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Performance of Cement Composites with Partial Replacement with Organic Aggregate from Waste Coconut Shell

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Abstract: The properties of cement composites with a partial replacement of sand with an organic aggregate from coconut shell are investigated. Due to the fact that the endocarp of coconut shell increases its volume up to 60–70% when swelling in water, which is many times greater than the volumetric deformation of the swelling of the cement matrix, the possibility of obtaining a cement composition capable of withstanding alternating deformations of wetting and drying was investigated in this work. When replacing 50% of the sand with coarse coconut aggregate of the fraction 5–20 mm, starting from the 5th–10th cycle of wetting–drying, progressive expansion and cracking of 28-day samples took place. When using a fine coconut aggregate fraction < 2.5 mm and replacing sand from 2 to 15%, a slight accumulated expansion was observed only in the first 4–5 test cycles, then accumulated shrinkage followed. A gradual decrease in the average weight of the samples from cycle to cycle was established. The loss of mass of the samples increases with the increase in the percentage of replacement of sand with coconut aggregate. Partial replacement of sand with fine coconut aggregate from 2 to 15% by weight reduces the bending and compressive strength of cement mortar from 14 to 40%.

Keywords: concrete; cement; coconut shell; coarse aggregate; fine aggregate; solid waste

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1. Introduction

The volume of global construction increases annually, and to match the growth of the construction sector, a huge amount of concrete is produced worldwide. Concrete production quickly depletes natural resources, sand and crushed stone, which are used as fine and coarse aggregates in concrete [1]. For this reason, the idea of completely or partially replacing aggregates in concrete with solid waste is becoming increasingly relevant [2,3], both from the point of view of preserving natural resources and because of the possibility of recycling waste, the storage of which often requires the use of large areas.

Alternative aggregates for concrete include industrial [4,5] and agricultural waste [6]. Industrial waste includes fly ash [7], blast furnace slag [8], microsilica [9], quarry rock dust [10], construction and demolition waste [11], waste foundry sand [12], old tires [13], asbestos waste [14], etc. Agricultural waste includes date seed [15], rice husk ash [16], rubber seed [17], oil palm shell [18], maize silage and beet pulp [19], rice straw ash [20], cockle shell [21] and coconut shell. However, the use of such waste should not reduce the quality of concrete.

A literature review shows that among agro-wastes, coconut shells generated during the production of coconut oil are the most suitable option for partial replacement of aggregates

in the production of structural concrete due to their hard structure [22]. Coconut palms are grown in 93 countries worldwide, of which Southeast Asian countries (Indonesia, Philippines, and India) account for almost 75% of the world's total production [23]. More than fifty million tons of coconuts are produced annually [24]. After obtaining coconut chips, the hard coconut shell is usually discarded, and this constitutes a large percentage of household waste in many countries [25]. Standard landfill processes for coconut shells result in the production of greenhouse gases, primarily methane, when the coconut shells decompose as organic matter and pollute water and soil [26]. Using coconut shell waste instead of conventional concrete aggregates has the potential to solve the problem of bio-waste disposal and environmental pollution [27].

Coarse aggregate from coconut shell changes many properties of both the concrete mix and concrete. Due to the roughness of aggregate grains, the mobility of concrete mix decreases [28], but the adhesion of aggregate to the cement–sand matrix improves [29]. The study of thermophysical properties of concrete with coarse aggregate from coconut shows that increasing the percentage of aggregate from 0% to 40% in concrete helps to reduce thermal conductivity by three times [30]. It is important to note that the use of coconut shell aggregate should not reduce the strength characteristics of concrete. In [31], the authors establish that the use of up to 10% coconut shell instead of natural coarse aggregate is justified and does not lead to a significant loss in strength characteristics. Moreover, replacing 5% of natural aggregate with coconut shell increases the compressive strength of concrete by 4.1%. Analysis of the microstructure of lightweight concrete shows that this occurs due to the penetration of cement paste into the pores of the shell, thereby increasing the adhesion of this aggregate to the cement–sand matrix [31]. During bending tests, it is recorded that with an increase in the coconut shell content to 10% of the natural aggregate weight, the bending strength increases [32]. When 20% of the coarse aggregate is replaced by coconut shell, the strength at 7 and 14 days slightly increases compared to the control sample, but at 28 days the strength decreases [32]. A study of the behavior of concrete with coarse coconut shell aggregate at elevated temperatures shows that the loss in compressive strength after 30 min of exposure to a temperature of 300 °C is 12.4% and 20.5% for a coconut shell content of 10% and 20%, respectively [33]. Based on the obtained results, the optimum content of coconut shell is 10% of the natural coarse aggregate mass.

Fine aggregate made from crushed coconut shell is used as a replacement for natural sand in concrete. It has been found that the workability of concrete mix increases with the increase in the percentage of sand replacement with coconut shell [34]. The compressive strength obtained with 10%, 20% and 30% replacement of sand with coconut shell is 91%, 80% and 72% of the total compressive strength of M20 concrete [34]. The flexural strength of the control sample and concrete samples with 10%, 20% and 30% coconut shell is 3.063 MPa, 3.045 MPa, 2.97 MPa and 2.912 MPa, respectively [35]. In general, all concretes with coconut shells have flexural strength values similar to those of the control sample.

The study of the microstructure of concrete with coconut shell aggregate [29] obtained using scanning electron microscopy (Figure 1a,b) show that the presence of a rough surface of the coconut shell helps it to adhere to concrete, and when the shell is added to concrete, calcium silicate hydrate is formed (Figure 1c), which contributes to the binding properties of concrete.

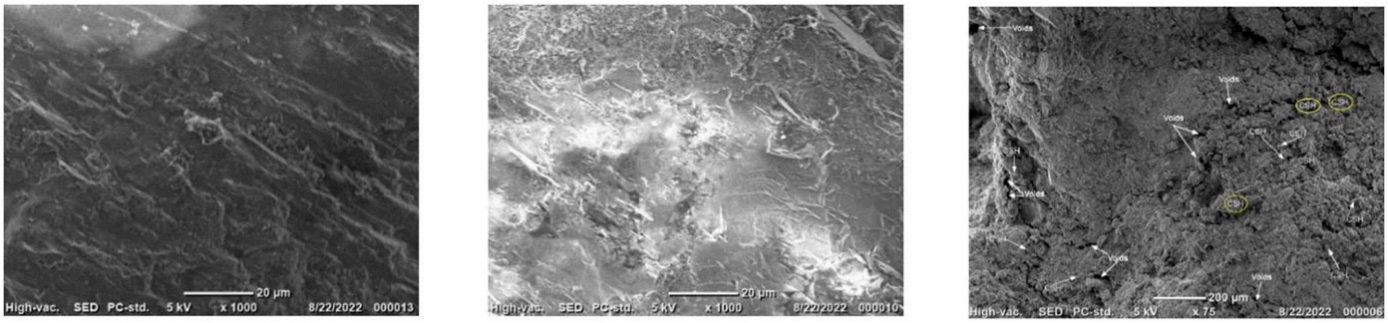


Figure 1. Scanning electron microscopy images showing (**a**) external and (**b**) internal surfaces of coconut shell; (**c**) concrete with coconut shell aggregate (white arrows show voids, yellow circles show calcium hydrosilicates) [29].

Despite numerous studies of coconut shells as a partial replacement for concrete aggregates, there are no studies on the compatibility of deformations of such aggregates and cement matrix, which does not allow us to describe the durability of concrete using coconut shells as aggregates in concrete.

This work aims to assess the compatibility of deformations in a cement–sand matrix and coconut shell aggregates in fine-grained concrete.

2. Results and Discussion

2.1. Physical and Mechanical Properties of Coconut Shell

As a result of sifting the aggregate through a set of sieves, five fractions of coconut shell were obtained, which are presented in Figure 2.

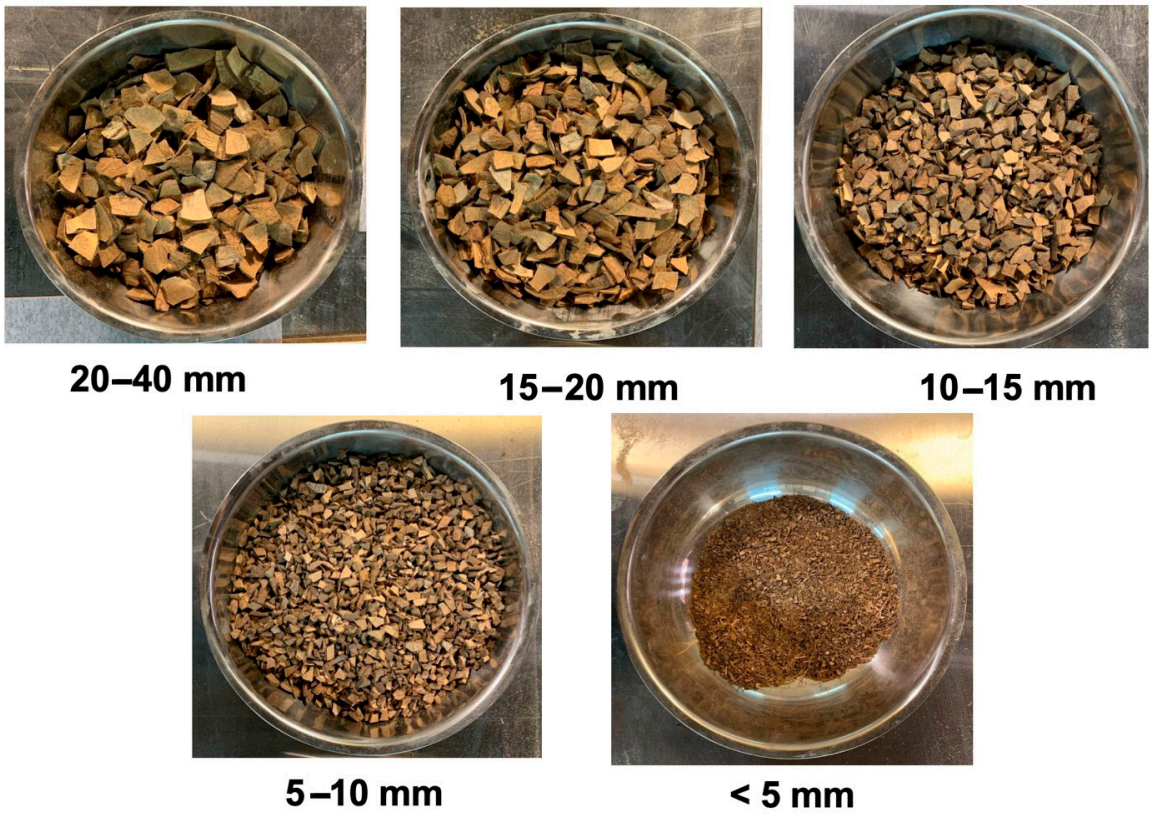


Figure 2. Grain size distribution of coconut aggregate for concrete.

Table 1 shows the grain size composition of the aggregate.

Table 1. Grain composition of coconut aggregate.

Fraction Size, mm	%
<5	4
5–10	29.7
10–15	20.4
15–20	26.3
20–40	19.6

The analysis of the data presented in Table 1 allows us to conclude that the largest content is the fraction from 5 to 10 mm in size, the smallest residue on the sieve is the fraction less than 5 mm. The obtained grain composition allows us to select the optimal composition of the fraction mixture to achieve compact packing of all concrete components.

The bulk density of the coconut shell aggregate is 510 kg/m^3.

The true density of the powder fraction of the filler is 1667 kg/m^3.

The average density is 1060 kg/m^3.

The compressive strength in a composite cylinder of the 5–20 mm fraction is 1.67 MPa, which corresponds to the P75 grade for expanded clay following Russian State Standard GOST 32496-2013 "Fillers porous for light concrete. Specifications" [36].

Water absorption by weight of the 5–20 mm fraction is 29.9%. The graph of the dependence of aggregate water absorption on the time of exposure to water is shown in Figure 3, from which it is evident that about 60% of the total amount of water is absorbed by the endocarp during the first hour, after which the rate of absorption drops sharply and complete saturation of the samples with water occurs after 35–40 h.

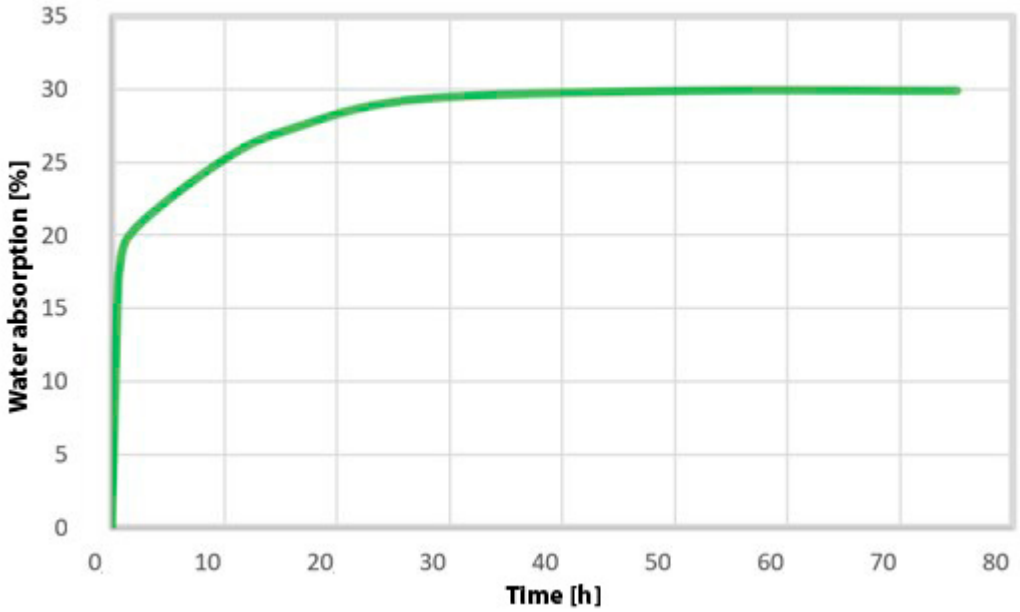


Figure 3. Water absorption of coconut shell endocarp depending on time.

Water absorption by weight of the powder fraction after exposure to water for 20 days is 33.7%. Increased water absorption by the powder fraction compared to crushed shell is explained by a higher specific surface area and, accordingly, a larger proportion of open surface pores.

Water saturation under vacuum with fivefold repetition is 30.7%, which slightly exceeds the water absorption obtained under atmospheric pressure. Considering the error

of this type of testing associated with the quality of surface water removal from particles of water-saturated material, these two indicators can be considered practically the same and, therefore, water absorption by volume, equal to 31.7%, can be equated with a certain approximation to open porosity.

The true porosity of the coconut shell endocarp is calculated to be 36.4%.

Conventionally closed porosity is 6.5%.

When studying the swelling of coconut shell powder after 20 days, the sample volume on the graduated cylinder scale is 202 mL. The increase in the powder volume against the initial volume depending on the swelling time in water is shown in Figure 4.

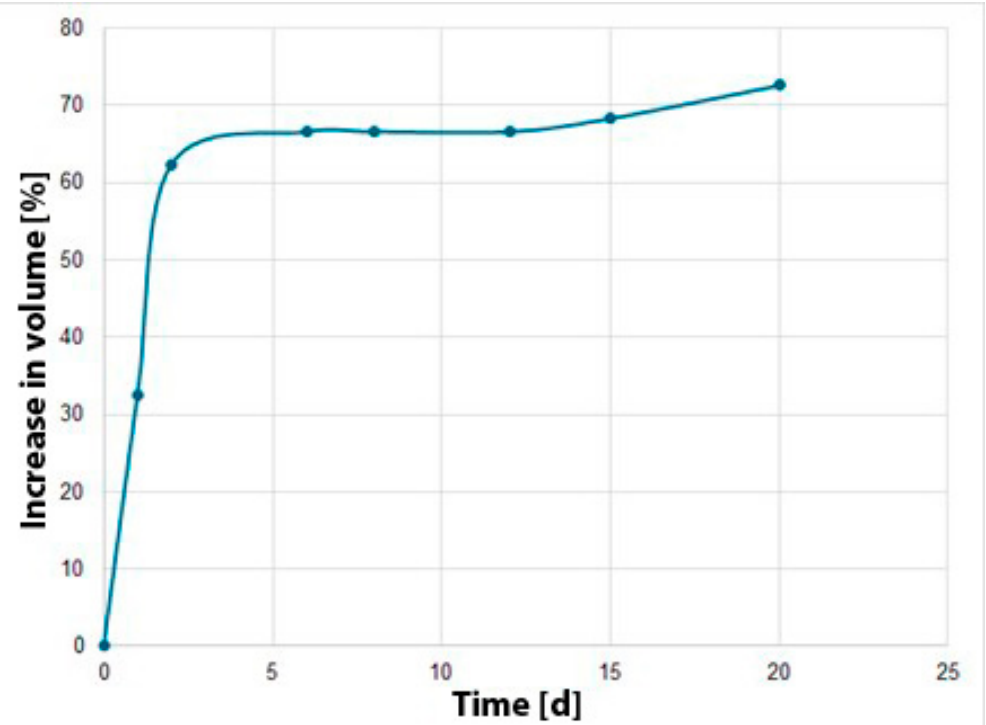


Figure 4. Increase in powder volume during swelling in water depending on time.

As can be seen from Figure 4, a significant increase in the volume of the powder fraction to 60% occurred in the first two days of the material's stay in water. Subsequently, the swelling process slowed sharply.

In the case of the crushed fraction, the results were different. The initial volume of dry material was 205 mL (Figure 5a). One day after soaking the material in water, its total volume increased to 230 mL and then did not change over the course of 21 days of observation (Figure 5b).

The volume expansion of this fraction is only 12.2%, which is 5–6 times less than the volume expansion of the powder fraction. This result is difficult to explain by water absorption, since the latter has close values for these materials. Perhaps this is due to the anisotropy of the coconut shell, which manifests itself mainly in the case of large particles. In powder, the particles, orienting randomly in different directions, level out the anisotropy.

Thus, the volume expansion of the coconut shell endocarp during swelling in water is at least 10 times greater than the volume deformation of swelling of the cement matrix, which is no more than 1%.

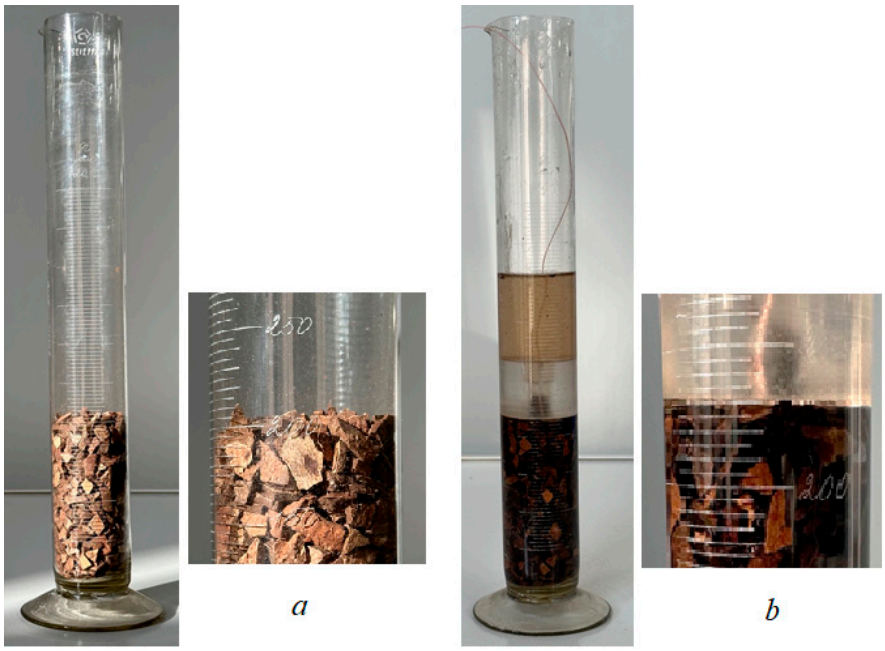


Figure 5. Testing crushed fraction of organic aggregate for volume increase upon swelling in water: (**a**) is volume at the beginning of the test and (**b**) is volume at end of test.

2.2. Evaluation of Compatibility of Deformations of Cement–Sand Matrix and Coconut Aggregate

Figures 6 and 7 show graphs of the accumulation of relative deformation by samples as the number of water absorption–drying cycles increases. The average value of deformation between the maximum expansion during water absorption and the minimum value after drying in each half-cycle is taken as the value of accumulated deformation. Figure 5 shows the results of testing samples with a large fraction of coconut aggregate (5–20 mm).

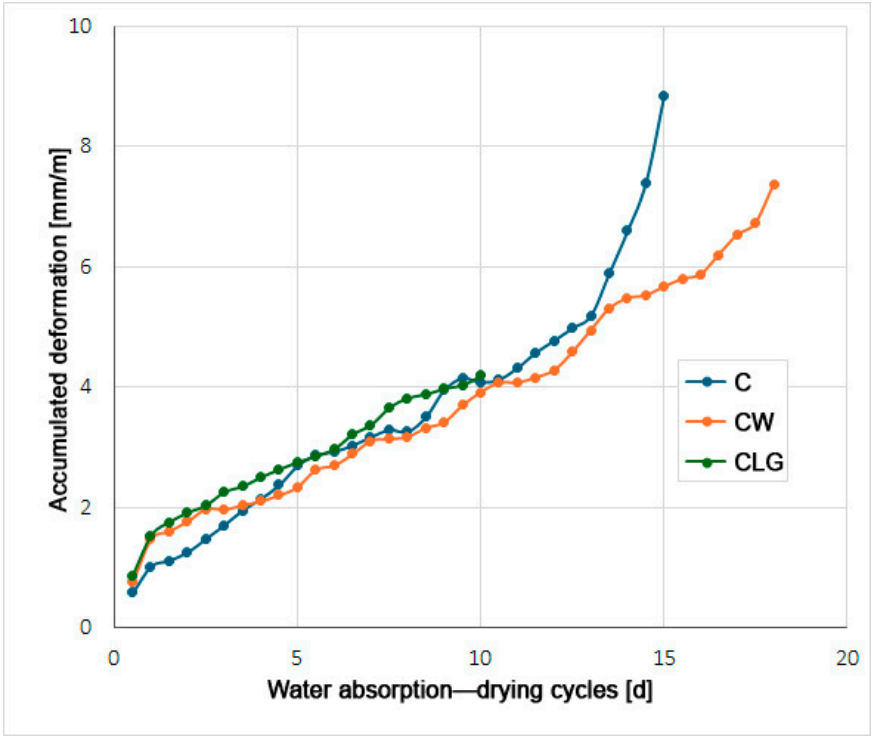


Figure 6. Cumulative relative expansion of samples with a large fraction of coconut aggregate: C is without aggregate pre-treatment; CW is with water-saturated aggregate; CLG is with mineralized aggregate.

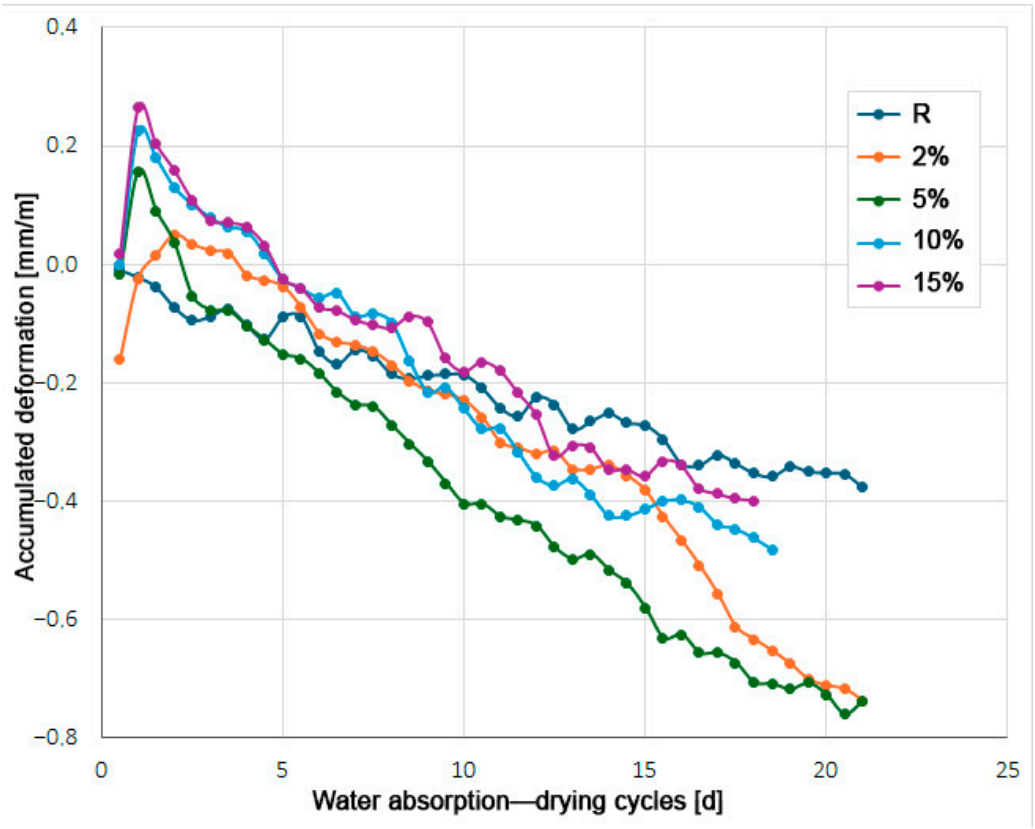


Figure 7. Cumulative relative deformation of samples when replacing 2, 5, 10 and 15% of the volume of standard sand with fine powder fraction of coconut aggregate.

As can be seen from Figure 6, the accumulation of expansion deformation occurred approximately equally for all samples until their cracking and partial destruction, when the growth of deformation was observed due to the opening of cracks. Disruption of continuity began from the 5th–10th cycle. Before that, the deformation developed approximately linearly depending on the number of cycles.

The closeness of the curves indicates the insignificant effectiveness of the measure used—saturation of the coconut aggregate with water and its mineralization. When using preliminary saturation of the organic aggregate with water, it was assumed that the cell volume in the matrix, specified by the swollen grain of the organic aggregate, would remain greater than the volume of the grain itself in subsequent cycles. That is, the grain would peel off from the cell walls and its swelling and shrinkage deformations would not affect the matrix. The experimental results show that this is not the case. The expansion of the samples occurs almost equally, both in the case of dry and in the case of water-saturated organic aggregate. Apparently, the deformations of these components occur together. The lack of effect from preliminary saturation of coconut aggregate with water is explained by the fact that during the first drying, the water-saturated aggregate is dehydrated and then behaves in the same way as the initially dry one. Mineralization of the coconut shell endocarp probably does not provide sufficient impermeability of the grains.

We explain the formation of cracks and destruction of samples in the experiments described above by the use of too large a fraction of the aggregate and its excess content. Therefore, in further tests, a powder fraction of coconut aggregate is used as a replacement for standard sand from 2 to 15% by volume (Figure 7).

In this case, expansion accumulated in the first 4–5 test cycles, followed by shrinkage accumulation. The initial accumulated expansion increases with the percentage of natural sand replaced by coconut fine aggregate. Since the control concrete accumulated only

shrinkage, the coconut aggregate should be considered responsible for the expansion of the samples. After the initial expansion, shrinkage deformation accumulates in all cases. The final shrinkage value of the test samples, despite the initial expansion, is higher than that of the control samples, so it can be assumed that the organic aggregate also contributes to the shrinkage deformation as a result of its own gradual shrinkage. On the other hand, it is possible that, unlike natural sand, the more easily compressible coconut aggregate provides less resistance to the shrinkage of the cement stone.

Figures 8 and 9 show the accumulated mass loss of the samples in relation to the initial value, obtained as the average between the maximum and minimum values in each half-cycle.

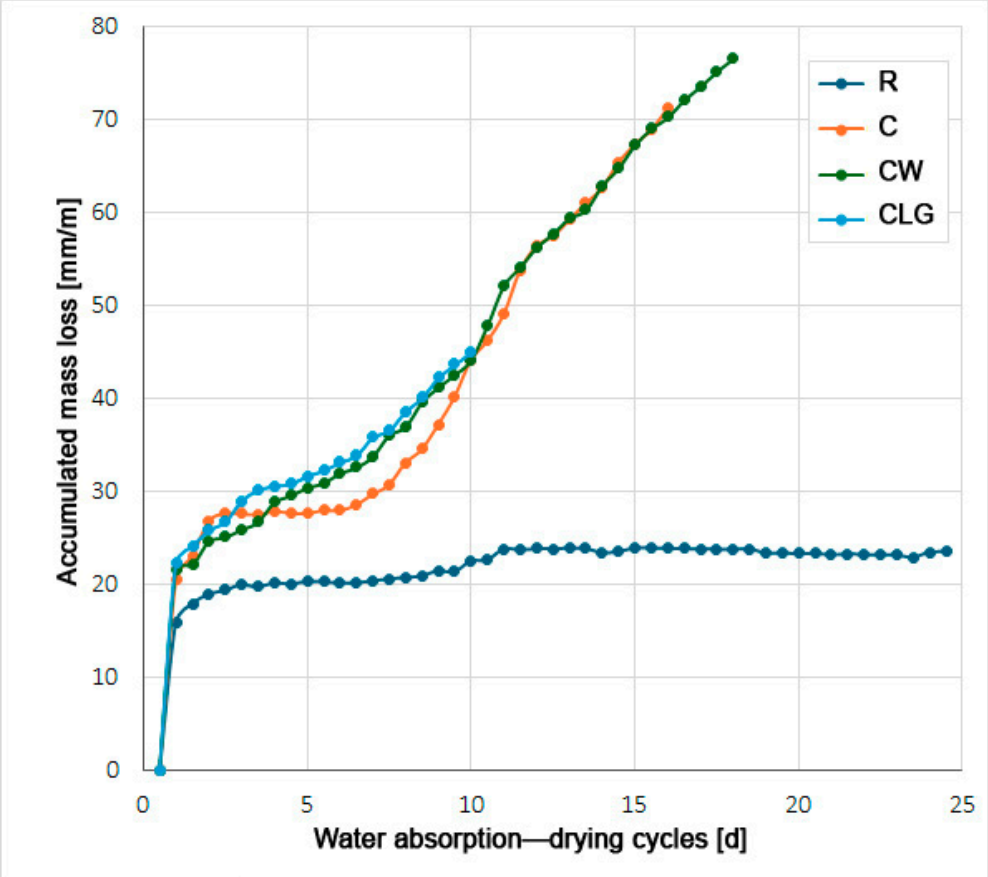


Figure 8. Cumulative mass loss of samples with coarse coconut aggregate: C is without pre-treatment of coconut aggregate; CW is with water-saturated coconut aggregate; CLG is with mineralized coconut aggregate.

The decrease in the mass of the samples is mainly due to the evaporation of water during drying. From Figure 8, it is evident that the amount of water lost by the control composition is significantly less than that of the samples with coconut aggregate and changes little after the third cycle. This suggests that moisture accumulation occurs only in the first three cycles, after which the amount of absorbed and released water in each subsequent cycle is approximately the same. In the presence of coconut aggregate, the accumulated mass loss of the C, CW and CLG samples increases from cycle to cycle. Consequently, with each subsequent cycle the volume of space filled with absorbed water increases, which is the cause and effect of swelling of the coconut shell endocarp, causing expansion and cracking of the cement matrix.

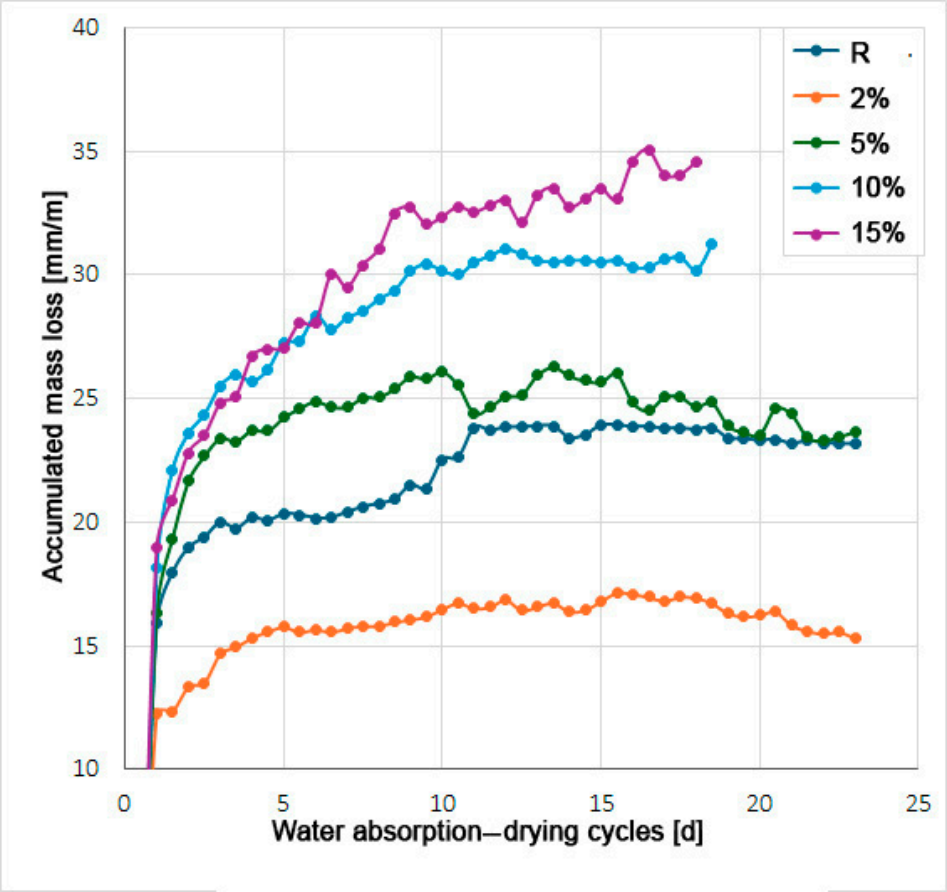


Figure 9. Accumulated loss of mass in samples with fine coconut aggregate with the aggregate content at 2, 5, 10 and 15% of the total aggregate volume.

The curves of accumulated water loss for the C, CW and CLG compositions are located very close to each other, which also confirms the low efficiency of mineralization of the organic aggregate.

Figure 9 shows the results of changing the mass of samples with a powder fraction of coconut aggregate at different contents—from 2 to 15% of the total volume of aggregates.

The accumulated mass loss of the samples with fine organic aggregate gradually increases, but after about 10–12 cycles it reaches its maximum value and begins to decrease. This is consistent with the development of accumulated deformation, passing from expansion to shrinkage. The accumulation of both expansion deformation and mass loss of the samples is associated with an increase in the pore space filled with water from cycle to cycle during saturation of the endocarp as a result of its swelling and developing cracking of the cement matrix. During subsequent shrinkage of the organic aggregate, the volume of pores available for water decreases and shrinkage occurs.

The higher the content of coconut shell endocarp in the fine aggregate, the greater the mass loss, and, consequently, the greater the water absorption capacity of the material.

Judging by the accumulated mass loss, the water absorption capacity of the compositions with coarse and fine coconut aggregate differs by approximately two times. In the first case, the mass loss by the end of the experiment reaches 70–80 g per sample; in the second case, it does not exceed 35 g. However, the volumes of lost water should be compared in relation to the volume of coconut aggregate in samples of different compositions (Table 2).

Table 2. Ratio of accumulated volumes of lost water and volumes of coconut aggregate contained in samples of different compositions.

Name of the Indicator	Fraction 5–20 mm	Fine Coconut Aggregate with Aggregate Content in the Mixture, %			
		2	5	10	15
Volume of coconut aggregate considering expansion during swelling V_c, mL/sample	79.4	4.7	11.2	22.8	34.0
Volume of lost water V_w, mL/sample	75	17	26	31	35
V_c/V_w ratio	0.94	3.62	2.32	1.36	1.03

As can be seen from Table 2, the volume of coarse organic aggregate exceeds the volume of lost water and, consequently, absorbed water, but the volume of the latter is greater than the pore volume, which is 36.4% of the filler volume. In the case of the powder fraction, the volume of lost water exceeds the volume of organic aggregate contained in the samples at all dosages of the latter. With an increase in dosage, the V_c/V_w ratio decreases, approaching the value for the coarse fraction. This situation indicates that as the wetting–drying cycles proceed, the pore space accessible to water in the cement matrix itself opens up, apparently due to the inclusion of conditionally closed pores and microcracking.

Figures 10 and 11 show graphs of the accumulation of relative deformation by samples depending on the change in the amount of absorbed water as the number of water absorption–drying cycles increases.

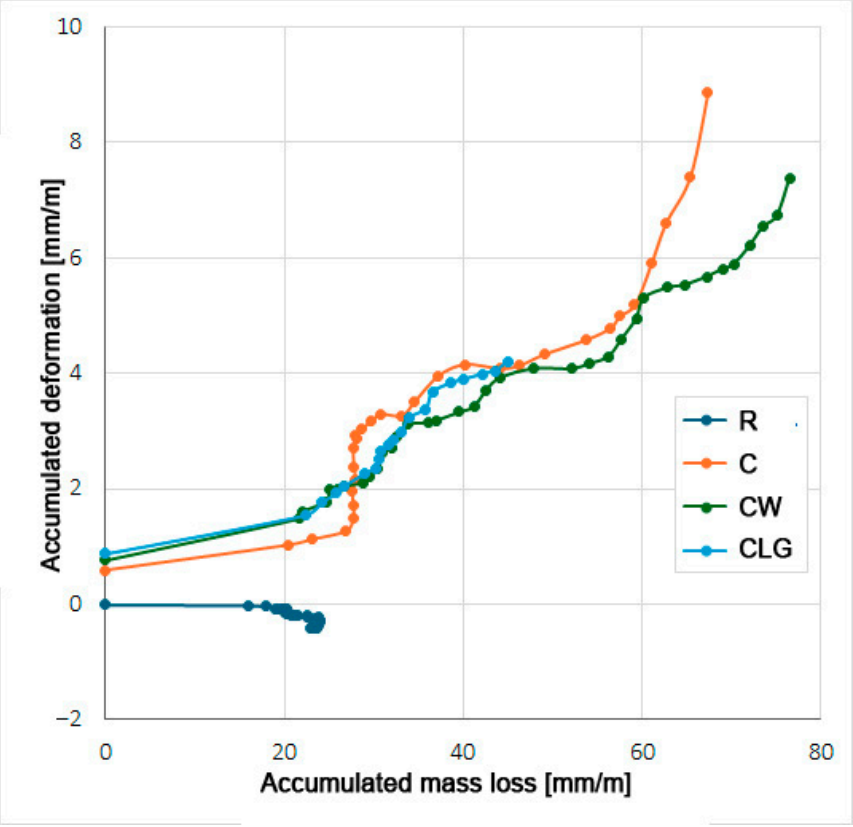


Figure 10. Cumulative relative expansion of samples with a coarse coconut aggregate depending on the accumulated mass of lost/absorbed water: C is without pre-treatment of coconut aggregate; CW is with water-saturated coconut aggregate; CLG is with mineralized coconut aggregate.

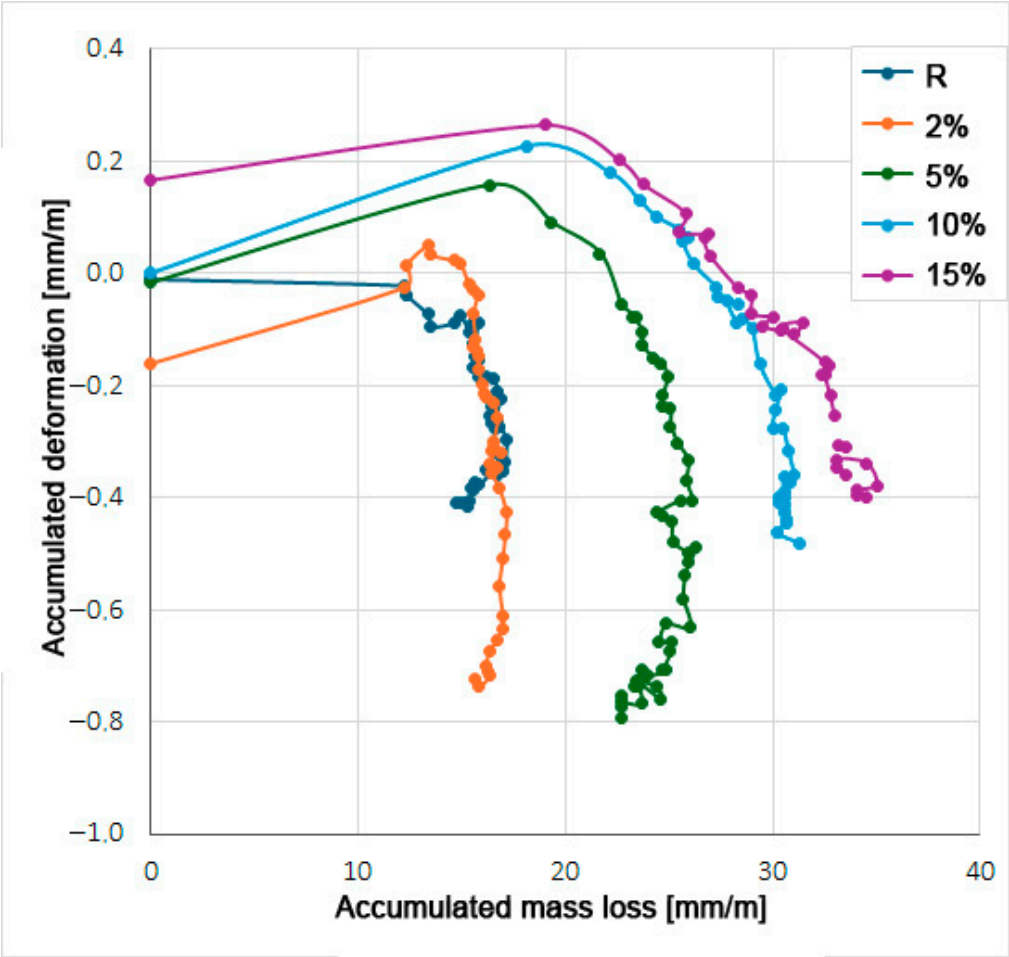


Figure 11. Cumulative relative expansion of samples with fine coconut aggregate depending on the accumulated mass of lost water.

Based on the shape and relative position of the curves in Figure 10, it can be concluded that for the coarse coconut aggregate, there is a relatively direct relationship between the accumulated values of expansion and water absorption. In the first cycles, the expansion is due to a more significant swelling of the organic aggregate than the cement matrix. At the same time, tensile stresses arise in the cement stone, leading to the formation of microcracks in the matrix and the opening of conditionally closed pores, which contributes to an increase in water absorption and swelling, which in turn leads to an increase in crack formation. Such an interdependent process leads to rapid destruction of concrete, which we observed in our experiments, starting from the 10th cycle.

Figure 11 shows the results of determining the deformation of samples with a fine coconut aggregate depending on the accumulated mass of lost water.

The graphs in Figure 11 show that the higher the content of coconut shell endocarp in the aggregates, the more water participates in the cyclic process of absorption and evaporation. The shift in the curves to the right along the abscissa axis indicates that the greatest amount of water is absorbed in the first cycle and causes the greatest expansion of the samples. After this, shrinkage begins, but the amount of absorbed water increases, i.e., more water is absorbed (evaporated) in each cycle than was absorbed (evaporated) in the previous cycle. After the samples have reached an accumulated shrinkage of approximately 0.4 mm/m, the curves bend toward a decrease in the amount of absorbed (evaporated) water from cycle to cycle. This indicates the occurrence of two oppositely directed processes, one of which, for example, is associated with a decrease in the volume

of the samples as a result of general shrinkage and a corresponding decrease in the internal empty volume filled with water, and the other with an increase in the latter as a result of contraction.

2.3. Results of Strength Tests of Samples

The samples of the first series with a coarse coconut aggregate, unlike the samples of the second series with a fine aggregate, did not withstand the effects of periodic wetting and drying and were subject to cracking after the first few cycles (Figure 12).

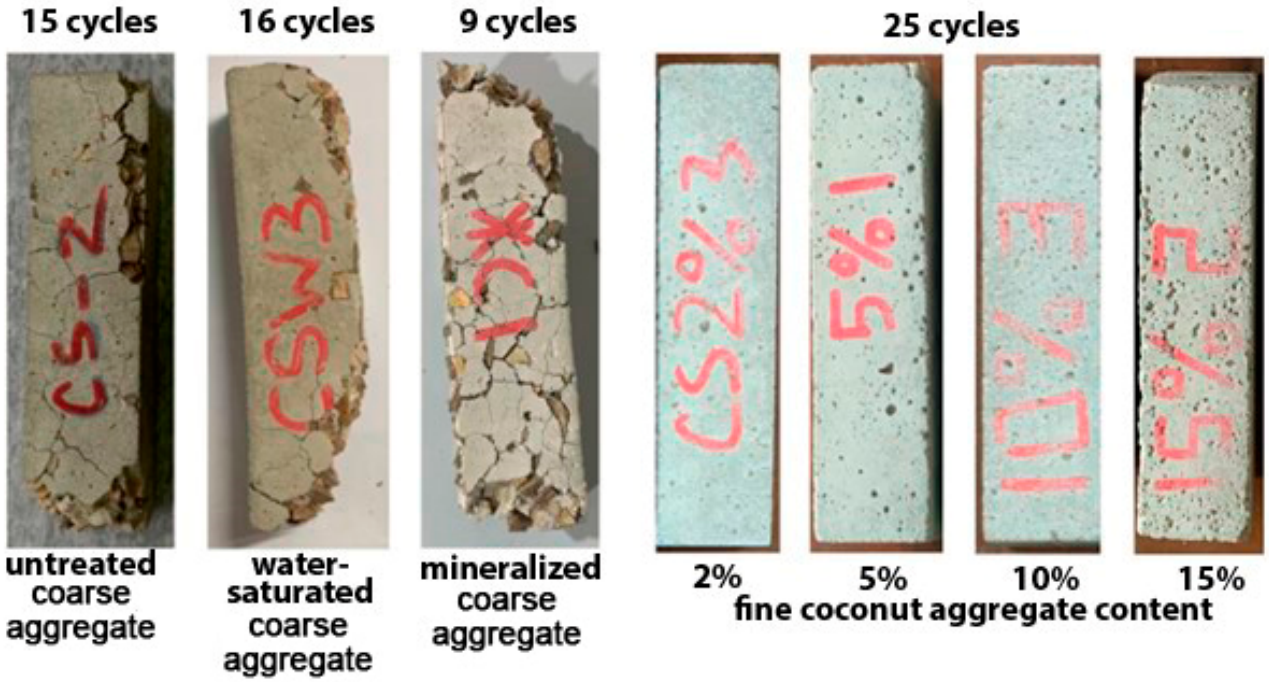


Figure 12. Appearance of samples after passing a specified number of cycles.

The samples of the first series were unsuitable for strength tests due to severe cracking. The results of strength tests of the samples of the second series are given in Table 3.

Table 3. Results of strength tests of samples with fine coconut aggregate.

Coconut Aggregate Content, %	Strength, MPa	
	When Bending	Under Compression
0	7.16	54.2
2	6.13	45.9
5	6.73	49.2
10	4.78	40.0
15	4.34	33.9

Compositions with coconut aggregate are inferior in strength to control samples and, the more so, the higher the content of organic aggregate. Thus, with a content of this component of 15%, the compressive strength of concrete was only 63%; the bending strength was 61% of the strength of the standard. The smallest loss of strength compared to the standard (9% in compression and 6% in bending) was shown by the composition with an organic aggregate content of 5%. This is also confirmed by data from other researchers [29]. In contrast to our results in [23], the use of coarse coconut filler increased the strength of 28-day concrete. This contradiction is explained by the fact that the authors of the cited work used air-dry organic filler, which absorbed and retained part of the mixing water

in the concrete mix, which led to a decrease in W/C and, accordingly, to an increase in strength. While in our case, coconut shell in a water-saturated state was used.

3. Materials and Methods

3.1. Materials

1. Portland cement CEM I 42.5N produced by "Peterburgcement" (Slantsy, Leningrad region, Russia) with the following characteristics:
   - Compressive strength at the age of 2 days is not less than 10 MPa;
   - Compressive strength at the age of 28 days is 42.5–62.5 MPa;
   - Setting begins no earlier than 60 min;
   - Specific surface area is 350 m^2/kg.

The mineralogical composition of clinker is presented in Table 4. The chemical composition of clinker is presented in Table 5.

Table 4. Mineralogical composition of cement clinker.

C_3S	C_2S	C_3A	C_4AF
62.91	11.36	5.36	11.76

Table 5. Chemical composition of Portland cement clinker (PCC) and coconut shell ash (CSA) [2].

Material	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	$K_2O + Na_2O$
PCC	66.33	20.57	4.49	3.87	3.22	1.26	1.18
CSA	3.55	52.55	13.74	7.65	1.60	0.57	2.82

2. Quartz polyfractional sand for cement testing according to EN 196-1 [37].
3. Endocarp of mature coconut shell from Thailand, which is a waste product of coconut shavings production. Coconut shell contains amorphous and crystalline carbonaceous materials, which contain 21.8% fixed carbon, 70.8% volatile matter, 5.6% moisture and 1.8% ash [38]. The chemical composition of coconut ash according to [39] is given in Table 2.

In this work, coconut shell endocarp used as an organic aggregate was in the form of two fractions: powdered, obtained by grinding the 5–10 mm fraction in a laboratory mill (Figure 13a), and crushed, with grain sizes from 5 to 20 mm (Figure 13b).



(**a**) (**b**)

Figure 13. Organic aggregate from coconut shell: (**a**) powder; (**b**) crushed, fractions 5–20 mm.

3.2. Methods of Studying the Endocarp of Coconut Shell

The following properties were obtained as average values from the results of at least three tests:

The grain composition of the coconut shell was obtained by sieving it through a standard set of sieves.

The bulk density of the coconut shell aggregate was calculated based on the mass and volume of the sample, which was dried to a constant mass and poured into a 5 L measuring vessel without additional compaction, from a height of 10 cm.

The true density of the powder fraction of the coconut shell aggregate was determined by measuring the volume of distilled water displaced by the filler from the pycnometer. The coconut shell was pre-crushed until it completely passed through sieve No. 008 (Figure 14a).



(**a**) (**b**) (**c**)

Figure 14. Tests of coconut shell aggregate to determine true density (**a**), average density (**b**) and compressive strength (**c**).

The average density of coconut shell grains was determined by hydrostatic weighing of 5–20 mm fraction organic aggregate grains saturated with water to a constant mass using a perforated bucket (Figure 14b). Volume expansion at full saturation with water, amounting to 12.2%, was considered. The aggregate compressive strength was determined on 10–20 mm fraction coconut shells. First, the aggregate was poured into a steel composite cylinder from a height of 10 cm, and then the cylinder was compressed with a hydraulic press until the punch was immersed by 20 mm. The compressive strength test is shown in Figure 14c.

Water absorption in one hour in % was calculated based on the difference in the sample mass before and after its saturation with water for 1 h relative to the initial mass. Before the saturation procedure, the organic aggregate sample was dried until a constant mass was achieved. To determine the total water absorption, the sample mass was measured initially and then again after attaining a constant mass following saturation with water.

Open porosity in % was numerically equal to the water absorption of the aggregate in one hour, true porosity in % was calculated using formula 1. Closed porosity in % was obtained by subtracting true porosity from open porosity.

$$P_o = \left(1 - \frac{p_a}{p_t}\right) \cdot 100\% \quad (1)$$

where P_o is open porosity, p_a is average density and p_t is true density.

Water saturation under vacuum of the 5–20 mm fraction was carried out in a desiccator at a residual pressure of 0.3 Pa until the release of air bubbles from the sample completely ceased.

Swelling (volume increase) of endocarp in water was determined in a graduated cylinder (Figure 15), into which the powdered material was initially poured to a level of 117 mL (Figure 15a), after which water was added to a level of 300 mL. To evenly distribute water between the particles, the material was stirred (Figure 15b). After the powder had settled for 24 h, its level was set at 155 mL (Figure 15c). The volume of water between this level (155 mL) and the initial volume of dry powder (117 mm) should be attributed not only to the swelling of the particles, but also to the formation of water layers between them, i.e., to some separation of the grains. However, as experience shows, soaking powdered materials, as a rule, reduces the total volume of the material as a result of a denser packing of the particles. Neglecting these initial oppositely directed effects, we will consider the volume of dry powder as the initial one. A similar test was conducted on crushed coconut shells.



(**a**) (**b**) (**c**)

Figure 15. Testing the powder fraction of organic aggregate for volume increase upon swelling in water: (**a**) is coconut powder in a flask before adding water, (**b**) is coconut powder in a flask after adding water and (**c**) is coconut powder in a flask with water after 24 h.

3.3. Composition and Production of Samples from Cement Mortar with Partial Replacement of Sand with Coconut Shell Aggregate

The mortar mixture was prepared in an automatic mortar mixer Matest E093 (Treviolo, Italy). The preparation of the mortar mixture included two mixing stages. During the first

stage, water, cement and standard sand were mixed for one minute. During the second stage, coconut aggregate was added and mixing continued for another two minutes.

Two series of samples measuring 40 × 40 × 160 mm were prepared, 3 pieces for each test. The base (control) composition of the solution for both series had a cement–sand ratio of 1:3 and W/C = 0.50.

The first series of samples were prepared by replacing 50% of the standard sand with an equivalent volume of coarse coconut aggregate (5–20 mm). The symbols for the prepared compositions and the material consumption are given in Table 6.

Table 6. Composition of mixtures with coarse coconut aggregate.

Mix Name	Characteristics of Mix	Consumption of Components, kg/m^3			
		Cement	Water	Sand	Coarse Coconut Aggregate
R	Without coconut aggregate	488	244	1464	0
C	With coconut aggregate	488	244	732	293
CW	With water saturated coconut aggregate	488	244	732	293
CLG	With coconut aggregate treated with lime milk and liquid glass	488	244	732	293

These compositions differed only in the type of preliminary treatment of the coconut aggregate. Composition C contained untreated aggregate. Composition CW contained the coconut aggregate pre-soaked in water for 48 h to neutralize hemicellulose and lignin, which were contained in coconut shells and acted as retarders of the cement hydration process [40]. Coconut aggregate for composition CLG was soaked in lime milk for 24 h, then dried, immersed in a liquid glass solution for 24 h and dried again.

Samples of the second series were prepared by replacing 2 to 15% of standard sand with an equivalent volume of powdered fraction of coconut aggregate. The symbols for the prepared compositions and material consumption are given in Table 7.

Table 7. Composition of mixtures with fine coconut aggregate.

Mix Name	Consumption of Components, kg/m^3			
	Cement	Water	Sand	Fine Coconut Aggregate
R	488	244	1464	0
2%	488	244	1435	12
5%	488	244	1391	29
10%	488	244	1317	59
15%	488	244	1244	88

The manufactured samples of all series were stored for the first day in forms in a humid environment, and the rest of the time in water at a temperature of 20 °C. Testing began when the samples reached the age of 28 days.

3.4. Methodology for Testing Samples

To assess the compatibility of moisture deformations in the cement matrix and coconut aggregate, which significantly affects the durability of concrete, all samples were subjected to wetting–drying cycles. One cycle included saturation of the samples with water at a temperature of (20 ± 2) °C; for 7 h and drying in a drying oven for 7 h at a temperature of 105 ± 5 °C;. During transfer, the samples were briefly exposed to ambient air in a

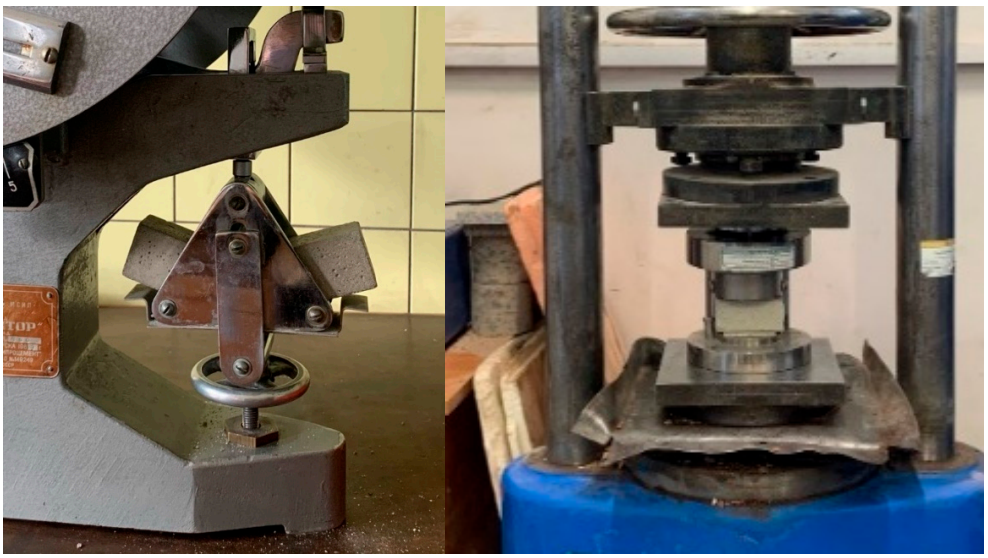
laboratory room with controlled temperature values of (20 ± 2) °C; and relative humidity of 55–60%. These operations were carried out as provided in the method for determining water absorption. At the end of each half-cycle, after water saturation and after drying, the mass and deformation of the samples were measured. The deformation was measured using the Matest E077 device (Treviolo, Italy), shown in Figure 16.



Figure 16. Device for measuring sample shrinkage.

At the end of each half-cycle, after water saturation and after drying, the mass and deformation of the samples were measured.

After completion of 25 cycles, the samples were tested for tensile bending, and the resulting halves of the samples were tested for compression using the standard method (Figure 17).



(**a**) (**b**)

Figure 17. Tests of specimens for strength in bending (**a**) and compression (**b**).

4. Conclusions

The properties of cement composites with partial replacement of standard sand with organic aggregate from coconut shell were investigated.

1. The properties of coconut shell endocarp, such as average and true density, water absorption, porosity, and volumetric expansion upon swelling in water, were experimentally established.
2. It was established that the volume expansion of the coconut shell endocarp during swelling in water is at least 10 times greater than the volume deformation of the swelling of the cement matrix, which is no more than 1%.
3. When using the fine coconut aggregate and replacing standard sand from 2 to 15%, the accumulated expansion is observed only in the first 4–5 test cycles, and then followed by accumulated shrinkage. The initial accumulated expansion increases with the percentage of replacement of natural sand with coconut aggregate. Since the control mix shows only shrinkage, the expansion of the samples is due to swelling of the coconut aggregate. The final value of the accumulated shrinkage of the studied samples, despite the initial expansion, is higher than that of the control ones, so it can be concluded that the organic aggregate contributes to the shrinkage deformation as a result of its own gradual shrinkage from cycle to cycle.
4. A gradual decrease in the average mass of the samples from cycle to cycle was established, caused by the evaporation of water during drying. Consequently, with each subsequent cycle, the volume of space filled with absorbed water increases, which is a consequence of swelling of the coconut shell endocarp and microcracking in the cement matrix.
5. There is a direct relationship between expansion and the amount of water absorbed in cycles for the coarse coconut aggregate. Swelling of the aggregate leads to the formation of microcracks in the matrix and the opening of closed pores, which contributes to the growth of further water absorption and swelling, this in turn leads to an increase in crack formation. Such an interdependent process ensures the rapid destruction of the cement matrix.
6. The obtained dependences of accumulated moisture deformations of expansion and shrinkage on the amount of water absorbed and lost in cycles show that during the period of accumulated shrinkage development, the loss of mass of the samples increases. After the samples reach the accumulated shrinkage of approximately 0.4 mm/m, the amount of absorbed (evaporated) water decreases from cycle to cycle. This indicates the occurrence of two oppositely directed processes, one of which, for example, is associated with a decrease in the volume of the samples as a result of general shrinkage and a corresponding decrease in the internal empty volume filled with water, and the other with an increase in the internal empty volume as a result of contraction.
7. Partial replacement of sand with coconut aggregate reduces the strength of the cement mortar, and increases with a larger replacement percentage. The use of a coarse coconut aggregate (5–20 mm) when replacing 50% of the sand led to the destruction of 28-day samples after several wetting–drying cycles. When using a powder fraction, no external signs of destruction of the cement samples were observed.

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References

1. Liu, H.; Li, Q.; Ni, S.; Wang, L.; Yue, G.; Guo, Y. Effect of nano-silica dispersed at different temperatures on the properties of cement-based materials. *J. Build. Eng.* **2022**, *46*, 103750. [CrossRef]
2. Bai, G.; Zhu, C.; Liu, C.; Liu, B. An evaluation of the recycled aggregate characteristics and the recycled aggregate concrete mechanical properties. *Constr. Build. Mater.* **2020**, *240*, 117978. [CrossRef]
3. Tam, V.W.Y.; Butera, A.; Le, K.N.; Li, W. Utilising CO_2 technologies for recycled aggregate concrete: A critical review. *Constr. Build. Mater.* **2020**, *250*, 118903. [CrossRef]
4. Kumar, K.; Dixit, S.; Arora, R.; Vatin, N.I.; Singh, J.; Soloveva, O.V.; Ilyashenko, S.B.; John, V.; Buddhi, D. Comparative Analysis of Waste Materials for Their Potential Utilization in Green Concrete Applications. *Materials* **2022**, *15*, 4180. [CrossRef]
5. Kolesnikova, O.; Syrlybekkyzy, S.; Fediuk, R.; Yerzhanov, A.; Nadirov, R.; Utelbayeva, A.; Agabekova, A.; Latypova, M.; Chepelyan, L.; Volokitina, I.; et al. Thermodynamic Simulation of Environmental and Population Protection by Utilization of Technogenic Tailings of Enrichment. *Materials* **2022**, *15*, 6980. [CrossRef]
6. Chin, W.Q.; Lee, Y.H.; Amran, M.; Fediuk, R.; Vatin, N.; Kueh, A.B.H.; Lee, Y.Y. A Sustainable Reuse of Agro-Industrial Wastes into Green Cement Bricks. *Materials* **2022**, *15*, 1713. [CrossRef]
7. Barabanshchikov, Y.; Usanova, K. Influence of Silica Fume on High-Calcium Fly Ash Expansion during Hydration. *Materials* **2022**, *15*, 3544. [CrossRef]
8. Barabanshchikov, Y.; Usanova, K.; Akimov, S.; Bílý, P. Low heat concrete with ground granulated blast furnace slag. *IOP Conf. Ser. Mater. Sci. Eng.* **2020**, *896*, 012098. [CrossRef]
9. Khanzada, F.A.; Nazir, K.; Ishtiaq, M.; Javed, M.F.; Kashif-Ur-rehman, S.; Aslam, F.; Musarat, M.A.; Usanova, K.I. Concrete by Preplaced Aggregate Method Using Silica Fume and Polypropylene Fibres. *Materials* **2022**, *15*, 1997. [CrossRef]
10. Ibraheem, M.; Butt, F.; Waqas, R.M.; Hussain, K.; Tufail, R.F.; Ahmad, N.; Usanova, K.; Musarat, M.A. Mechanical and Microstructural Characterization of Quarry Rock Dust Incorporated Steel Fiber Reinforced Geopolymer Concrete and Residual Properties after Exposure to Elevated Temperatures. *Materials* **2021**, *14*, 6890. [CrossRef]
11. Arandara, K.P.; Paranavithana, G.N.; Priyadarshana, S.T.; Pitawala, H.M.T.G.A.; Dissanayake, R. Evaluation of coconut shell biochar on recycled aggregate concrete through petrographic studies. *Constr. Build. Mater.* **2024**, *428*, 136342.
12. Ahmad, J.; Zhou, Z.; Martínez-García, R.; Vatin, N.I.; De-Prado-gil, J.; El-Shorbagy, M.A. Waste Foundry Sand in Concrete Production Instead of Natural River Sand: A Review. *Materials* **2022**, *15*, 2365. [CrossRef] [PubMed]
13. Karunarathna, S.; Linforth, S.; Kashani, A.; Liu, X.; Ngo, T. Effect of recycled rubber aggregate size on fracture and other mechanical properties of structural concrete. *J. Clean. Prod.* **2021**, *314*, 128230.
14. Durczak, K.; Pyzalski, M.; Sujak, A.; Juszczyk, M.; Sala, D.; Ustinovichius, L. Efficient Management of Asbestos Waste Through Utilization as Mineral Additives in Portland Cement Production. *Materials* **2024**, *17*, 5793. [CrossRef]
15. Palh, A.R.; Mangi, S.A.; Odho, M.A.; Kalhoro, A.A. Experimental Study on Concrete Incorporating Date Seed as Partial Replacement of Coarse Aggregates. *Neutron* **2021**, *20*, 113–122.
16. Sanchaya, M.; Harikaran, M.; Cheran, K.; Varoon, M.; Jayaprakash, P.; Navin, G. Experimental investigation on rice husk ash and coconut shell used in building materials. *Mater. Today Proc.* **2022**, *68*, 1697–1702.
17. Muthusamy, K.; Nordin, N.; Vesuvapateran, G.; Ali, M.; Mohd Annual, N.A.; Harun, H.; Ullap, H. Exploratory Study of Rubber Seed Shell as Partial Coarse Aggregate Replacement in Concrete. *Res. J. Appl. Sci. Eng. Technol.* **2014**, *7*, 1199–1202.
18. Hamada, H.M.; Skariah Thomas, B.; Tayeh, B.; Yahaya, F.M.; Muthusamy, K.; Yang, J. Use of oil palm shell as an aggregate in cement concrete: A review. *Constr. Build. Mater.* **2020**, *265*, 120357.
19. Durczak, K.; Pyzalski, M.; Pilarski, K.; Brylewski, T.; Sujak, A. The Effect of Liquid Slurry-Enhanced Corrosion on the Phase Composition of Selected Portland Cement Pastes. *Materials* **2021**, *14*, 1707. [CrossRef]
20. Athira, G.; Bahurudeen, A. Rheological properties of cement paste blended with sugarcane bagasse ash and rice straw ash. *Constr. Build. Mater.* **2022**, *332*, 127377.
21. Mohamad, N.; Muthusamy, K.; El Gelany Ismail, M.A.K. Cockle Shell as Mixing Ingredient in Concrete: A Review. *Construction* **2021**, *1*, 9–20.

22. Prusty, J.K.; Patro, S.K. Properties of fresh and hardened concrete using agro-waste as partial replacement of coarse aggregate: A review. *Constr. Build. Mater.* **2015**, *82*, 101–113.
23. Gunasekaran, K.; Kumar, P.S.; Lakshmipathy, M. Mechanical and bond properties of coconut shell concrete. *Constr. Build. Mater.* **2011**, *25*, 92–98.
24. Lejano, B.; Elevado, K.J.; Fandiño, M.A.; Ng, E.A.; Nicole Datinguinoo, Z.A.; Oliveros, S.B. Experimental investigation of utilizing coconut shell ash and coconut shell granules as aggregates in coconut coir reinforced concrete. *Clean. Eng. Technol.* **2024**, *21*, 100779.
25. Thilagashanthi, T.; Gunasekaran, K.; Satyanarayanan, K.S. Microstructural pore analysis using SEM and ImageJ on the absorption of treated coconut shell aggregate. *J. Clean. Prod.* **2021**, *324*, 129217.
26. Ranatunga, K.S.; del Rey Castillo, E.; Toma, C.L. Evaluation of the optimal concrete mix design with coconut shell ash as a partial cement replacement. *Constr. Build. Mater.* **2023**, *401*, 132978.
27. Saadullah, S.T.; Haido, J.H.; Al-Kamaki, Y.S.S. Mechanical properties of sustainable green self-compacting concrete incorporating recycled waste PET: A state-of-the-art review. *Adv. Concr. Constr.* **2023**, *16*, 35–57.
28. Tomar, R.; Kishore, K.; Singh Parihar, H.; Gupta, N. A comprehensive study of waste coconut shell aggregate as raw material in concrete. *Mater. Today Proc.* **2021**, *44*, 437–443.
29. Bhoj, S.; Manoj, A.; Bhaskar, S. Usage potential and benefits of processed coconut shells in concrete as coarse aggregates. *Mater. Today Proc.* **2023**, *in press*. [CrossRef]
30. Mathew, S.P.; Nadir, Y.; Arif, M.M. Experimental study of thermal properties of concrete with partial replacement of coarse aggregate by coconut shell. *Mater. Today Proc.* **2020**, *27*, 415–420.
31. Stel'makh, S.A.; Beskopylny, A.N.; Shcherban', E.M.; Mailyan, L.R.; Meskhi, B.; Shilov, A.A.; El'shaeva, D.; Chernil'nik, A.; Kurilova, S. Alteration of Structure and Characteristics of Concrete with Coconut Shell as a Substitution of a Part of Coarse Aggregate. *Materials* **2023**, *16*, 4422. [CrossRef] [PubMed]
32. Arora, R.; Kumar, K.; Dixit, S.; Tipu, R.K.; Kaul, P.; Chauhan, S.; Raju, Y.K.; Nijhawan, G.; Haindavi, P. Parametric investigation of coconut shells as partial replacement of coarse aggregates in sustainable concrete. *E3S Web Conf.* **2023**, *430*, 01180. [CrossRef]
33. Serda, M.; Becker, F.G.; Cleary, M.; Team, R.M.; Holtermann, H.; The, D.; Agenda, N.; Science, P.; Sk, S.K.; Hinnebusch, R.; et al. Behaviour of Coconut Shell Aggregate (CSA) Concrete at Elevated Temperature. *Int. J. Sci. Res. Dev.* **2020**, *8*, 390–393.
34. Kumbhar, M.V.P.; Pawar, M.R.E. Experimental Analysis of Concrete for Replacement of Sand by Coconut Shell Crush. *Int. J. Sci. Eng. Res.* **2021**, *12*, 420–424.
35. Azunna, S.U.; Aziz, F.N.A.A.; Cun, P.M.; Elhibir, M.M.O. Characterization of lightweight cement concrete with partial replacement of coconut shell fine aggregate. *SN Appl. Sci.* **2019**, *1*, 649.
36. *GOST 32496-2013*; Fillers Porous for Light Concrete. Specifications. Standartinform: Moscow, Russia, 2013.
37. *EN 196-1:2016*; Methods of Testing Cement—Part 1: Determination of Strength. European Committee for Standardization: Brussels, Belgium, 2016.
38. Herring, T.C.; Nyomboi, T.; Thuo, J.N. Ductility and cracking behavior of reinforced coconut shell concrete beams incorporated with coconut shell ash. *Results Eng.* **2022**, *14*, 100401.
39. Kabir Ahmad, R.; Anwar Sulaiman, S.; Yusup, S.; Sham Dol, S.; Inayat, M.; Aminu Umar, H. Exploring the potential of coconut shell biomass for charcoal production. *Ain Shams Eng. J.* **2022**, *13*, 101499.
40. Sloane, N. The Packing of Spheres. *Sci. Am.* **1984**, *250*, 116.

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