MECHANICAL PROPERTIES OF EPOXY AND CONCRETE BASED COMPOSITE MATERIALS REINFORCED WITH OIL SHALE ASH

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Abstract. The elastic properties of composites with reinforcing particles are under investigation in this research. Oil shale ash (OSA), produced in Estonia, is a powder and a by-product of the combustion process in generation of electricity at power plants. During previous years many thousands of tons of OSA have been collected. Starting from 2018 in the European Union, oil shale ash is no longer considered as toxic and hazardous waste, this is opening new possibilities for commercial application of OSA in new products and giving it a second life. Recycling and reusing oil shale ash is therefore an important problem for environmental sustainability. The use of this product as a reinforcement for epoxy resin matrix as well as for the concrete matrix in Epoxy/OSA and Concrete/OSA composites was experimentally investigated in this work. Several sets of Epoxy/OSA specimens were experimentally fabricated: Epoxy resin without OSA, Epoxy resin with 10% of oil shale ash by weight and sets of specimens with increasing ratios (10% increase between) of oil shale ash by weight, until a 50% ratio of OSA was reached. The tensile test experiments were conducted with the goal to obtain elastic modulus of the oil shale ash composite material. Tensile tests were realized according to ISO 527-1:2012 and specimens were fabricated according to ISO 527-2:2012 1B standards. Tensile tests were performed, and the experimental results were compared with predictions from analytical approaches. According to the obtained tensile test results and analytical predictions (using the rule of mixture and other approaches), the modulus of elasticity increases in direct proportion with increasing weight fraction of the oil shale ash in the composite material. Finally, samples of concrete with OSA were fabricated and tested until failure. Different amounts (by weight) of cement (0%, 5%, 10%, 15%, and 35%) in the samples were replaced by OSA. The elastic properties of these samples were also experimentally evaluated and compared with theoretical prediction results.

Keywords: oil shale ash; composite material; particle composite; concrete.

Introduction

The environmental concerns about bypass products (wastes) obtained during utilization of fossil fuels for energy production are a growing global challenge. Both solid and gas wastes obtained using fossil fuels have become subjects of extensive investigation for safe and environment friendly disposal and/or reuse in a circular economy application [1]. One of these fuels is oil shale (OS).

Oil shale ash (OSA) is a product of combustion of oil shale (OS). Oil shale is a sedimentary rock that releases energy while burning. Solid organic substance contained in oil shale is insoluble in most of the organic solvents found in nature. For this reason, the combustible organic part remains inside the oil shale rocks. The organic part of oil shale mainly consists of hydrocarbons, which are very similar in structure to oil. The liquid part of hydrocarbons that is extracted from the oil shale rock is called shale oil [2]. Oil shale rock contains hydrocarbon compounds such as paraffin, olefin, aromatic and heteroaromatic hydrocarbons just like oil. The oil-like compounds from oil shale are extracted using thermal extraction processes [3]. The oil shale is mechanically pulverised and then burned. Circulating fluidised-bed (CFB) combustion method is a low-temperature combustion method in which temperatures range from 700 to 850 degrees Celsius, while Pulverized firing (PF) is a high-temperature combustion method in which temperatures can reach 1400 to 1500 degrees Celsius. Oil shale ash from power plants was classified as hazardous material according to the Material safety data sheet for burnt oil shale enforced with the EC regulations No 1907/2006 and EU No 453/2010. But starting from 2018 oil shale ash is no longer classified as hazardous material [1-4]. This gives OSA many new opportunities in applications, such as a particle filler for epoxy based composite materials or as an additive to concrete [5-7].

Epoxy based composites have a wide range of applications in different industries [8-11]. The mechanical properties of the composite can be adjusted changing of the proportion, shape and material of the constituents. Epoxy resins have an advantage of low shrinkage after curing, good adhesion and high strength comparing to different polymers [12; 13]. Uncured epoxy resins are more like a highly viscous liquid. They have poor strength and stiffness properties as well as other mechanical properties. For curing epoxy resins curing agents are used. After curing, epoxy polymers become strong isotropic thermoset structures with high elastic modulus and high tensile strength. At the same time, due to ductility and low fracture toughness, epoxy resins are not suitable for applications involving mechanical components such as gears or shafts. Different types of reinforcement have therefore been developed depending on the objectives to be achieved.

A study made by Ban A. Yousif [14] shows that fly ash reinforced polymer matrix with a small amount of short carbon fibers randomly distributed evenly around the volume of the material helps increase the tensile strength and elastic modulus, in the study the volume of fly ash was gradually increased without changing the weight ratio of the carbon fiber used with the polymer. Several hybrid composite specimens with 5%, 10%, 15% weight ratios of fly ash with constant ration of the polyester matrix with 5% carbon short fiber were tested. The tests were made according to ASTM D 638 for tensile testing and ASTM D 695 for compression tests [14]. The results showed an increase in the elastic modulus and tensile strength. In case of the compressive strength, it increases till 10% fly ash then decreases at 15% [15]. In the study made in [16], 5 types of epoxy and fly ash specimens were tested. Specimens with 0%, 5%, 10%, 15% and 20% weight ratio were made and tested according to ASTM: D 638 standard for the tensile test displacement 5 $mm \cdot min^{-1}$ and in the compression test 2 $mm \cdot min^{-1}$ (ASTM: D 695) was used. Tensile strength of the fly ash composite increases as the ratio of fly ash increases. The same happens with the compression strength. And in the situation with the elastic modulus, it increases till 5% of the weight ratio of the fly ash and then further addition of fly ash decreases the elastic modulus. The highest tensile and compression strength parameter was shown by 20% fly ash weight ratio [16]. In our study, the mechanical properties of different concentration oil shale ash epoxy composites were tested depending on the concentration of the filler.

Materials

Interest in polymer matrix composite materials with particles is permanently stable. Different authors are testing and investigating composites with different polymeric resins [17-19] as a matrix and different size and origin particles [20]. In a similar way, investigations are performed with concretes [20] and polymer concretes [21] with fly ash particles and different types of resin. Not many investigations were done working with oil shale ash powders [22] and interest in such materials is growing. Mechanical investigations are mainly focused on material strength [18-20; 22] and the question about the elastic modulus in many situations is the subject for future investigations.

Materials used in this study are epoxy resin *Wela EP100* and hardener *Wela EH08*. Oil shale ash came from Auvere (Estonia) thermal power plant. The concrete samples were made using: cement, sand, quartz, water, some plasticizers, micro silica and gravel.

Epoxy sample fabrication

Specimens were fabricated according to the ISO 527-2:2012 standard (Fig. 1) [16].

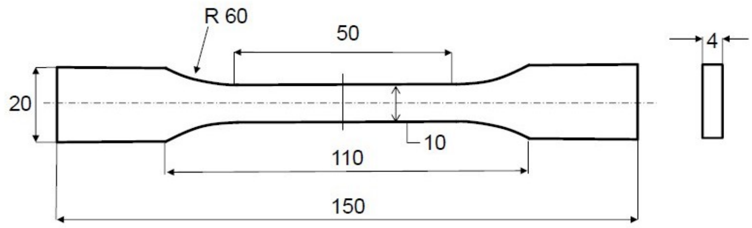

Fig. 1. **Geometry for tensile test specimen type 1B (EN ISO 527-2:2012)**

Moulding silicone with cold curing tin catalyst was poured into the precision laser cut mould (Fig. 2). After silicone curing, epoxy resin with oil shale mixed was poured into the silicone mould. The specimens were cured for 48 hours (Fig. 3) and after curing in the silicone mould, the epoxy samples were removed for further machining.

The machining process includes grinding the sharp ends and refining geometry according to ISO 527-2:2012 type 1B geometry standard. Afterwards, the specimens are put into the tensile test machine (Fig. 4).

Fig. 2. **Mould for the silicone mould. Sample type 1B (EN ISO 527-2:2012)**

Fig. 3. **EN ISO 527-2:2012 1B Specimen with 0% OSA filler in the silicone mould curing**

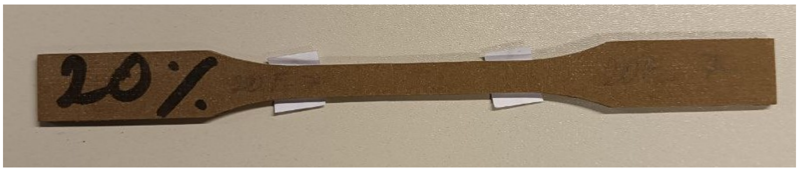

Fig. 4. **EN ISO 527-2:2012 1B specimen with 20% OSA filler by weight ready to test**

Tensile tests were conducted on the Zwick Roell Z150 testing machine according to the above mentioned standard. Test velocity was 5 mm·min^{-1} (Fig.5).

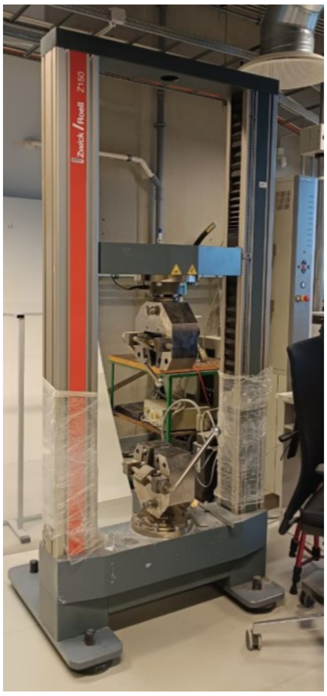

Fig. 5. **Zwick Roell Z150 testing machine**

The obtained test results were compared with the analytical prediction results. Two theoretical predictions approaches were used, one - the rule of mixture and second numerical modelling results based on the 3D FEM approach. The material filler is oil shale ash, and OSA is a powder with particles changing in linear size from hundreds of nano-meter until hundreds of microns. In order to determine the particle size distribution, OSA was investigated using the lasergranulometric equipment Malvern Mastersizer 3000. Its measurement scale is: 10 nm to 3.5 mm [23]. The particle size distribution for OSA is shown in Fig.6. Small particles are precise round balls, with increase of the diameter the particles can slightly deviate from the round ball shape obtaining forms of ellipsoids and slightly non-regular shape bodies. In our FEM modelling, we observed particles with round ball shape, having stochastically average diameter.

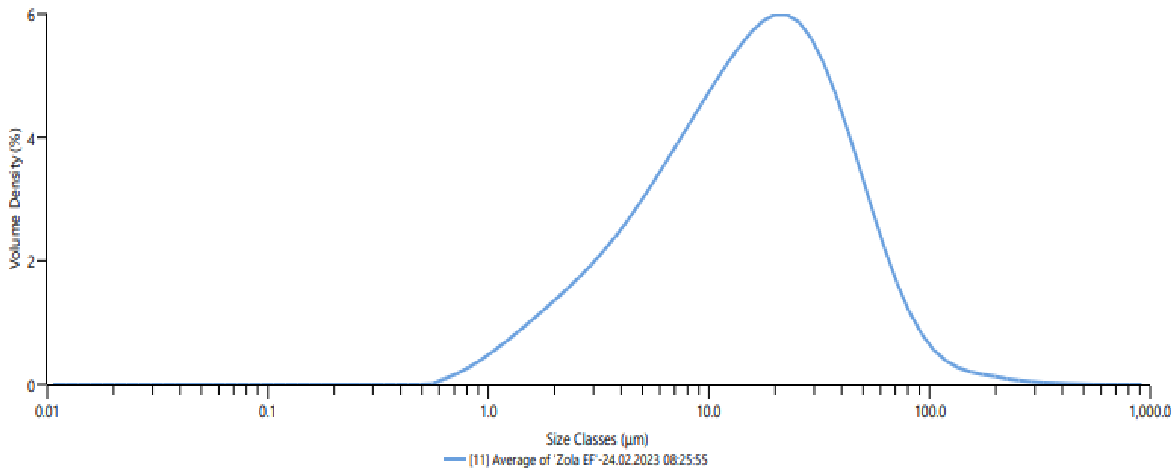

Fig. 6. **OSA granulometry**

Experimental results (Epoxy matrix composite with OSA)

The obtained test results in tensile experiments. The samples were fabricated measuring the weight ratio, which is easy to do using laboratory scales. The results and volumetric ration of them are given in the table below. Volumetric values will be used in analytical calculations.

Table 1

Experimental results of the tensile test

Mass of OSA, %	Elastic modulus, Pa	Mass of epoxy resin, g	Mass of OSA, g	Epoxy resin volume, cm^3	Volume of OSA, cm^3	Volume fraction of OSA V_f, %
0	3.24E + 09	15.6	0	13	0	0
10	3.28E + 09	14.04	1.56	11.7	0.58	4.76
20	3.62E + 09	14.4	3.6	12	1.35	10.10
30	5.07E + 09	13.3	5.7	11.08	2.13	16.15
40	5.74E + 09	13.2	8.8	11	3.29	23.05
50	6.25E + 09	12.5	12.5	10.42	4.68	31.01

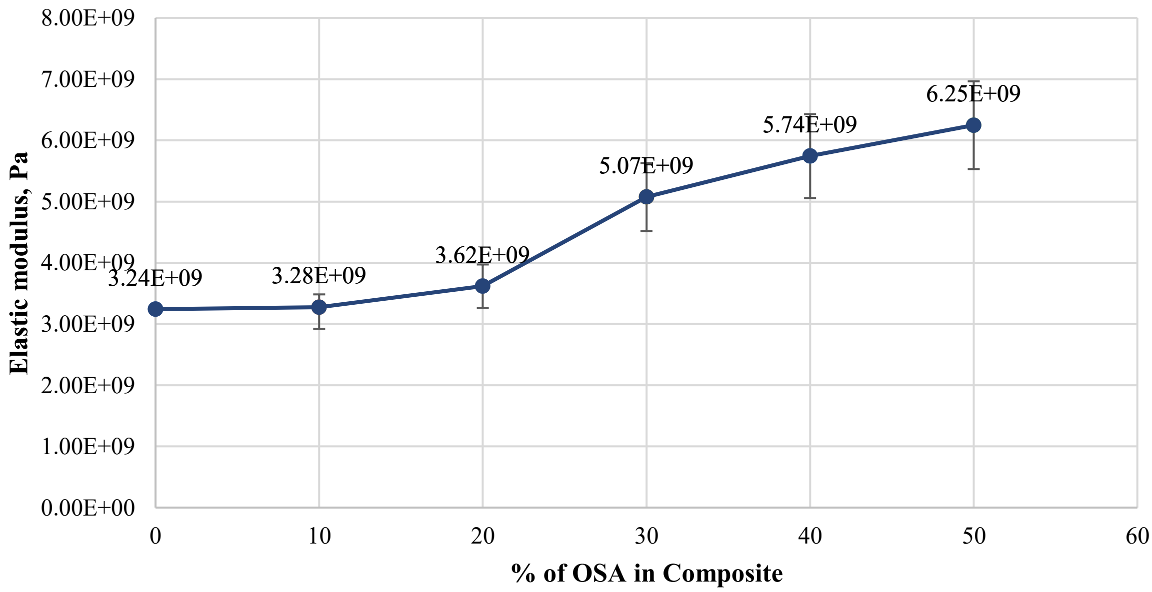

Fig. 7. **Experimentally measured elastic modulus of Epoxy/OSA composite depending on amount of OSA**

As seen in Figure 6, the elastic modulus of the oil shale ash filled material increases making it more brittle and less elastic. The tensile strength shows almost no change with values around 25 MPa.

Young modulus prediction for Epoxy/OSA composite

Rule of mixtures (1) is widely used for composite material properties determination depending on volume ratios [24].

$$E_c = E_f \cdot V_f + E_m \cdot V_m, \tag{1}$$

where E_c – elastic modulus of composite, Pa;
E_f – elastic modulus of filler (OSA), Pa;
E_m – elastic modulus of matrix (epoxy resin), Pa;
V_f – particle volume fraction, unitless;
V_m – matrix volume fraction, unitless.

Using the data about the volume ratio of oil shale ash in the composite material, it is possible to find the elastic modulus of the composite. The density of our oil shale ash is 2300 $kg \cdot m^{-3}$ (in literature the density of the ash sediment varies between 1800 and 2500 $kg \cdot m^{-3}$ [24]).

Table 2

OSA volume ratio in the specimens

Volume of matrix (epoxy resin), cm^3	Volume of filler (OSA), cm^3	Volume ratio of OSA in composite material
13.00	0	0
11.70	0.68	5.48
12.00	1.57	11.54
11.08	2.48	18.27
11.00	3.83	25.82
10.42	5.43	34.29

According to the calculations by the rule of mixtures, the values are constantly increasing. The difference between the values is shown in the figure in the results and discussions section below.

Software used for numerical for simulation and modelling is Solidworks. A 30 µm x 30 x 30 µm cube was modelled. Round ball shaped particles with OSA elastic properties were inserted into the cube. The radius of the sphere was changing according to the oil shale ash volume fraction. According to symmetry conditions, only one 4th part of the model was used in calculations to optimise the computational process time. A displacement of Δ mm was applied to the free face of the cube located to the observer (Fig. 8). The obtained stress in the direction of the applied displacement was divided by strain. In this way the elastic modulus was obtained.

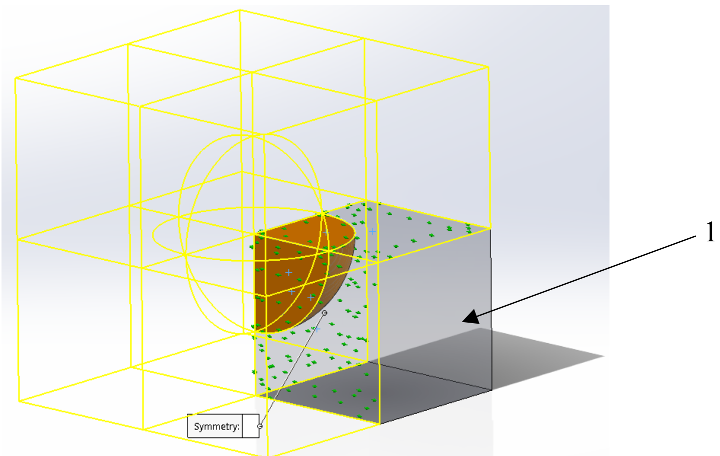

Fig. 8. **Geometrical model for numerical calculation (Solidworks) for 20% of oil shale ash by weight:** 1 – displacement of Δ mm is applied on this face

The simulation results, rule of mixtures results and experimental data are shown in Fig. 8.

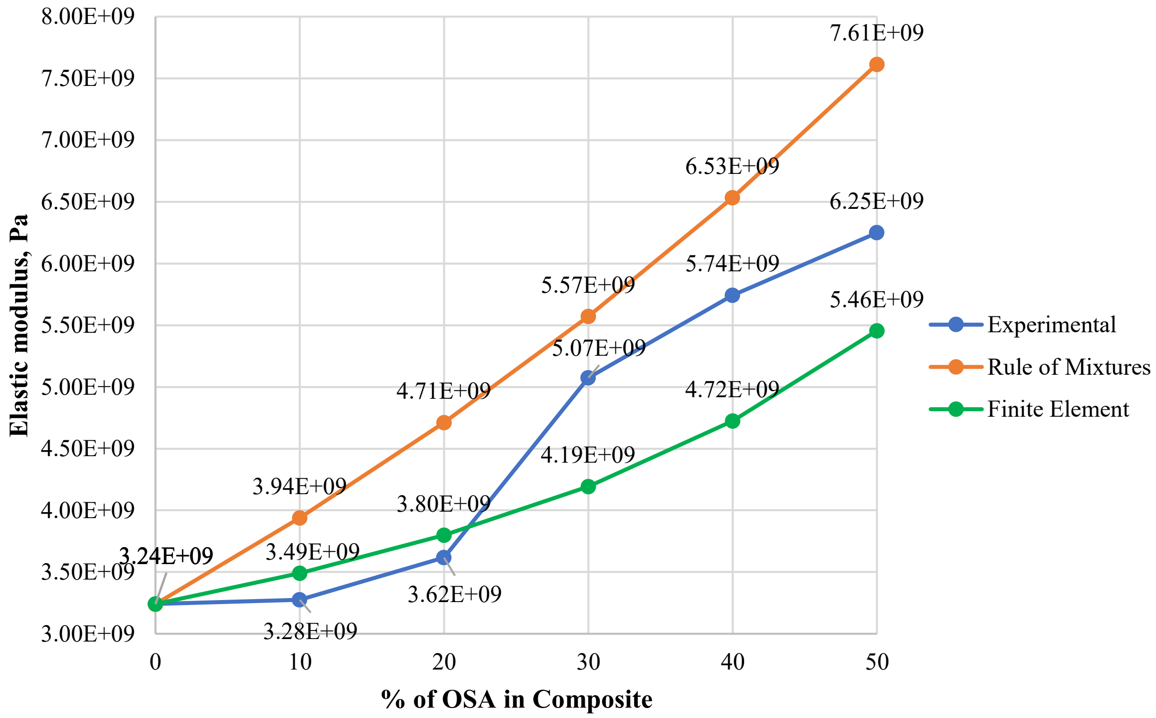

Fig. 9. **Epoxy/OSA elastic modulus dependence on OSA content. Experimental, analytical (rule of mixture) and numerical modelling (FEM) data**

The performed investigation was done with homogeneous epoxy resin matrix and rigid OSA particles. Theoretical predictions show a relatively good fit with the experimental data.

The next investigation was done with a highly heterogeneous material – concrete. Concretes with inclusions, such as short fibres, are materials with high interest to its mechanics [26- 30]. An important question is: can we use the rule of mixture and numerical FEM predictions in this situation? If inclusion properties – density, modulus will be highly different from the properties of plane concrete, we can expect the rule of mixture will predict properly mechanical properties of the composite. The situation is not clear if inclusions will be OSA. To clarify this situation because of importance of OSA for use in concrete composites, we mathematically predicted properties of Concrete/OSA composites using the rule of mixture approach. Concrete surrounding OSA particle is observed as homogeneous material with averaged over volume properties. Concrete with 0% OSA is the matrix material in our calculations. We used data calculates according to all three formulas (2), (3), (4). Using data about elastic modulus of OSA material and OSA volume fraction values, we calculated dependence of the concrete/OSA composite modulus on the OSA volume fraction value V_f.

Concrete sample fabrication

Concrete samples were prepared by mixing the concrete components together. Concrete without gravels and with the largest sand particle linear size 2.5 mm in the mixture was reinforced by OSA particles.

Experimental results (Concrete matrix/OSA composite)

The results of the compressive strength experimental testing, on the concrete cubes (10 cm x 10 x 10 cm), are given in the table below. Tests were conducted according to standards. The test results are shown in Table 3 and Fig. 10.

Table 3

Compressive strength of the concrete specimens

OSA by weight or by volume	Compressive strength, MPa
0%	31.2
10%	28.7
15%	26.5
20%	27.2
25%	25.2
30%	29.6
35%	28.4
55%	25.9

Elastic parameter evaluation

Several approaches are used for calculating the elastic modulus of concrete knowing the compressive strength [25]. Clause 6.2.3.1 of IS 456: 2000 says that short-term elastic modulus can be found by the formula (2).

$$E_c = 5\sqrt{f_{ck}} = 5.59\sqrt{f_c'} \tag{2}$$

where E_c – elastic modulus of composite, GPa;
f_c' – concrete cylinder compressive strength, Pa;
f_{ck} – concrete cube compressive strength, Pa.

Clause 19.2.2.1 of ACI 318:2019 and also AASHTO-LFRD-2006 use the formula (3) for the modulus of elasticity, considering the density (unit weight) of concrete

$$E_c = 0.043\rho_c^{1.5}\sqrt{f_c'} \times 10^{-3} \tag{3}$$

where E_c – elastic modulus of composite, GPa;
ρ_c – unit weight of concrete (2300 kg·m^{-3}), kg·m^{-3};
f_c' – concrete cylinder compressive strength, Pa.

Russian SP 52-101-2003 uses a different approach for the concrete elastic modulus calculation (4).

$$E_c = 11.652\ln(f_c') - 7.4713 \; with \; 10MPa \le f_c' \le 60MPa \tag{4}$$

where E_c – elastic modulus of composite, GPa;
f_c' – concrete cylinder compressive strength, Pa.

The calculations using these formulas are shown in Table 4 and Figures 11-13 below.

Table 4

Elastic modulus calculated by rule of mixture

OSA percent by weight	Elastic modulus obtained by rule of mixture using formula (2), GPa	Elastic modulus obtained by rule of mixture using formula (3), GPa	Elastic modulus obtained by by rule of mixture using formula (4), GPa
0%	27.93	26.49	32.62
10%	27.47	26.06	32.22
15%	27.24	25.84	32.02
20%	27.02	25.63	31.82
25%	26.79	25.41	31.62
30%	26.56	25.20	31.42
35%	26.34	24.99	31.23
55%	25.45	24.14	30.45

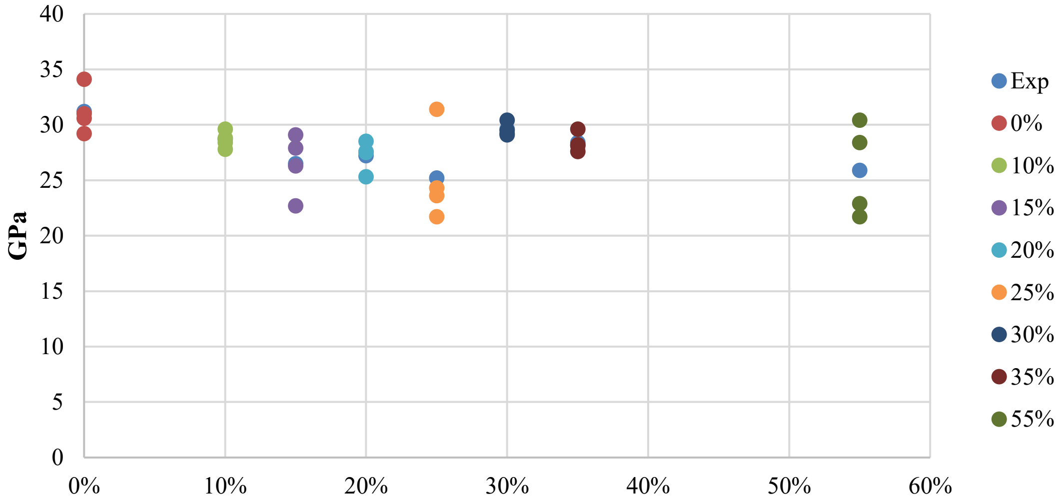

Fig. 10. **Concrete/OSA composite experimentally measured compression strength depending on OSA content**

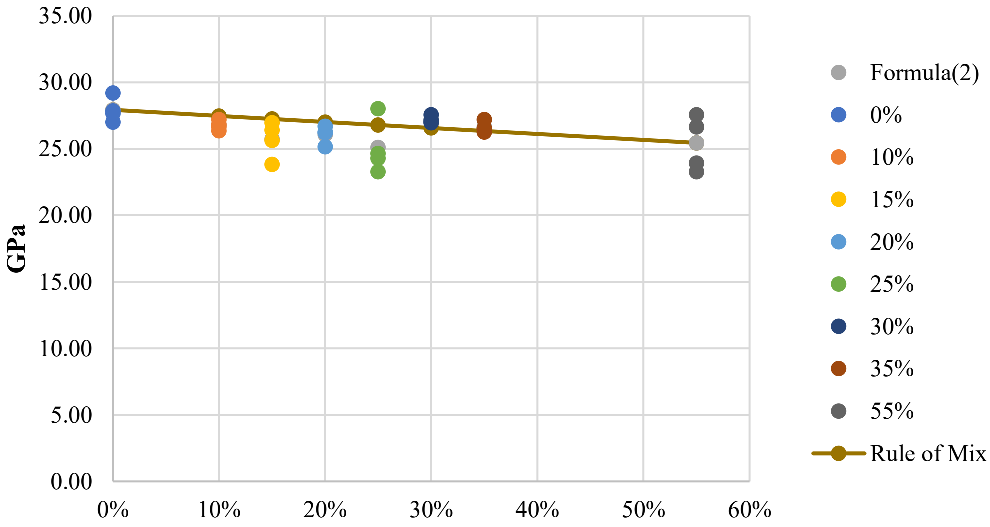

Fig. 11. **Based on experimental data calculated elastic modulus according to formula (2)**

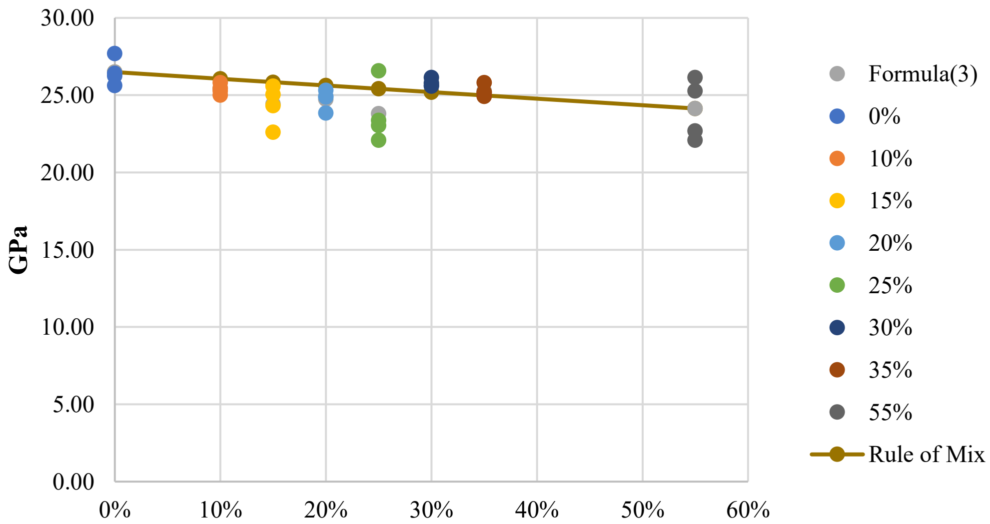

Fig. 12. **Based on experimental data calculated elastic modulus according to formula (3)**

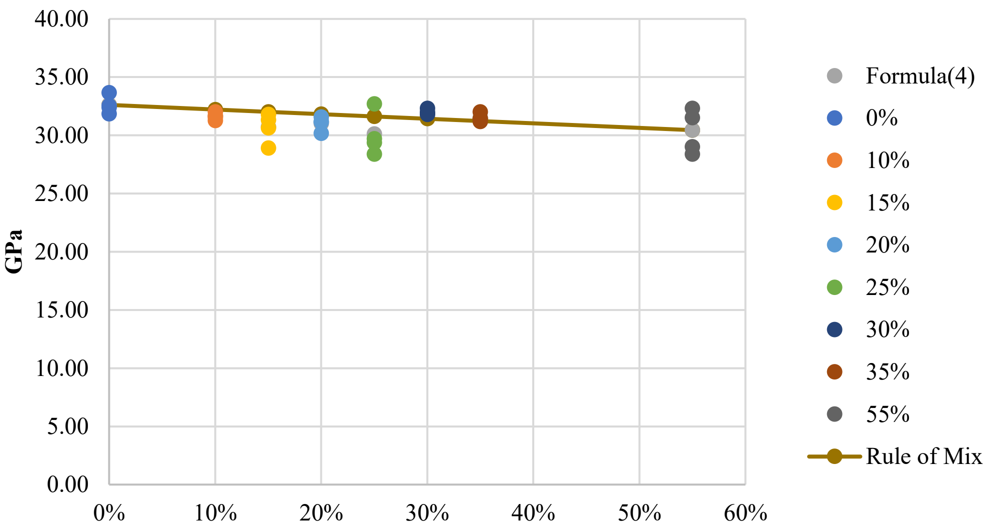

Fig. 13. **Based on experimental data calculated elastic modulus according to formula (4)**

Discussion of the results

The graph above (Figure 9) shows the elastic modulus results obtained experimentally testing the polymer matrix composite (epoxy resin *Wela EP100* hardener *Wela H08*), reinforced with different concentrations of small OSA particles, in comparison with the prediction made according to the "rule of mixture" and realising the numerical FEM model. Particle distribution according to diameters was obtained experimentally and is shown in Figure 6. Microscopical investigation shows, for a small size particle, its form is very close to sphere, for bigger it is possible to find deviations from this form. Modulus prediction according to the "rule of mixture" is giving higher results comparing to the experiment, because the particle form and geometry is not included in it. Numerical FEM modelling prediction better fits the experimental data, at the same time, it used averaged one diameter size for all particles. Experimentally it is possible to see small particle agglomeration. These factors are playing a bigger role with increase of OSA concentration (see Fig. 9).

The experimentally obtained elastic modulus values for concrete with different amounts (concentrations) of OSA are shown in Figures 10-11. Data were obtained according to the rule of mixtures calculation and recalculated from the experimental data about the compression strength according to formulas (2), (3), (4). As it can be seen, the elastic modulus of the Concrete/OSA composite slightly decreases with increasing of the volume of OSA. The rule of mixture prediction, shown in Fig.11a-11c, fits well with the experimental data. The modulus is decreasing because the modulus of OSA material is smaller than the emodulus of the surrounding concrete matrix. In general, the prediction data according to the rule of mixture and numerical calculations based on the FEM approach are well approximating the elastic modulus of the composite Epoxy/OSA in the situation when the modulus of OSA is bigger than the epoxy matrix modulus and the material matrix is highly homogeneous. The rule of mixture predicts well dependence of the Concrete/OSA Young modulus when we are changing the amount of OSA (0% to 55%) in heterogeneous material. This result is not obvious since concrete is a highly heterogeneous material with internal particles (sand) larger than reinforcing particles of OSA. Prediction data are giving the right tendency of decrease of the Young modulus in a wide range of OSA implementation (till 55%).

Conclusions

1. In the present research the elastic parameter (Young modulus) of Epoxy resin/OSA (Oil shale ash came from Auvere (Estonia) thermal power plant) composite was measured experimentally, depending on the amount of OSA (0%-55%). Modulus prediction according to the "rule of mixture" is giving higher results comparing to the experiment, because the particle form and geometry are not included in it. Numerical FEM modelling prediction better fits with the experimental data. Next improvement may be obtained using particle size distribution in numerical prediction.

2. Similar parameters were predicted using the rule of mixture analytical formula and FEM numerical modelling. The prediction result comparison with the experiment shows good agreement (especially for the numerical approach).
3. The elastic modulus of fine-grained concrete was obtained experimentally using strength data for concretes having different amount of OSA (0%-55%).
4. Elastic modulus of fine-grained concrete theoretical prediction was done using the rule of mixture analytical formula. Comparison with the experiments shows well agreement, despite the fact - concrete is a highly heterogeneous material with internal particles (sand) larger than reinforcing particles of OSA. The predictions data are giving the right tendency of decrease of the Young modulus in a wide range of OSA implementation.

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Author contributions

Conceptualization, A.M.; methodology, A.M. and E.G.; software, I.J.; validation, M.V. and E.G.; formal analysis, M.V. and E.G.; investigation, I.J.; and A.M.; writing – original draft preparation, I.J.; writing – review and editing, I.J.

All authors have read and agreed to the published version of the manuscript.

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