unit prices of various restoration mortars are given in Table 7.

Table 7 - Average unit prices of various restoration mortars.

Composite mortars abbreviations	Unit price ($ per ton)
Pozzolanic lime-based mortar	628
Natural hydraulic lime mortar	601
Khorasan mortar	732
Gypsum mortar	147
Composite mortar obtained in this research	240–600

According to the data in Table 7, it is seen that the composite mortars produced within the scope of this study have lower costs than other traditional restoration mortars, except gypsum mortar. On the other hand, composite mortars are cheaper than pozzolanic lime mortar, natural lime mortar, and Khorasan mortar by approximately 4.46% to 61.78%, 0.16% to 60.07% and 18.03% to 67.21% respectively. However, composite mortars obtained in this research are approximately 63.27% to 308.16% more expensive than gypsum mortar.

4. CONCLUSIONS

In this research, epoxy, and polyester matrix-based repair mortars were developed by utilizing the recycling of Sille Stone Powder (SSP) to be used in the restoration of old historical buildings such as madrasas, mosques, palaces, libraries, fountains, and caravanserais and in new generation construction buildings. The physical and mechanical properties of these new polymer-based mortars were determined non-destructively by ultrasonic method, while their morphology was analyzed by SEM. SEM images revealed that SSP showed a more homogeneous distribution in PR resin compared to ER. The findings of the research showed that except the values of Poisson's ratio, the values of density, ultrasonic longitudinal wave velocity, ultrasonic shear wave velocity, ultrasonic micro-hardness, acoustic impedance, and elastic modulus (L, G, K, E) of the ER/SSP, and PR/SSP composites are higher than those of the values of neat ER, and PR. In addition, it was determined that the most suitable combination ratio between ER and SSP in the ER/SSP composite mortars was 25:75, and the most appropriate combination ratio between PR and SSP in the PR/SSP composite mortars was 30:70. One of the most important results of the research is that the costs of the repair mortars obtained are more affordable than traditional repair mortars. In conclusion, this research showed that the ultrasonic testing method can be used as a beneficial method in the characterization of composite mortars and the evaluation of their elastic properties. For this reason, it may be recommended to conduct similar research using SSP and other environmentally polluting wastes such as SSP with other polymer matrices such as polystyrene and polyurethane, as in ER and PR, and characterizing these new repair mortars with non-destructive ultrasonic methods.

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