



# Article Enhanced Eco-Friendly Concrete Nano-Change with Eggshell Powder

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Abstract: One of the unifying factors for all countries is the large consumption of chicken, and other, eggs in food and other types of economic activity. After using various types of eggs for their intended purpose, a large amount of waste accumulates in the form of eggshells. Currently, this problem exists and needs a non-trivial, original solution. The aim of the work was to fill the scientific gap in the direction of studying the microstructure formation of improved nano-modified environmentally-friendly concrete based on eggshell powder and obtaining a concrete composition for the manufacture of an industrial sample of such a material. An environmentally-friendly concrete was obtained, the characteristics of which were improved relative to standard concrete by modifying it with eggshell powder, for which the optimal dosage was determined. The most effective was the replacement of part of the cement with eggshell powder in the amount of 10%. The maximum increase in strength characteristics ranged from 8% to 11%. The modulus of elasticity increased by 4% compared to the control samples without eggshell powder. The maximum reduction in deformations under axial compression and tension in comparison with the control values ranged from 5% to 10%. The study of the composite's microstructure nano-modified with eggshell powder, and an analysis of the changes occurring in this microstructure due to nano-modification, confirmed the improvement in characteristics and the optimal dosage of eggshell powder.

Keywords: eggshell concrete; eggshell powder; nano-modification; sustainable concrete; recycling

# 1. Introduction

Modern production and industrial sectors of the economy, especially those of industry, agriculture, and construction, face the problem of ecology. More and more questions are raised regarding the achievement of the UN sustainable development goals. A large role in this is given to the problem of accumulated waste from various types of economic activity of a person, society, business, and the state. Construction is a versatile sector that often offers non-trivial solutions to solve several problems, including economic and environmental ones, to achieve greater comfort, sustainable development goals and, in general, provide a higher standard of living for the world's population.

One of the unifying factors for all countries is the large consumption of chicken, and other, eggs in food and other types of economic activity. After using various types of eggs



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**Copyright:** © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). for their intended purpose, a large amount of waste accumulates in the form of eggshells. Thus, this problem exists and needs a non-trivial, original solution.

Eggshells are high in calcium, mostly composed of several layers of calcium carbonate, which is one of the four main ingredients for cement production. The main cement compounds are dicalcium silicate ( $C_2S$ ), tricalcium silicate ( $C_3S$ ), tricalcium aluminate ( $C_3A$ ), and tetracalcium aluminoferrite ( $C_4AF$ ). Thus, the eggshell can be used as a replacement for part of the cement. The use of eggshell powder ( $CaCO_3$ ) as a partial replacement for cement improves compressive strength by stabilizing ettringite and monocarbonate [1].

As a rule, the main enterprises that produce eggshells in large quantities are bakeries, restaurants, and bakery factories. Before using the eggshell instead of the binder component, the raw shell goes through a certain preparation process. The standard stages of eggshell preparation to obtain an additive are its washing, ultrasonic cleaning, and drying in an electric oven at a temperature of 120  $^{\circ}$ C for 24 h [2–4].

For example, the preparation of nanosized CaO in most cases is carried out by calcination (formula (1)) [2,3]:

$$CaCO_3 \xrightarrow{900 \,^{\circ}C \text{ for } 2 \text{ h}} CaO + CO_2 \tag{1}$$

Ignition at a temperature of 900  $^{\circ}$ C for 3 h makes it possible to completely burn out all organic material and convert a significant proportion of CaCO<sub>3</sub> into CaO [4].

As for the chemical composition of the eggshell, it mainly consists of  $CaCO_3$  (approximately 94% of the total weight), organic matter (approximately 4%), calcium phosphate (approximately 1%), and magnesium carbonate (approximately 1%) [1]. Table 2 presents the chemical compositions of eggshell powder obtained from the results of some studies [5–9].

Table 1. Chemical composition of eggshell powder.

Source	$SiO_2$	CaO	$Al_2O_3$	Fe <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	Cl	K <sub>2</sub> O	SO <sub>3</sub>	MgO	$P_2O_5$	SrO	LOI
[5]	0.09	50.7	0.03	0.02	0.19	0.219	-	0.57	0.01	0.24	0.13	-
[6]	0.13	50.09	0.03	0.03	0.16	-	-	0.61	0.02	-	-	-
[7]	0.24	93.2	0.23	-	0.13	-	0.07	0.2	0.85	0.47	0.28	-
[8]	0.05	98.0	0.05	0.02	-	-	0.11	0.49	1.12	0.1	-	-
[9]	0.1	53.6	1.55	0.62	-	-	0.01	1.57	-	-	-	40.7

Table 2 shows that the main component of eggshell powder is CaO, the content of which ranges from 50% to 98%. Silicon oxide is present, but in a much smaller amount. The strength characteristics of concrete directly depend on the amount of eggshell powder used as an additive or replacement for part of the cement (Table 2).

**Table 2.** The use of eggshell powder in concrete or mortar and its effect on the strength characteristics of finished composites.

The Role of Eggshell in Concrete	Source	Influence of Eggshell Powder on the Physical and Mechanical Characteristics of Concretes and Mortars		
	[10]	The addition of eggshell powder in an amount of 7–9% by weight of cement provides an increase in compressive strength up to 55% in comparison with the strength characteristics of samples of Portland cement.		
Additive	[11]	According to the results of the study, it was found that the addition of powder in an amount of 15% is the most optimal and provides a decrease in water absorption up to 30%, an increase in compressive strength up to 29%, and a decrease in the thermal conductivity index up to 40% in comparison with similar indicators for samples of the control composition.		

The Role of Eggshell in Concrete	Source	Influence of Eggshell Powder on the Physical and Mechanical Characteristics of Concretes and Mortars
	[4]	Increasing the compressive strength to 20.16 $\pm$ 4.39 MPa in combination with the addition of nanosilica sol
-	[12]	Eggshell-derived calcium carbonate improved compressive strength.
	[13]	It was found that 10% eggshell powder as a partial substitute for cement provides a 57% increase in strength compared to the control composition samples.
	[14]	Part of the cement was replaced with 20–50% fly ash (FA) and 5–10% eggshell powder (ESP). The optimal percentage of replacement of cement in the concrete mixture for FA was 30–40%, and for ESP 5%. The mixtures with 5% ESP and 30% FA and with 5% ESP and 40% FA showed the highest results compared to other mixtures, 58.9 MPa and 56 MPa—compressive strength results, respectively.
	[15]	It has been established that partial replacement of cement with eggshell powder in an amount of 10% is optimal for fluidity and workability, and 20% for compressive strength and flexural strength.
-	[16]	10% eggshell powder, when used in mortar, has been tested to provide optimum compressive strength.
Replacing part of the cement or sand	[17]	Replacing part of the sand with eggshell powder in an amount of 40% is the most optimal, as it accelerates carbonization and increases compressive strength.
-	[18]	Replacing cement with eggshell powder in an amount of 5% provides a solution with optimal consistency and strength characteristics. However, the water absorption of the solution with eggshell powder is lower compared to the control composition.
	[19]	Cement was partially replaced by eggshell powder at 5%, 10%, 15% and 20%. The compressive strength of the concrete increased by up to 13%, and the bending strength by up to 11% at 10% ESP dosage compared to 0% ESP dosage.
	[20]	Replacement of ESP cement at 5, 10 and 20% maintained the standard mortar compressive strength. The spent eggshell was subjected to heating up to 450 °C. Heating up to 450 °C allows the removal of the organic membrane before crushing and grinding into a powder form without changing the CaCO <sub>3</sub> composition.
	[21]	The addition of ESP instead of part of the cement in an amount of 5% to 10% leads to an increase in the compressive strength of the concrete. This replacement is most optimal for mixture compositions with a higher water content.
	[22]	Obtaining pure CaO by heating eggshells to 900 °C and replacing part of the cement (5%, 10%, 15%) with it made it possible to obtain new antibacterial composites.

#### Table 2. Cont.

Regarding the corrosion resistance of concretes with eggshell powder, according to generally accepted facts, concrete with ESP has a large loss in weight and strength when exposed to solutions of sulfates and chlorides [23,24]. For example, in [23], based on the results of experimental studies, it was found that the percentage of weight loss increases with the percentage of eggshell. Thus, an increase in the content of eggshell powder reduces the resistance of concrete to acid attack, since calcium hydroxide Ca(OH)<sub>2</sub> is the component most vulnerable to acid attack [23].

Production and construction have a point of contact in the form of production of building materials, products, and structures. There are several works devoted primarily to solving environmental and economic problems through the disposal of various types of waste by using them in building materials [3,5,12–16,19,25–40]. In addition, there are works related to the improvement of the properties of composite building materials, products, structures, and first, concretes due to their nano-modification [41–55].

The aim of the work is to fill the scientific gap in the direction of studying the microstructure formation of improved nano-modified environmentally-friendly concrete based on eggshell powder and obtaining a concrete composition for the manufacture of an industrial sample of such a material.

Thus, in our study, we aim to combine the concept of "waste disposal" with eggshell specification and develop a method of processing to the smallest sizes to achieve the degree of nano-modifying powder for its subsequent use in concrete. The use of nano-modifying powder in concrete as a nano-modifier is aimed at improving the quality of concrete in general, increasing its performance, improving its structure, and hence the research objectives are formulated:

- a detailed review and analysis of the literature on the issues of eggshell utilization in concrete technology;
- development of a method and optimal dosage of nano-modifying eggshell powder for concretes with improved characteristics;
- experimental verification of the achieved results through laboratory research and processing of the results with their analysis;
- study of the microstructure of concretes formed nano-modified by powder based on eggshell with an analysis of changes occurring in such a microstructure due to nano-modification.

The scientific novelty of this research is the obtaining of new relationships between the dosage of the nano-modifying eggshell powder, the characteristics and microstructure of the resulting improved concretes, the development of the theory of various types of nano-modifiers in concretes, and the acquisition of new knowledge about fundamental and applied interactions occurring at the micro level in concretes of a new type.

The practical significance of the study is the developed method for obtaining the optimal dosage of eggshell powder as a nano-modifier for concrete.

Therefore, our scientific goal was aimed at filling scientific deficits in terms of microstructure formation at the nano level, and the practical significance was based on reducing the cost of not only concrete, but also of the process of its manufacture.

## 2. Materials and Methods

#### 2.1. Materials

Portland cement without additives CEM I 52.5 N produced by OAO Novoroscement (Novorossiysk, Russia) was used as a binder in experimental studies. The main characteristics, and chemical and mineralogical composition of cement are presented in Tables 3–5.

Table 3. Characteristics of cement.

Properties	Value	
Specific surface, cm <sup>2</sup> /g	3306	
Normal density, %	27	
True density, $kg/m^3$	3157	
Setting time, h.—min.		
- Start	2–15	
- Finish	3–35	
Compressive strength at the age of 28 days, MPa	56.7	

Element	Amount, %
Chemica	l composition
SiO <sub>2</sub>	21.04
$Al_2O_3$	4.01
Fe <sub>2</sub> O <sub>3</sub>	4.15
MgO	1.37
CaO	65.53
$SO_3$	1.44
TiO <sub>3</sub>	0.064
LOI	1.97
Na <sub>2</sub> O	0.17
К <sub>2</sub> О	0.24
Chlorine ion CI <sup>–</sup>	0.011

Table 4. Chemical composition of cement.

Table 5. Mineralogical composition of cement.

Cement Type	C <sub>3</sub> S	$C_2S$	C <sub>3</sub> A	C <sub>4</sub> AF
PC 500 D0	68	13	7	12

Granite crushed stone produced by Yug-Nerud (Pavlovsk, Russia) was used as a coarse aggregate. Physical and mechanical characteristics of coarse aggregate according to GOST 8267-93 "Crushed stone and gravel of solid rocks for construction works. Specifications" are presented in Table 6.

Table 6. Physical and mechanical characteristics of crushed granite.

Fraction	Bulk Density, kg/m <sup>3</sup>	True Density, kg/m <sup>3</sup>	Crushability, wt. %	The Content of Lamellar and Needle-Shaped Grains, wt. %	Void, %
10–20	1423	2625	11.6	8.0	45

Quartz sand produced by OAO Arkhipovsky Quarry (village Arkhipovskoe, Russia) was used as a fine aggregate. Fine aggregate characteristics according to GOST 8736–2014 "Sand for construction works. Specifications" are given in Table 7.

Table 7. Characteristics of quartz sand.

I	ndicator Title			Va	lue			
	Sieve size, mm	2.5	1.25	0.63	0.315	0.16	<0.16	
Grain composition of sand	Private balances, %	2.22	2.03	2.90	) 34.65 4 41.80 1.53 0.54 2697 1438	55.79	2 / 1	
-	Total balances, %	2.22	4.25	7.14	41.80	315 0.16 4.65 55.79		
		1.53						
Content of d	ust and clay particles, %		0.54					
	ain density, kg/m <sup>3</sup>		2697					
	1438							
	Bulk density, kg/m <sup>3</sup> Voidness of sand, %			45				

As a nano-modifying additive, eggshells obtained from a bakery were used. The eggshell used as a raw material for the nano-modifier was obtained during the processing of chicken eggs. After collecting the required amount of eggshell, it was initially processed by crushing to a monofraction-dispersed crushed material, homogeneous in structure, size,

and other external characteristics. After preliminary grinding by pounding, the material was dried and brought to homogenization. After that, the shell was crushed to the state of a finely-dispersed powder using the Activator-4M mill, the detailed characteristics and parameters of which are presented in [37,56].

Types of experimental compositions are presented in Table 8.

Table 8. Types of experimental compositions.

<b>Composition Number</b>	C, %	ESP, %	S, %	CS, %	W, %
1	100	0	100	100	100
2	95	5	100	100	100
3	90	10	100	100	100
4	85	15	100	100	100
5	80	20	100	100	100
6	75	25	100	100	100
7	70	30	100	100	100

Note: C-cement, S-sand, CS-crushed stone, W-water.

## 2.2. Methods

The production of eggshells was carried out as follows. First, the resulting shell was soaked for a day in water at a temperature of  $20 \pm 5$  °C, then the eggshell was washed under running water until it was completely cleansed of organic residues. Next, the fragments of the eggshells were placed in an oven. The drying process was carried out for 24 h at a temperature of 150 °C, which is necessary to remove the moisture contained in the eggshell. Grinding of dried eggshells was carried out in an Activator-4M planetary ball mill (Novosibirsk, Russia) for 6 h at a speed of 800 rpm. The resulting powder was passed through a sieve with a mesh size of 60  $\mu$ m, from Vibrotekhnik LLC (St. Petersburg, Russia), after which this powder was used in experimental studies.

The granulometric composition of the eggshell powder was evaluated using a MicroSizer-201C laser particle analyzer, from OOO VA Install (St. Petersburg, Russia). Figure 1 shows the particle size distribution curve of eggshell powder.

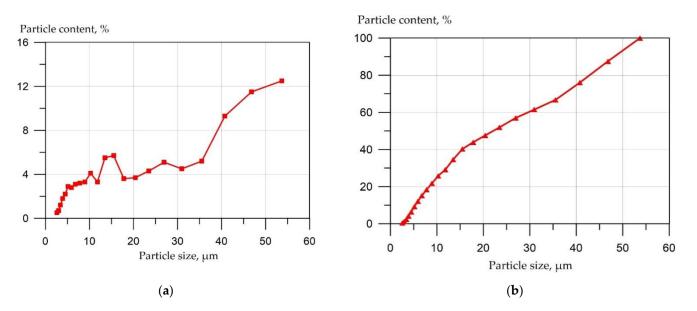


Figure 1. Eggshell Powder Particle Size Distribution Curve: (a) particle distribution; (b) cumulative curve.

Heavy concrete of class B30 was designed as a control composition. The parameters of the composition of the concrete mixture are presented in Table 9.

Parameter Title	W/C	C, kg/m <sup>3</sup>	W, L/m <sup>3</sup>	CS, kg/m <sup>3</sup>	S, kg/m <sup>3</sup>	$\rho_{cm}$ , kg/m <sup>3</sup>
Value	0.5	375	190	1158	702	2425

Table 9. Parameters of the composition of the concrete mixture.

For the manufacture and further testing of samples in this study, the following were used:

technological equipment—laboratory concrete mixer BL-10 (ZZBO LLC, Zlatoust, Russia);

- testing equipment—hydraulic press IP-1000 (NPK TEHMASH LLC, Neftekamsk, Russia), R-50 tensile testing machine (IMash LLC, Armavir, Russia);
- measuring instruments [56–60].

The preparation of the concrete mixture was carried out in the following sequence: at the first stage, cement and sand were mixed. At the second stage, the resulting eggshell powder was introduced At the third stage, mixing water was introduced and at the fourth stage, coarse aggregate was introduced into the resulting mortar mixture. The mixture was stirred until a homogeneous consistency was obtained. Further, the finished concrete mixture was poured into metal molds of Smolensk SKTB SPU OJSC (Smolensk, Russia) and subjected to vibration at the laboratory vibration platform SMZh-539-220A IMASH LLC (Armavir, Russia). Then, the molds with the mixture were placed in the normal hardening chamber of RNPO RusPribor LLC (St. Petersburg, Russia) and kept there for a day. Next, the samples were removed from the molds and placed again in the hardening chamber, where they were kept for another 27 days. After 28 days, the samples were tested.

Compressive strength, tensile strength in bending, axial compression, and axial tension were determined according to GOST 10180 "Concretes. Methods for strength determination using reference specimens".

During compression tests, cube specimens  $100 \times 100 \times 100$  mm in size are installed with one of the selected faces on the lower base plate of the testing machine, centrally relative to its longitudinal axis, using the risks applied to the plate of the testing machine. The sample is loaded to failure at a constant rate of load increase (0.6 ± 0.2) MPa/s.

When testing for tensile bending, a prism specimen  $100 \times 100 \times 100$  mm in size is installed in a testing machine according to [51] and loaded to failure at a constant rate of load increase ( $0.05 \pm 0.01$ ) MPa/s.

When testing for axial tension, a prism specimen with a size of  $100 \times 100 \times 400$  is fixed in a tensile testing machine and loaded to failure at a constant rate of load increase  $(0.05 \pm 0.01)$  MPa/s.

The determination of the axial compressive strength is carried out in accordance with the requirements of GOST 24452 "Concretes. Methods of prismatic, compressive strength, modulus of elasticity and Poisson's ratio determination".

Deformations under axial compression and tension were measured using a chain of strain gauges with a base of 50 mm and dial gauges with a division value of 0.001 mm.

The compressive strength of concrete  $R_{b,cub}$ , MPa, was calculated with an accuracy of 0.1 MPa using the formula (2):

$$R_{b,cub} = \alpha \, \frac{F}{A} \tag{2}$$

The tensile strength of concrete in bending  $R_{btb}$ , MPa, is calculated with an accuracy of 0.1 MPa by the formula (3)

$$R_{btb} = \delta \frac{F \cdot l}{a \cdot b^2} \tag{3}$$

The axial tensile strength of concrete  $R_{bt}$ , MPa, is calculated with an accuracy of 0.1 MPa by the formula (4)

$$R_{bt} = \beta \frac{F}{A} \tag{4}$$

where *F* is the breaking load, N; *A* is the area of the working section of the sample, mm; *a*, *b*, *l* are the width, height of the cross section of the prism, and the distance between the

supports, respectively, when testing specimens for tensile bending, mm;  $\alpha = 0.95$ ,  $\delta = 0.92$ ,  $\beta = 0.92$  are scale factors for converting concrete strength to concrete strength in samples of basic size and shape.

The prism strength Pp is calculated for each sample using the formula (5)

$$R_b = \frac{P_p}{A_a} \tag{5}$$

where Pp is the breaking load measured on the scale of the press force meter and  $A_a$  is the average value of the cross-sectional area of the sample.

#### 3. Results

After 28 days of curing, laboratory tests of experimental compositions for strength and deformation characteristics were carried out. Strength tests were carried out on three samples of each mixture, and then the average value was used for the purposes of analysis and discussion.

Table 10 shows the strength and deformation characteristics of the samples of the control composition.

Table 10. Strength and deformation characteristics of concrete samples of the control composition.

Indicator Title	Value
Compressive strength, MPa, <i>R</i> <sub>b.cub</sub>	48.9
Flexural tensile strength, MPa, $R_{btb}$	5.8
Axial compressive strength, MPa, $R_b$	36.8
Axial tensile strength, MPa, $R_{bt}$	3.3
Limit deformations under axial compression, mm/m $ imes 10^{-3}$ , $\varepsilon_b$	2.20
Limit deformations in axial tension, mm/m $\times 10^{-4}$ , $\varepsilon_{ht}$	1.32
Modulus of elasticity, MPa, E	33.7

#### 3.1. Compressive Strength

Figure 2 shows the dependence of the change in the compressive strength of concrete on the amount of eggshell powder.

The dependence of the compressive strength of concrete on the amount of eggshell powder is well approximated by a 4th degree polynomial with a determination coefficient  $R^2 = 0.98$ 

$$R_{b,cub} = 48.8 + 0.248 * x + 0.06301 * x^2 - 0.006684 * x^3 + 0.0001303 * x^4$$
  

$$R^2 = 0.98$$
(6)

As shown in Figure 2, the compressive strength of concrete when replacing 5%, 10%, 15%, 20%, 25%, and 30% cement with eggshell powder was 50.5 MPa, 53.2 MPa, 49.5 MPa, 47.3 MPa, 40.5 MPa, and 38.1 MPa, respectively. It can also be seen that the maximum compressive strength of concrete was recorded when replacing 10% of cement with eggshell powder. With a further increase in the dosage of the powder, the strength values decrease. A particularly sharp drop is observed at the points of 25% and 30%.

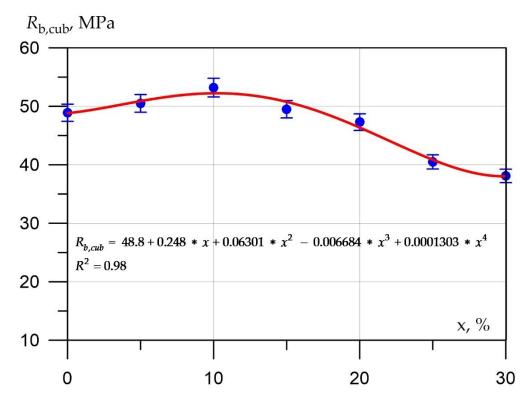


Figure 2. Dependence of the compressive strength of concrete on the amount of eggshell powder.

## 3.2. Flexural Tensile Strength

Figure 3 shows the dependence of the tensile strength in bending of concrete on the content of eggshell powder.

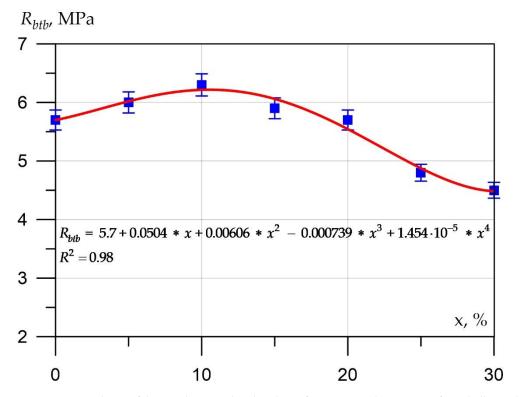


Figure 3. Dependence of the tensile strength in bending of concrete on the amount of eggshell powder.

The dependence of the concrete flexural tensile strength on the amount of eggshell powder is also well approximated by a 4th degree polynomial with a determination coefficient of 0.98

$$R_{btb} = 5.7 + 0.0504 * x + 0.00606 * x^2 - 0.000739 * x^3 + 1.454 \cdot 10^{-5} * x^4$$
  

$$R^2 = 0.98$$
(7)

As shown in Figure 3, the flexural tensile strength of concrete when replacing 5%, 10%, 15%, 20%, 25%, and 30% cement with eggshell powder, respectively, was 6 MPa, 6.3 MPa, 5.9 MPa, 5.7 MPa, 4.8 MPa, and 4.5 MPa. The maximum flexural tensile strength of concrete was observed when replacing 10% cement with eggshell powder. The trend of decreasing tensile strength in bending is similar to that for compressive strength.

#### 3.3. Axial Compressive Strength

Figure 4 shows the dependence of the axial compressive strength of concrete on the amount of eggshell powder.

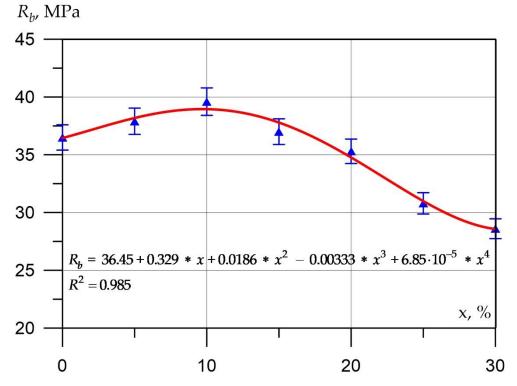


Figure 4. Dependence of the axial compressive strength of concrete on the amount of eggshell powder.

The dependence of the axial compressive strength of concrete on the amount of eggshell powder is also well approximated by a 4th degree polynomial with a determination coefficient of 0.985

$$R_b = 36.45 + 0.329 * x + 0.0186 * x^2 - 0.00333 * x^3 + 6.85 \cdot 10^{-5} * x^4$$
  

$$R^2 = 0.985$$
(8)

As can be seen in Figure 4, the axial compressive strength of concrete when replacing 5%, 10%, 15%, 20%, 25%, and 30% cement with eggshell powder, respectively, was 37.9 MPa, 39.6 MPa, 37.0 MPa, 35.3 MPa, 30.8 MPa, and 28.6 MPa. The axial compressive strength reached its maximum value when replacing 10% cement with eggshell powder. The trend of decreasing axial compressive strength is similar for both compressive and flexural tensile strengths.

### 3.4. Axial Tensile Strength

Figure 5 shows the dependence of the axial tensile strength of concrete on the amount of eggshell powder.

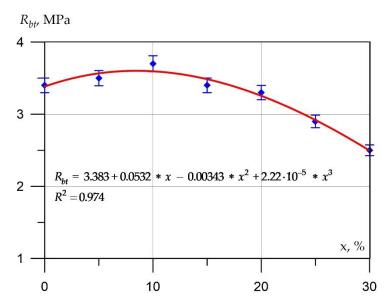


Figure 5. Dependence of the axial tensile strength of concrete on the amount of eggshell powder.

The dependence of the axial tensile strength of concrete on the amount of eggshell powder is well approximated by a 3rd degree polynomial with a determination coefficient of 0.974

$$R_{bt} = 3.383 + 0.0532 * x - 0.00343 * x^2 + 2.22 \cdot 10^{-5} * x^3$$

$$R^2 = 0.974$$
(9)

The axial tensile strength of concrete when replacing 5%, 10%, 15%, 20%, 25%, and 30% cement with eggshell powder, respectively, was 3.5 MPa, 3.7 MPa, 3.4 MPa, 3.3 MPa, 2.9 MPa, and 2.5 MPa (Figure 5). The maximum axial tensile strength of concrete was observed in samples in which 10% of the cement was replaced by eggshell powder.

#### 3.5. Elastic Modulus

Figure 6 shows the dependence of the modulus of elasticity of concrete on the amount of eggshell powder.

The dependence of the modulus of elasticity of concrete on the amount of eggshell powder is also well approximated by a 3rd degree polynomial with a determination coefficient of 0.977

$$E = 33.5 + 0.406 * x - 0.03 * x^{2} + 0.000289 * x^{3}$$
  

$$R^{2} = 0.977$$
(10)

Figure 6 shows that the values of the modulus of elasticity of concrete when replacing 5%, 10%, 15%, 20%, 25%, and 30% of cement with eggshell powder, respectively, were 34.5 MPa, 35.1 MPa, 33.7 MPa, 32.7 MPa, 28.7 MPa, and 26.8 MPa. The maximum modulus of elasticity was recorded when replacing 10% of the cement with eggshell powder.

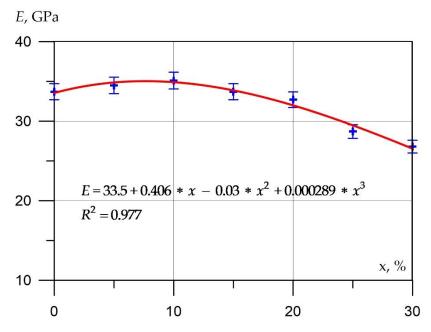
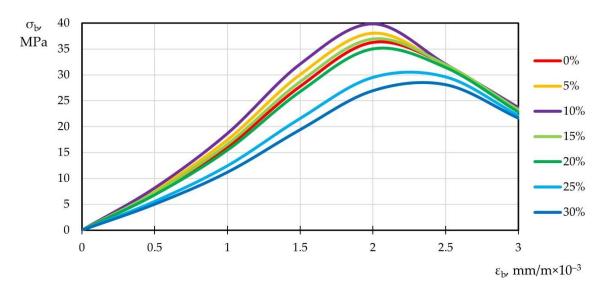
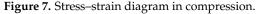


Figure 6. The dependence of the modulus of elasticity of concrete on the amount of eggshell powder.

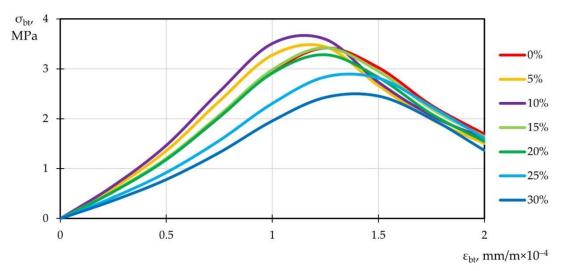
#### 3.6. Stress–Strain Diagrams

To assess the effect of the dosage of eggshell powder on the deformation characteristics of concrete, the diagrams of compression " $\varepsilon_{b}$ - $\sigma_{b}$ " and tension " $\varepsilon_{bt}$ - $\sigma_{bt}$ " were constructed. Graphic dependences of stress–strain are presented in Figures 7 and 8.





Obviously, the concrete composition with 10% eggshell powder has the best properties. The peak of its stress–strain diagram is shifted upward and to the left relative to others, which shows its high strength characteristics and low deformation characteristics. The worst composition in terms of deformability is the composition with a dosage of 30% of the powder, which is quite understandable due to the oversaturation of concrete with eggshell powder over a rational amount. Thus, we have a non-optimal dosage that harms concrete, and on the stress–strain diagram, its peak shifts down and to the right relative to other dosages. It should be noted that the remaining peaks of the corresponding diagrams are located between the indicated best and worst dosages of ESP in approximately the same sequence as in the identified dependencies above. The same description applies to the stress–strain diagram in tension. Obviously, the best composition in terms of deformability



was also the composition of concrete with a 10% dosage of eggshell powder. Thus, the nature of the deformability of the analyzed compositions also confirmed the identified rational composition of concrete.

Figure 8. Tensile stress-strain diagram.

#### 3.7. Microstructural Analysis

Figures 9–14 show the microstructure of cement stone with different dosages of eggshell powder.

In the presented photographs of the microstructure, one can notice that there are inconsistencies in the interaction between the phases of the materials (Figures 9 and 11–14). Moreover, gaps, cracks, and voids are observed in cement stone samples, which is due to the method of obtaining samples for SEM analysis. These samples were taken from pieces of the destroyed concrete composite. As for the inconsistencies in the interaction of various phases, it should be noted that, for samples with a dosage of 10% (Figure 10), there is a certain greater consistency in the interaction between the phases of materials in comparison with other samples (Figures 9 and 11–14). Thus, it can be concluded that replacing part of the cement with 10% eggshell powder improves the interfacial interactions between the various constituents of the concrete.

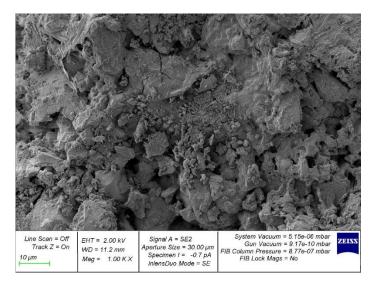


Figure 9. Sample with partial replacement of cement with eggshell powder at 5%.

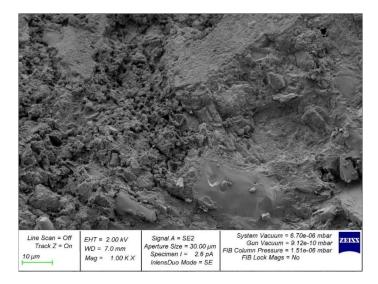


Figure 10. Sample with partial replacement of cement with eggshell powder at 10%.

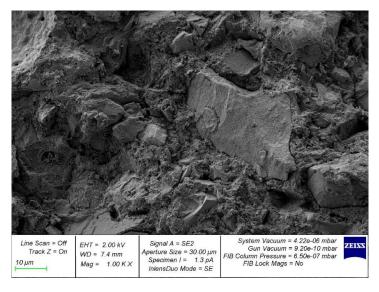


Figure 11. Sample with partial replacement of cement with eggshell powder at 15%.

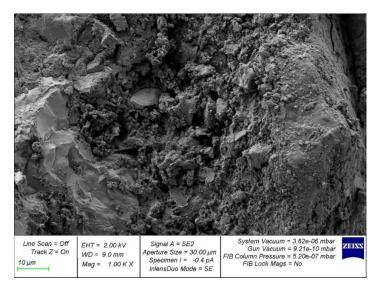


Figure 12. Sample with partial replacement of cement with eggshell powder at 20%.

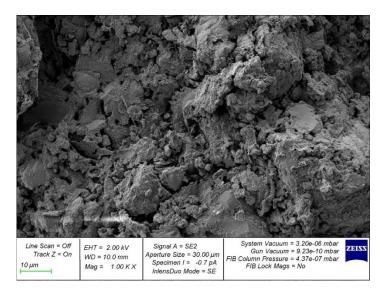


Figure 13. Sample with partial replacement of cement with eggshell powder at 25%.

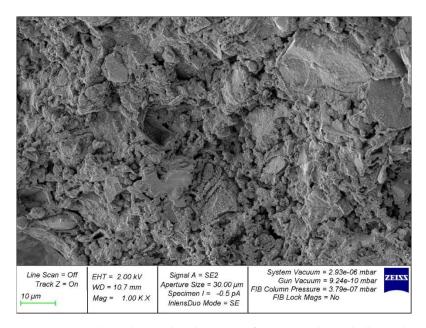


Figure 14. Sample with partial replacement of cement with eggshell powder at 30%.

Eggshells are primarily composed of calcium which, when added to a cement system, reacts extensively with the cement to form a larger network of calcium silicate hydrate (CSH) minerals. This increase in the amount of CSH gels causes the microstructure of the cement slurry to become denser. However, adding more than 10% eggshell powder as a binder replacement results in a stiffer mixture due to the increased limestone content in the mixture, which absorbs all free water molecules by absorption.

The addition of nano-modifying eggshell powder makes it possible to achieve an effect, not only at the macro level, but also at the micro level due to the redistribution of the structure and the occurrence of the phenomenon of nano changes in this structure. That is, the redistribution of crystallization centers occurs at the nano level, which provides a denser and more compact packing of particles, and additionally represents a resource in terms of cement hydration to achieve the best performance both in terms of strength and packing density of microstructure particles at the macro level, micro level, and including the nano level.

#### 4. Discussion

For a more complete analysis of the resulting concrete, changes in strength and deformation characteristics were determined, expressed as percentages (Table 11).

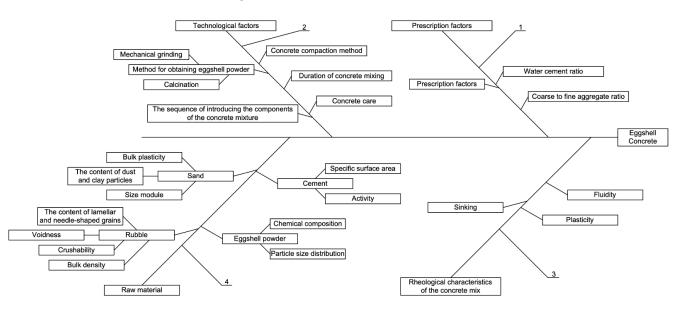
**Table 11.** Change in strength and deformation characteristics of concrete samples depending on the dosage of eggshell powder ( $\Delta$ ).

Concrete Characteristics	$\Delta\%$ for Content of Eggshell Powder in the Amount							
	0	5	10	15	20	25	30	
$R_{b, cub}$ , MPa	0	+3	+9	+1	-3	-17	-22	
<i>R<sub>btb</sub></i> , MPa	0	+5	+11	+4	0	-16	-21	
$R_b$ , MPa	0	+4	+8	+1	-3	-16	-22	
<i>R<sub>bt</sub></i> , MPa	0	+3	+9	0	-3	-15	-26	
$\epsilon_{bR}$ , mm/m $ imes 10^{-3}$	0	-3	-5	-1	0	7	20	
$\varepsilon_{btR}$ , mm/m $\times 10^{-4}$	0	-8	-10	-2	-2	8	9	
E, GPa	0	+2	+4	0	-3	-15	-20	

According to the data on changes in the strength and deformation characteristics of concretes modified with ESP, presented in Table 11, it was found that the optimal dosage of eggshell powder, introduced instead of part of the cement, is a dosage of 10%. This recipe solution achieves an increase in strength characteristics up to 11% and a decrease in deformations during axial compression and tension by 10%.

These results are in good agreement with studies [16,19]. In this case, the optimal dosage of eggshell powder as a replacement for part of the cement is 10%, as in [13,16,19,21], but the performance gains achieved in these works differ. The optimal dosages of ESP according to [10,13,16,18,20–22] are from 5 to 15%. However, sometimes there are also more optimal amounts of added ESP, for example, 20% [15]. There are quite a few studies in which the strength characteristics of samples modified with eggshell powder in the optimal dosage, replacing part of the cement, do not reach the values of control samples (without ESP) [16,18,20].

The most complete range of factors affecting the final physical and mechanical properties of concrete modified with the ESP additive is shown in Figure 15 in the form of an Ishikawa diagram.



**Figure 15.** Causal diagram of factors affecting the physical and mechanical properties of concrete modified with ESP additive.

The factors that have the greatest influence on the characteristics of concrete, nanomodified ESP, were divided into four main groups: prescription factors (branch 1), technological factors (2), rheological characteristics of the concrete mix (3), raw materials (4). Within each branch, subfactors were identified that specify the main factors. Thus, we have grouped and analyzed the most important and significant factors influencing the quality of concrete with the addition of eggshell powder.

The use of eggshell powder in an amount of 10% instead of a part of cement makes it possible to obtain concrete without loss of strength characteristics. Thus, the use of this additive makes it possible to reduce the consumption of cement, which will lead to a reduction in the cost of concrete, as well as solve the environmental problem of eggshell waste disposal.

#### 5. Conclusions

- (1) Concrete has been obtained, the performance of which is improved relative to standard concrete by modifying it with eggshell powder, for which the optimal dosage is determined. Concrete with improved characteristics has been obtained, which makes it possible to achieve an improvement in the environmental situation due to the competent rational disposal of accumulated waste in the form of eggshells.
- (2) The most effective was the replacement of part of the cement with eggshell powder in an amount of 10%.
- (3) The maximum increase in compressive strength of concrete nano-modified with eggshell powder was 9%, tensile strength in bending—11%, axial compressive strength—8%, axial tensile strength—9%, elastic modulus—4% in comparison with control samples without ESP. At the amount of ESP of 25% and 30%, a sharp drop in strength characteristics by 21–26% is observed in comparison with the strength characteristics of the control composition. The value of the modulus of elasticity decreased to 20%.
- (4) The most significant reductions in deformations in axial compression and tension, in comparison with the control values, ranged from 5% to 10%. The maximum values of deformations under axial compression and tension were recorded for concrete samples with the addition of ESP in the amount of 30%. Deformations under axial compression increased by 20%, and deformations under axial tension increased by 9%.
- (5) A study of the microstructure of composites nano-modified with eggshell powder was carried out, with an analysis of the changes occurring in such a microstructure due to nano-modification, confirming the obtained improvement in performance and the optimal dosage of ESP.

Research prospects are seen in the continuation of the study of the phenomenon of concrete nano-modification and, first, agricultural waste of a different type with a different content of various useful elements to improve the properties of concrete and at the same time improve the state of the environment.

The concrete obtained as a result of this work is an industrial sample. Authors plan its subsequent implementation in real production, and plan to reach several agreements with industrial partners to manufacture reinforced concrete and other reinforced products based on new improved concrete, which will be carried out by us in subsequent studies.

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