EFFECT OF VARIATION OF EGG-SHELL PARTICULATE REINFORCEMENT ON ULTRASONIC PULSE VELOCITY, ATTENUATION, AND HARDNESS PREDICTION IN CERAMIC-METAL COMPOSITES

by

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In this study, ceramic-metal composite material reinforced with additive prepared from waste egg-shell was produced. Waste egg-shells were cleaned, dried, ground and then sieved. The effects of egg-shell reinforcement ratio (0%, 1.66%, 3.34%, 5%, and 6.66 % by volume) and sintering temperature (1400 °C) applications on Fe-B_4C ceramic-metal powders were tested. For this purpose, tests such as ultrasonic test, hardness and density were applied to the composite material, and finally, microstructural analysis was performed on the composites by SEM application. The changes in the crystal structure of the egg-shell after sintering were revealed by diffractograms analysis. It was observed that the hardness, ultrasonic longitudinal and transverse wave velocities also increased with the increase in the reinforcement ratios of the egg-shell. Considering the 6.66% egg-shell addition to the ceramic-metal composite mixture, in the light of the numerical data obtained, 5091 m/s longitudinal wave velocity, 2809 m/s transverse wave velocity, 204.12 Hv hardness value, 0.315 dB per mm longitudinal attenuation and 0.214 dB per mm transverse attenuation values with gave the best physical and mechanical properties. According to the test results obtained, it was determined that it would be appropriate to use the egg-shell in the production of composite materials and to characterize it with the ultrasonic test method.

Key words: *waste egg-shell, ceramic-metal composite, ultrasonic test, recycles*

Introduction

In order to ensure sustainability in development, it is necessary to recycle food waste, contribute to the prevention of environmental pollution, and also produce new materials in a way that can strengthen the country economy [1-3] While some of the food wastes generated in the food industry and/or agricultural production are destroyed, some are used in the production of new products with lower economic value by using different technologies. Studies for the production of new materials and new products are carried out [4-6]. Egg-shell is a biomaterial containing 95% by weight of calcium carbonate and 5% by weight of organic material in the form of calcite. It is very rich in calcium carbonate. These

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features show that egg-shell can be used in the production of new materials with appropriate conversion processes and different mixture recipes and it is worth researching [7-10]. Continuously developing and changing technologies need to continuously improve the properties expected from materials to be used in industrial applications such as high strength, low cost, toughness and low weight ratio to meet the optimum standard [11-13]. Therefore, the search for new advanced materials always remains a matter of interest. Many researchers have successfully proven in their research that metal and ceramic matrix composite meets the expected standard and found that it is a promising material class [14-19]. The difficulties experienced in shaping ceramics with traditional powder metallurgy methods have led to the development of composite materials and the emergence of new processing approaches. Since ceramics are brittle, hard, resistant to heat and corrosion, difficult to compress, and have a high melting temperature in industrial terms, metal materials are used to ensure easy machinability. For this purpose, waste egg-shell reinforced composite materials have been developed [20-30]. In this study, ceramic-metal composite material reinforced with additive prepared from waste egg-shell was produced. In this context, it will help the researches on the production of new materials with lighter and improved mechanical properties by evaluating the wastes. In the study, ultrasonic testing technique, which is one of the most preferred non-destructive testing techniques, was used to characterize material properties such as residues, stresses, fracture strength, elastic modulus, error size and directions, and to determine the safe operation of the structure. The effects of ultrasonic vibration on the microstructure and mechanical properties of the material were investigated [31-34]. The non-destructive testing method is a versatile technique that not only reveals the defects in the material, but also gives information about the quality of the material. The ultrasonic wave moving in the solid material is affected by the density and elastic properties of that material. The quality of a material sometimes changes depending on these elastic properties, hence the ultrasonic velocity. Ultrasonic inspection is used to determine the location of the defects and the fault zone in the material, as well as to evaluate these defects by acoustically transferring them to the screen, to measure the thickness of the parts, and to easily measure ultrasonic test parameters such as transit time, attenuation, scattering and frequency. It can also be used to measure the basic mechanical, structural and compositional properties of solids and liquids with high-frequency sound waves [35-42].

The aim of the study is to investigate the ultrasonic, mechanical and metallographic effects of egg-shell powders added at different rates in the production of ceramic-metal composites and to characterize the produced samples.

Materials and method

Samples and production

The most suitable samples for the research were prepared by powder metallurgy method by using different ratios of egg-shell powder, which is used as waste in the production of ceramic-metal composite material. The Fe and B_4C powders with 99% purity and a particle size of less than approximately 70 μm were obtained from Sigma Aldrich. The volumetric composition of the samples and the codes given to the samples produced are given in tab. 1. After weighing the powders, composition mixture was mixed in a mixer rotating at 20 rpm for 24 hours to ensure that the composition was homogeneous. Homogeneously mixed powders were shaped circularly in a uniaxial hydraulic press in 30 g samples under a pressure of 407.9 kg/cm^2 in a cylindrical mold and sintered for two hours at 1400 °C in an argon atmosphere in a conventional sintering furnace. After the sintering process, the prepared samples were left to cool naturally in the furnace under an argon atmosphere. The microhardness values of the samples were measured with a Mettest-HT (Vickers) brand microhardness tester. An average hardness value was determined by taking measurements from 12 different regions of each sample. In order to evaluate the microstructural analysis of the composites reinforced with egg-shell powder, Fe, and B_4C powders with different additive ratios, the images of the material surfaces were taken using the LEO 1430 VP model SEM.

Table 1. Composition of samples

Codes	Composition of samples	
	Metal-ceramic powder	Egg-shelter powder
Sample	98.34% Fe-1.66% B_4C	–
Egg-shell I	96.68% Fe-1.66% B_4C	1.66%
Egg-shell II	95% Fe-1.66% B_4C	3.34%
Egg-shell III	93.34% Fe-1.66% B_4C	5%
Egg-shell IV	91.68 % Fe-1.66% B_4C	6.66%

The ultrasonic pulse velocity method

Before taking the ultrasonic measurements, the geometry and dimensions of the samples were prepared with considering that there is no side wall effect. In material characterization studies using ultrasonic, the determination of the dimensions of the samples, the determination of the sample surface width, the sample thickness, and the flat and smoothness of the sample surface are important factors to be considered. The thickness of the egg-shell powder added composite samples was measured with a micrometer at the same signal reception points with an accuracy of ±0.01 mm [43, 44]. Ultrasonic testing was performed using a commercially available system consisting of an ultrasonic flaw detector (Sonatest Sitescan 150 seconds), transducers, and a computer. Two different converters were used to obtain signals in pulse-echo configuration.

A 2 MHz and 4 MHz transceiver probe is used for ultrasonic longitudinal and transverse wave velocity measurements. A 4 MHz (Sonatest SLH4-10, T/R) transducer with a radius of 10 mm was used to generate longitudinal waves and a 4 MHz (GE Inspection Technologies MB 4Y, T/R) single-crystal transducer with a radius of 15 mm was used to generate transverse waves. Ultrasonic measurements were performed using the direct contact method between the transducer and the samples in pulse-echo configuration. Sonatest sonagel-W was used as binder to ensure effective contact between transducers and sample surfaces. The basic principle of ultrasonic testing in non-destructive testing methods is based on the propagation of elastic waves of high frequency (0.1-20 MHz) produced by the probe in the test material environment and their reflection back to the probe after hitting a discontinuity. Among the ultrasonic inspection methods, it is the pulse-echo technique that is most used in practical life. The waves detected by the probe are converted into electrical signals and appear on the cathode ray tube screen as echoes that herald the material internal structure. The positions and amplitudes of the observed echoes on the screen provide information about the location and dimensions of the error. Velocity measurements were obtained with the reflection peaks coming from the front and back surfaces in the A-Scan image of the ultrasound wave sent to the sample by the transducer. The time taken for these peaks, which are obtained by the waves coming and going to the other side of the material, was determined on the oscilloscope screen in terms of Δt [μs]. This process was repeated five times for each material surface, and the average transition time was determined. After determining the thickness of the materials and the transit times of the longitudinal and transverse waves through the materials, they were substituted in eq. (1) and ultrasonic longitudinal wave velocities and transverse wave velocities were calculated in [ms^{-1}].

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

During ultrasonic examination, the structural relations of sound waves traveling through the material with their scattering are handled. The factors affecting the scattering of ultrasonic waves in the material are the wavelength of the sound waves used, the size, shape, direction, anisotropy and compound structure of the particles in the structure. Also, ultrasonic attenuation, which is the sum of absorption and scattering, mainly depends on the scattering and damping capacity from the grain boundary in the material. As the ultrasonic wave passes through a solid, its amplitudes decrease due to the interaction with the particles in the medium. This reduction is expressed as attenuation [45-47].

Using the ratio of the amplitudes of A_1 and A_2, the following formula is used to calculate the frequency-dependent attenuation coefficient:

$$\alpha = \frac{1}{d} 20 \log \frac{A_1}{A_2} \tag{2}$$

where d is the thickness of the sample, A_1 and A_2 are the amplitude of the ultrasonic wave, respectively. Its logarithmic unit is expressed in [Neper] or [dB].

Experimental results and discussion

Metallographic analysis

The SEM micrograph of the sample produced without using egg-shell in the composition of fig. 1(a) is given. In addition to homogeneous distribution in the composition, it is seen that the porosity is quite high. The hardness values of the sample also support the porosity. In the composition of fig. 1(b), the porosity was slightly decreased and the grains grew. Changes in surface morphology were observed in the composition of figs. 1(c) and 1(d). With the increase in the composition of egg-shell powder, grain coarsening increased and porosity decreased and grain boundaries became clear. In fig. 1(e) (91.68%Fe-1.66%B_4C-6.66% egg-shell) composition, the porosity was greatly reduced and the surface morphology acicular structure image was obtained. The highest hardness measurements were obtained in 91.68% Fe-1.66%B_4C-6.66% egg-shell composition.

Lineal EDX analysis was applied to the first produced (98.34% Fe-1.66%B_4C) sample in fig. 2. After the analysis, Fe, Al, Si, and Mg peaks were obtained. The low rate of Al, Si, and Mg peaks is due to the fact that the purity level of the Fe powder used is not 100%. The absence of the Ca peak is due to the use of egg-shells in the composition.

In fig. 3, (91.68% Fe-1.66%B_4C-6.66% egg-shell) lineal EDX analysis was applied to the composition sample. The Fe, Al, Mg, and Ca peaks were obtained after the analysis. The low rate of Al and Mg peaks is due to the fact that the purity level of the Fe powder used is not 100%. Since approximately 93-97% of the egg-shell consists of $Ca(CO_3)$, it is rich in calcium. The presence of the Ca peak in the EDX analysis is due to egg-shell powders.

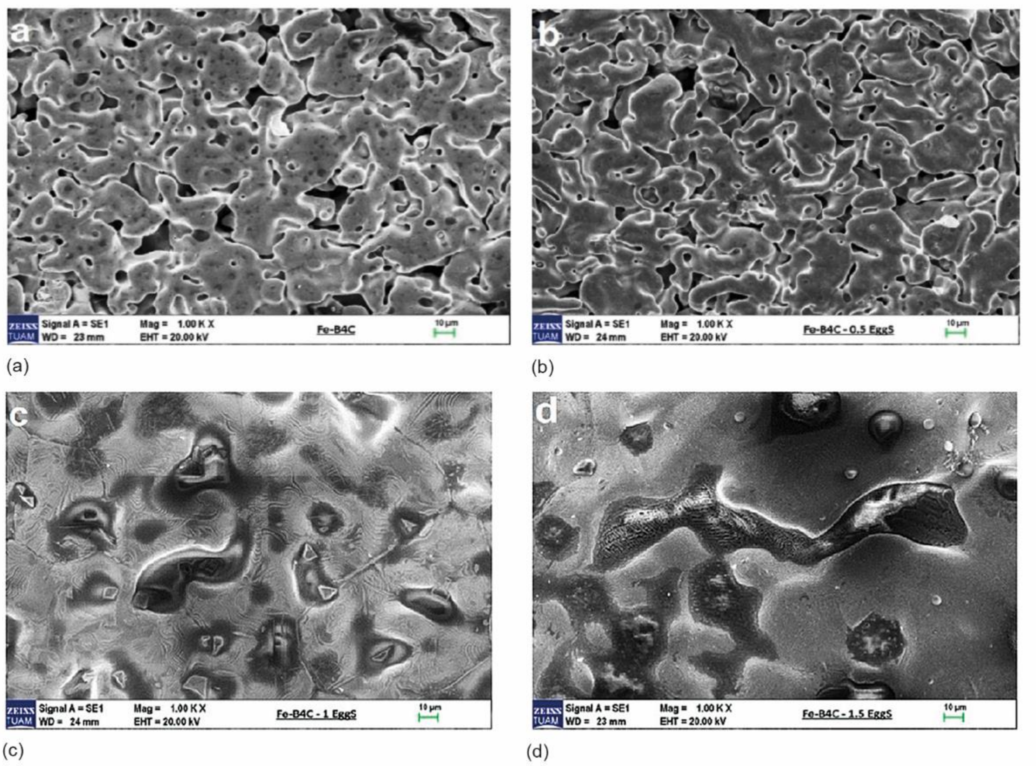

Figure 1. The SEM micrographs; (a) 98.34% Fe-1.66% B_4C, (b) 96.68% Fe-1.66%B_4C-1.66% egg-shell, (c) 95% Fe-1.66%B_4C-3.34% egg-shell, (d) 93.34% Fe-1.66%B_4C-5% egg-shell, and (e) 91.68% Fe-1.66%B_4C-6.66% egg-shell

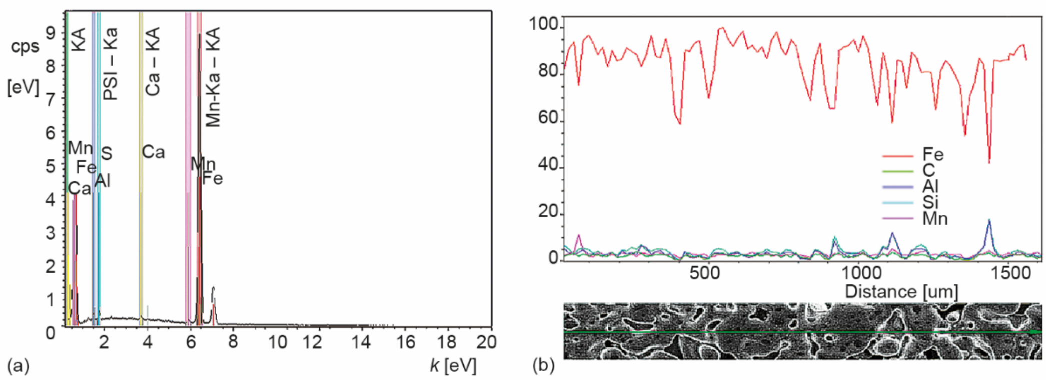

Figure 2. The EDX analysis of 98.34% Fe-1.66%B_4C specimens

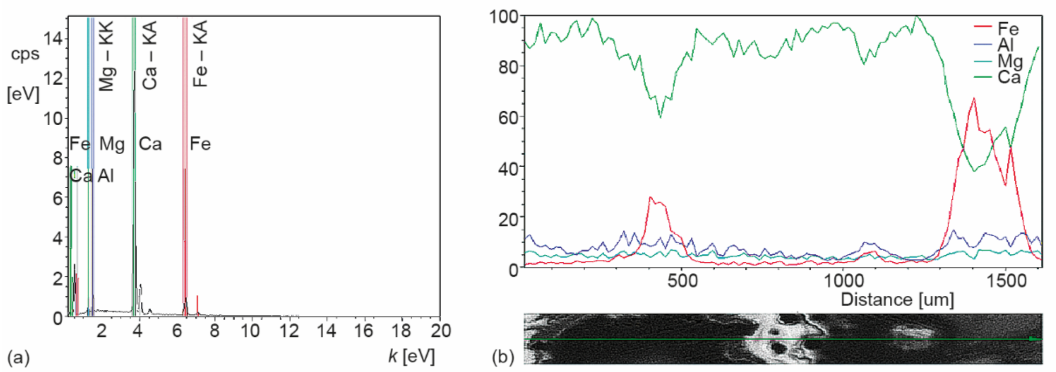

Figure 3. The EDX analysis of 96.68% Fe-1.66%B_4C-6.66% egg-shell specimens

Ultrasonic measurements

The aim of the study is to examine the effects of sound wave vibration sent by ultrasonic method in the production of ceramic-metal composites of ground waste egg-shell on the microstructure and mechanical properties of the material and to produce new samples that will contribute to the economy both in terms of recycling waste and reducing the use of raw materials. Ultrasonic longitudinal and transverse wave velocities, attenuation values, hardness and density measurement results of Fe-B_4C composite samples with sintered egg-shell reinforcement in different compositions are shown in tab. 2.

Table 2. Ultrasonic and mechanical properties of ceramic-metal composites

Composite samples	Hardness [HV]	Longitudinal wave velocity [ms^{-1}]	Shear wave velocity [ms^{-1}]	Longitudinal attenuation [$dBmm^{-1}$]	Shear attenuation [$dBmm^{-1}$]	Density [gcm^{-3}]
Sample	178.25	4261±43.1	1920 ±28.3	0.589	0.352	5.28
Egg-shell I	181.54	4334±4.2	2020 ±42.4	0.449	0.304	5.35
Egg-shell II	194.58	4554±32.5	2315 ±53.7	0.423	0.284	6.34
Egg-shell III	198.71	4945±36.8	2547 ±7.1	0.326	0.245	6.03
Egg-shell IV	204.12	5091±49.5	2809 ±28.9	0.315	0.214	5.93

In the study, ultrasonic pulse-echo technique was used effectively for the characterization of Fe-B_4C% egg-shell composite properties. In this study, the velocity of sound waves passing through the composite material is determined. The arrival time of the ultrasonic waves from the receiver at the starting point to the receiver on the other side is measured, and it is calculated with the help of the distance that the wave passes. As a result of the tests, it was observed that the ultrasonic longitudinal and transverse velocity values of the egg-shell powder increased with increasing volume ratios. This is an indication that there is no loss of sound waves as they move through the structure. This indicates that denser and coarser grained structures are formed. As the amount of additives in the composite material increases and the voids decrease, the arrival time of the sound wave at the receiving point is shortened and the ultrasonic wave transmission speed increases. When fig. 4(a) is examined, ultrasonic longitudinal and transverse velocity values are respectively. It is observed that while it was 4261 m/s and 1920 m/s in sample with no additive egg-shell powder, it increased to 5091 m/s and 2809 m/s in egg-shell IV with 6.66% egg-shell powder additive. By means of a typical ultrasonic waveform obtained for composite samples, the ultrasonic attenuation coefficient was determined as the ratio of the amplitudes of two ultrasound beams reflected from the peaks corresponding to the posterior wall reflection of the sample as a function of the distance passing through the imaging medium. To calculate the longitudinal wave velocity and attenuation in the sample, the position in the time axis and the corresponding amplitude are noted for the first two backwall reflections. In fig. 4(b), as the additive ratio increases, the porosity in the composite material decreases and the absorption of the sound wave in the structure decreases. The fact that the egg content of the prepared ceramic-metal composite is gradually higher and the sintering temperature also causes a denser structure with a lower pore content, which explains the reason for the decrease in attenuation values.

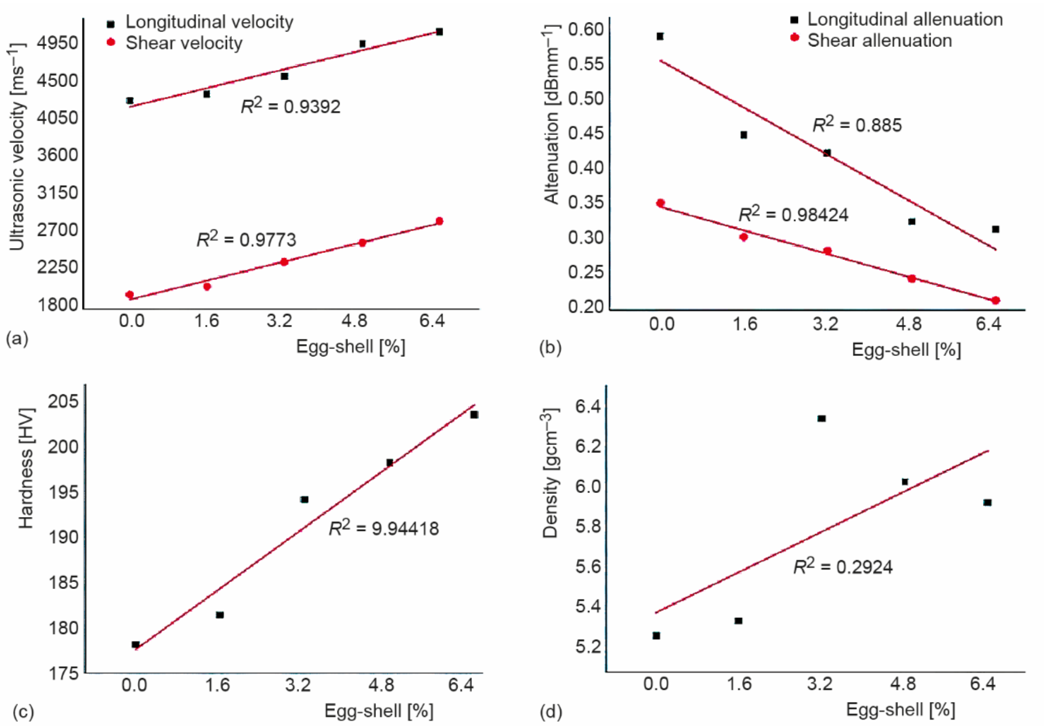

Figure 4. Graphs showing the atomic effect of the addition of egg-shell powder on; (a) ultrasonic velocity, (b) ultrasonic attenuation, (c) hardness, and (d) density

The hardness values measured in the composite samples prepared in fig. 4(c) are given by measuring from 12 different points and taking the average. In order for the hardness values to be sensitive and reliable, the measurements were taken from 12 different points in the composite samples prepared and the average was given. The lowest hardness value of 178.25 HV was measured in the composite sample without egg-shell powder. By increasing the egg-shell powder atomically up to 6.66%, different ceramic-metal composite samples with different compositions were obtained. The highest hardness value was measured in the sample belonging to the egg-shell reinforced (egg-shell IV) composite composition with 204.12 HV.

When the reaction mechanism in the literature is evaluated, as seen in the hardness, ultrasonic wave velocities, attenuation and density graphs, fig. 4, as the sintering temperature increased, the reactivity of the egg-shell in the ceramic-metal composite increased and a stronger interface bond was achieved. As a result, it showed better mechanical properties [48, 49]. Composite materials have become more durable with the increase of the amount of calcium carbonate ($CaCO_3$) in volume ratio. The $CaCO_3$ is known as a compound that gives strength to composite materials structurally. Since $CaCO_3$ adds rigidity and strength to the structure used, the reason for the increase in hardness with increasing volume ratios is the increase in the percentage of $CaCO_3$ [50]. Therefore, hardness values of composite materials increased with increasing volume ratios, as it increases strength and durability. In the production of composite samples, the graph showing the relationship between the atomic ratios of the egg-shell powders additive between 0% and 6.66% and the density is given in fig. 4(d). When egg-shell powder was not added to the ceramic-metal composite structure (sample), the density was calculated as 5.28 g/cm^3. The density value was calculated as 5.93 g/cm^3 when egg-shell powder was supplemented atomically up to 6.66% (egg-shell IV). In SEM images, it was seen that the addition of 3.34% atomic egg-shell powder provided better sintering. While the density values increase up to egg-shell II, there is a slight decrease in the density values in egg-shell III and egg-shell IV. However, there is an existing increase over the composite sample without egg-shell additives. As a result, the addition of egg-shell powder positively affected the sintering. In this study, it was revealed that the egg-shell additive improved the mechanical and ultrasonic properties of composite materials in a positive way.

Conclusion

This study evaluated the usability and strength performance of ground waste egg-shell as a building material. Egg-shell powder reinforced ceramic-metal composite material was produced. According to the results obtained, ultrasonic wave velocities, hardness and density values increased with increasing volume ratios of waste egg-shell powder additive in the produced composite samples, while naturally a decrease was observed in ultrasonic attenuation values. In our country, such studies have become increasingly important as they will contribute to the economy in terms of recycling wastes and environmental pollution risk, as well as reducing the use of raw materials for different purposes, and will pave the way for their use in different fields of industry.

References

[1] Vinod, A., *et al*., Renewable and Sustainable Biobased Materials: An Assessment on Biofibers, Biofilms, Biopolymers and Biocomposites, *J. Clean. Prod., 258* (2020), June, 120978
[2] Das, O., *et al*., Natural and Industrial Wastes for Sustainable and Renewable Polymer Composites, *Renew. Sust. Energ. Rev., 158* (2022), Apr., 112054
[3] Palencia, M., *et al*., Polymer-Metal Oxide Composites from Renewable Resources for Agricultural and Environmental Applications, in: *Renewable Polymers and Polymer-Metal Oxide Composites*: *Synthesis, Properties, and Applications* (Ed. Sajjad Haider and Adnan Haider), Elsavier, Amsterdam, The Netherlands, 2022, pp. 341-370
[4] Igwe, I. O., Onuegbu, G. C., Studies on Properties of Eggshell and Fish Bone Powder Filled Polypropylene, *Am. J. Polym. Sci., 2* (2012), 4, pp. 56-61
[5] Yıldız, A., Mechanical Properties of Mortar Using Eggshell, *Karaelmas Sci. Eng. J., 8* (2018), 2, pp. 570-574
[6] Kuru, D., *et al*., Effect of Chicken Feather and Boron Compounds As Filler on Mechanical and Flame Retardancy Properties of Polymer Composite Materials, *Waste Manag. Res., 36* (2018), 11, pp. 1029-1036

[7] Oliveira, D. A., *et al*., A Literature Review on Adding Value to Solid Residues: *Egg Shells, J. Clean. Prod., 46*, (2013), May, pp. 42-47

[8] Lee, M., *et al*., Reusing Shell Waste as a Soil Conditioner Alternative? A Comparative Study of Egg-shell and Oyster Shell Using A Life Cycle Assessment Approach, *J. Clean. Prod., 265* (2020), Aug., 121845

[9] Nys, Y., Gautron, J., Structure and Formation of the Eggshell, in: *Bioactive Egg Compounds*, (eds. R. Huopalahti, R. López-Fandino, M. Anton, R. Schade), Springer, Berlin, 2007, pp. 99-113

[10] Mukarami, F. S., *et al*., Physicochemical Study of $CaCO_3$ from *Egg Shells, J. Food Sci. Technol., 27,* (2007), 3, pp. 658-662

[11] Srivastava, A. K., *et al*., A Review on the Intensification of Metal Matrix Composites and Its Non-conventional Machining, *Sci. Eng. Compos. Mater., 25* (2016), 2, pp. 213-228

[12] Sohag, M. A. S., *et al*., Effect of Ceramic Reinforcement on the Microstructural, Mechanical and Tribological Behavior of Al-Cu Alloy Metal Matrix Composite, *Mater. Today: Proc., 21* (2020), 3, pp. 1407-1411

[13] Rajaram, S., *et al*., Experimental Investigation of Mechanical Properties of AA6063/ZrO_2 Metal Matrix Composites, *Mater. Today: Proc., 74* (2023), 1, pp. 105-109

[14] Yönetken, A., Production and Investigation of Ceramic Metal Composite from Electroless Ni Plated AlN and Al Powders, *Int. J. Innov. Res. Technol. Sci. Eng., 6* (2017), 10, pp. 21-26, ISSN(Online): 2319-8753

[15] Gupta, P. K., Srivastava, R. K., Fabrication of Ceramic Reinforcement Aluminium and Its Alloys Metal Matrix Composite Materials: A Review, *Mater. Today: Proc., 5* (2018), 9 (3), pp. 18761-18775

[16] Kim, E. H., *et al*., Fabrication and Mechanical Properties of Metal Matrix Composite with Homogeneously Dispersed Ceramic Particles, *Ceram. Int., 39* (2013), 6, pp. 6503-6508

[17] Ozer, S., *et al*., Effects of Fusel Oil Use in a Thermal Coated Engine, *Fuel*, *306* (2021), Dec., 121716

[18] Demircioğlu, P., *et al*., Improving Manufacturing Process of Mobile Recorder, in: *Digital Conversion on the Way to Industry 4.0. ISPR 2020. Lecture Notes in Mechanical Engineering*, (Eds. Durakbasa, N. M., Gençyılmaz, M. G.), Springer, Cham., Berlin, Germany, 2021

[19] Tufan, O., *et al*., Design and Development of Drum Granulator, in: *Digital Conversion on the Way to Industry 4.0. ISPR 2020. Lecture Notes in Mechanical Engineering*, (Eds. Durakbasa, N. M., Gençyılmaz, M. G.), Springer, Cham., Berlin, Germany, 2021

[20] Yonetken, A., *et al*., Characterization of Egg-Shell Powder-Doped Ceramic-Metal Composites, *Open Chemistry, 20* (2022), 1, pp. 716-724

[21] Yonetken, A., *et al*., Production and Characterization of Ti-10Cr-3,33Co-3,33 Egg Shelter Composite Materials Using by Powder Metallurgy, *J. Mech. Eng. Res. Dev., 12* (2020), 1, pp. 158-165

[22] Hassan, S. B., Aigbodion, V. S., Effects of Eggshell on the Microstructures and Properties of Al-Cu-Mg/Eggshell Particulate Composites, *J. King Saud Univ. Eng. Sci., 27* (2015), 1, pp. 49-56

[23] Hussein, M. A., *et al*., Processing and in Vitro Corrosion Analysis of Sustainable and Economical Egg-shell Reinforced Mg and Mg-Zr Matrix Composite for Biomedical Applications, *Mater. Today Commun., 32* (2022), Aug., 103944

[24] Aigbodion, V. S., Insights in the Enhancement of Electrical Conductivity and Dielectric Constant of Epoxy-Carbon Nanotubes Decorated with $CaCo_3$-Derived from Waste Eggshell Hybrid Composites, *J. Indian Chem. Soc., 99* (2022), 10, 100736

[25] Demirdal, S., Aydın, F., The Influence of Low-Cost Eggshell on the Wear and Electrochemical Corrosion Behaviour of Novel Pure Mg Matrix Composites, *Mater. Chem. Phys., 277* (2022), Feb., 125520

[26] Yadav, R., *et al*., Eggshell and Rice Husk Ash Utilization As Reinforcement in Development of Composite Material: A Review, *Mater. Today: Proc., 43* (2021), 1, pp. 426-433

[27] Dwivedi, R., *et al*., Parametric Optimization of Process Parameters During the Friction Stir Processing of Al7075/SiC/Waste Eggshell Surface Composite, *Mater. Today: Proc., 47* (2021),13, pp. 3884-3890

[28] Sharma, S., Dwivedi, S. P., Effects of Waste Eggshells and SiC Addition on Specific Strength and Thermal Expansion of Hybrid Green Metal Matrix Composite, *J. Hazard. Mater., 333* (2017), July, pp. 1-9

[29] Li, Y., *et al*., The Physical Properties of Poly(l-lactide) and Functionalized Eggshell Powder Composites, *Int. J. Biol. Macromol., 85* (2016), Apr., pp. 63-73

[30] Khan, K., *et al*., Investigating the Feasibility of Using Waste Eggshells in Cement-Based Materials for Sustainable Construction, *J. Mater. Res. Technol., 23* (2023), Mar.-Apr., pp. 4059-4074

[31] Zhang, G., *et al*., Investigation of Frequency-Dependent Attenuation Coefficients for Multiple Solids Using A Reliable Pulse-Echo Ultrasonic Measurement Technique, *Measurement, 177*, (2021), June, 109270
[32] Robinson, D. E., *et al*., Measurement of Velocity of Propagation from Ultrasonic Pulse-Echo Data, *Ultrasound Med. Biol., 8,* (1982), 4, pp. 413-415, 417-420
[33] Tian, F., *et al*., An Ultrasonic Pulse-Echo Method to Detect Internal Defects in Epoxy Composite Insulation, *Energies, 12* (2019), 24, 4804
[34] Gür, C. H., Ogel, B., Non-destructive Microstructural Characterization of Aluminium Matrix Composites by Ultrasonic Techniques, *Mater. Charact., 47* (2001), 3-4, pp. 227-233
[35] Podymova, N. B., *et al*., Effect of Porosity on the Statistical Amplitude Distribution of Backscattered Ultrasonic Pulses in Particulate Reinforced Metal-Matrix Composites, *Ultrasonics, 108,* (2020), Dec., 106235
[36] Ozkan, V., *et al*., Influence of Mean Grain Size with Ultrasonic Velocity on Micro-Hardness of B4C-Fe-Ni Composite, *J. Alloys Compd., 574* (2013), Oct., pp. 512-519
[37] Erol, A., *et al*., Characterization of the Elastic Modulus of Ceramic–Metal Composites with Physical and Mechanical Properties by Ultrasonic Technique, *Open Chem., 20* (2022), July, pp. 593-601
[38] Unal, R., *et al*., The Mean Grain Size Determination of Boron Carbide (B4C) –Aluminium (Al) and Boron Carbide (B4C) –Nickel (Ni) Composites by Ultrasonic Velocity Technique, *Mater. Charact., 56* (2006), 3, pp. 241-244
[39] Yonetken, A., Bilici, V. Ö., Ultrasonic and Mechanical Characterization of Borided Ceramic-Metal Composite, *Russ. J. Nondestruct. Test, 58* (2022), Dec., pp. 779-789
[40] Toozandehjani, M., *et al*., Velocity and Attenuation of Ultrasonic Wave in Al-Al_2O_3 Nanocomposite and Their Correlation to Microstructural Evolution During Synthesizing Procedure, *J. Mater. Res. Technol., 15* (2021), Nov., pp. 2529-2542
[41] Filho, S. L. M. R., *et al*., Ultrasonic Pulse Velocity and Physical Properties of Hybrid Composites: A Statistical Approach, *Hybrid Adv., 2* (2013), Apr., 100024
[42] Parihar, H. S., *et al*., Effect of Variation of Steel Reinforcement on Ultrasonic Pulse Velocity Prediction in Concrete Beam, *Mater. Today: Proc., 65* (2022), 2, pp. 1486-1490
[43] Gültekin E. E., The Effect of Heating Rate and Sintering Temperature on the Elastic Modulus of Porcelain Tiles, *Ultrasonics, 83* (2018), Feb., pp. 120-125
[44] Toozandehjani, M., *et al*., Characterization of Aging Behavior of AA6061 Aluminum Alloy Through Destructive and Ultrasonic Non-destructive Testing Techniques, *Trans. Indian Inst. Met., 68* (2015), Dec., pp. 561-569
[45] Nan, J., *et al*., Particle Size Distribution Measurement Based on Ultrasonic Attenuation Spectra Using Burst Superposed Wave, *Results Phys., 13,* (2019), June, 102273
[46] Wang, J., *et al*., Frequency Dependence of Sound Speed and Attenuation in Fine-Grained Sediments from 25 to 250 kHz Based on A Probe Method, *Ocean Eng., 160* (2018), July, pp. 45-53
[47] Jodhani, J., *et al*., Ultrasonic Non-destructive Evaluation of Composites: A review, *Mater. Today: Proc., 78* (2022), 3, pp. 627-632
[48] Ramesh, C., *et al*., Evaluation of Mechanical Properties of Polyamide-Eggshell Powder Composite Materials, *Int. J. Sci. Technoledge, 2* (2014), 1, pp. 90-95
[49] Zaman, T., *et al*., Evolution and Characterization of Eggshell as A Potential Candidate of Raw Material, *Ceramica, 64* (2018), 370, pp. 236-241
[50] Katırcıoğlu-Bayel, D., Fire Retardant Mineral Fillers, OHU *J. Eng. Sci., 7* (2018), 3, pp. 1175-1179

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