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Carbonation of industrial calcium-containing waste for the production of sustainable construction materials

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ABSTRACT

The objective of this experiment was to use the waste from sugar processing industries, such as defecation sludge, hydrated lime, and pure quick lime in the form of an accelerated carbonation reaction for evaluation of their potential as a construction material. The chemical composition of the waste materials was studied in addition to their changes due to the carbonation reaction as well as the physical and mechanical properties. Various instruments, such as XRD, FTIR, DTA/TG, were used. Upon forcing carbonation, the compounds of calcium in the waste combined with CO_2 to form stable $CaCO_3$. From the DS-5 sample, it is observed that the carbonated waste contained 42.5% by weight of CO_2. From the most effective sample, it was evident that around 88.5% of the product was converted into marble-like crystals of $CaCO_3$. The samples have achieved a compressive strength of 27.5 MPa and water absorption of 3.1%. These observations revealed that through forcing carbonation of defecate, hydrated lime, and quicklime, one can effectively utilize industrial waste, sequestrate CO_2 in minerals, and convert the product into secondary raw material for manufacturing building materials. Consequently, the proposed technology not only stimulates waste reutilization and rational consumption but also helps reduce the anthropogenic load on the environment through the mineral binding of CO_2 in the form of $CaCO_3$. The obtained results may serve as a basis for the creation of ecologically effective and economical building materials using the carbonization of calcium-containing wastes from the sugar industry.

Keywords: climate change, circular economy, construction materials, resource recovery, waste management.

INTRODUCTION

Sugar defecate, which is an industrial waste that consists of calcium carbonate and can be utilized to produce economically viable construction materials with relatively small investment, is an example of such industrial wastes. The process of sugar production in sugar plants includes many stages during which a great amount of solid waste appears. At the stage of defecation, when the processing of sugar solution takes place, a huge amount of sludge (defecate) appears. Defecate is a high-volume industrial waste. That is why it is essential to study the composition of defecate elements [1]. Defecate consists of more than 50% calcium carbonate ($CaCO_3$). This composition makes defecate one of the possible sources of construction material production [2]. Defecate is also a raw material for mineral fertilizers and feed additives production, as well as calcium-containing additives for the metallurgy sector [3]. Large volumes of defecate formed during the processing of sugar beet and sugarcane have turned into an acute environmental issue. Only a small share of defecate is used in mineralization of soil and water purification, while the bulk

is being thrown into waste sites [4–5]. The same can be observed in the case of the defecate produced at "Khorezm shakar" LLC, which is one of the major producers of sugar in Uzbekistan.

Nevertheless, it is possible to regard such kinds of waste as potential secondary raw materials. Their use may not only bring additional economic profit but also positively influence the improvement of the environmental state [6–7]. Jothilingam et al., have scientifically proven that there is a possibility of producing environmentally sustainable construction materials via partial or full replacement of regular raw materials that consume much energy and resources and are responsible for environmental pollution with waste materials from industries [8]. Moreover, there were some investigations concerning the classification of defecate as non-hazardous waste since it contains no hazardous components and has nearly neutral pH [9]. James, J. carried out experiments on the effect of defecate on soil bonding and strength. The results indicated that adding defecate to the soil increased its strength [10]. According to an extensive study conducted by Biffi, and Janani, the compressive strength of defecate added to the soil increased with time and was enhanced by the addition of defecate [11]. Radwan et al. carried out an interesting study on the use of waste products for industrial uses in ternary blended cement. The innovative application included the use of glass powder (GP) and defecate as cement replacement in ternary blended cement. The effect of defecate replacement at different amounts of 5% to 20% defecate replacement and 20% glass powder in ternary cement was evaluated. The results indicated that adding 10% and 15% of defecate to binary cement improved the consistency and strength of the cement while decreasing sorptivity. In addition, the results indicated better microstructure because of reduced voids, improved reaction of glass powder particles, and hydration products [12]. The possible application of defecate for the stabilization of soil because of the high content of calcium and silicon in defecate has been explored by several scientists. For instance, James and Pandian [13–14] researched the possibility of utilizing defecate as an auxiliary agent in lime stabilization of expansive soils. The viability of defecate formulations for stabilizing dune sands was studied by Moghadam et al. [15]. Parthiban et al. [16] carried out research on the effect of defecate on the lime stabilization process. The potential application of defecate in cement-lime-stabilized blocks was researched by Mansoor et al. [17]. The use of defecate and granite dust mixture for increasing the subgrade strength of clayey soils was examined by Kumar and Garg [18].

One potential solution is the capture and storage of carbon-containing flue gases at construction material manufacturing plants. However, to date, no full-scale implementation has been developed or deployed [20]. Researchers worldwide are investigating alternatives to conventional cement to reduce dependence on Portland cement and to identify waste-derived substitute materials capable of lowering CO_2 emissions [21]. Industrial residues based on calcium (Ca) and magnesium (Mg) represent one of the largest groups of reactive alkaline solids from the standpoint of CO_2 reactivity [22]. By taking into account the chemical composition, Ca- and Mg-containing solid waste can be considered a potential raw material for mineralization due to its high alkalinity level. CO_2 fixation capability of most alkaline solid wastes, including ash, depends on their calcium and magnesium levels [23].

Calcium-containing waste materials, such as oil shale ash (OSA) and wood ash (WA), are abundant in Uzbekistan and comprise over 70% of all solid wastes produced at power plants. Leben et al. noted that the use of local alkaline waste streams via mineral carbonation with CO_2 could help to achieve the objectives of a circular economy and sustainable development [24–25]. Moreover, a share of CO_2 produced by the burning process can also be used [26]. The most remarkable feature that separates coal fly ash from other types of industrial waste is the pozzolanic effect of coal fly ash because of the high content of oxides of silica, calcium, and iron. Thus, coal fly ash can be utilized extensively as an additive in the cement and concrete industries [27–28]. As far as it is known, the disposal of industrial wastes in landfills leads to their self-heating in view of contact with atmospheric moisture and water, which provides exothermic reactions. This way, industrial wastes are extremely hazardous for humans and nature [29–32]. It should be noted that a new method of carbonation has been introduced by Laczny et al. In the process, carbonation was conducted to obtain a product having a certain amount of CaO in it. Thus, a three-component micro-aggregate of CaO, $CaCO_3$, and $CaSO_4$ was created. These results have shown that such an aggregate is suitable to serve as an active substance in cement mortars [33–34].

Atmospheric CO_2 levels rose to 419 ppm in 2021 from 360 ppm during the 19th century, thus illustrating the pressing need for worldwide action to tackle this environmental problem [35]. The CCS technology will become an economically feasible approach by 2050 and could avoid as much as eight billion tons of CO_2 emissions [36]. The mineral carbonation technique, which comprises geological and mineral-based carbon sequestration techniques, is part of the CCS technology. According to Lee et al., carbonation is a process involving two major reactions, namely the removal of metal ions such as Mg and Ca from the phases and the carbonation reaction of CO_2 with the solution obtained. They have pointed out that this reaction is associated with good reaction conditions, efficient carbonation, and production of valuable secondary products [37]. It must be noted that industrial wastes with high alkalinity and reactivity are highly appropriate as raw materials for the process under discussion. The carbonation capacity of the wastes regarding CO_2 sequestration shows that the carbonation of the mentioned minerals will allow decreasing the level of anthropogenic CO_2 emissions by 12.5% worldwide [38]. In accordance with scientific sources, industrial wastes such as wastes from the cement industry, red mud, fly ash, and steel slag may be considered as appropriate raw materials for carbonation processes [39]. Whereas accelerated carbonation, in which CO_2 is added at controlled temperatures and pressures, is an effective process, its application to the manufacture of construction materials needs to be investigated further. Furthermore, very few studies have examined the performance properties, structure, and environmental effects of such construction materials made using carbonated calcium-containing wastes [40,41].

Analysis of various studies shows that more importance is being given in recent years to defecate and other calcium-rich waste as prospective materials for the manufacture of new construction materials. However, as is evident from the existing literature, scientific investigation related to the manufacturing of construction materials through the process of forced carbonation of industrial calcium-rich wastes is quite rare. In the current study, the strength properties and physico-chemical characteristics of coating samples through the process of forced carbonation have been studied with varying amounts of defecate (byproduct of sugar industry), calcium-rich waste, and calcium-containing raw materials. What makes this work distinct from already published studies is the examination of the possibility of manufacturing building materials via forced carbonation of two calcium-containing waste streams produced by the same production site, using carbon dioxide: defecate and waste with a large amount of calcium hydroxide. The current study will give scientific answers to already existing questions about the environmental safety of using sugar industry wastes as part of construction materials.

The unique feature of the study is its integrative nature that links carbonation acceleration with the use of a calcium-containing waste not previously utilized for producing building materials. In terms of technology, the study presents a fast and effective way of CO_2 sequestration combined with the stabilization of dangerous waste. In scientific terms, the study sheds light on the carbonation kinetics of calcium-containing wastes and their effect on the material characteristics. According to the studies conducted, the wastes produced by the sugar industry, namely, defecate with calcium content and hydrated lime, can be processed via forced carbonation to obtain a useful calcium carbonate material. The methodology for quantifying the quantity of $CaCO_3$ produced by carbonation has been developed. The accuracy of the obtained data has been verified by a set of instrumental studies. The quantities of $CaCO_3$ and bound CO_2, estimated by the proposed equation, agree with the findings of the calcimetric study, X-ray diffraction (XRD), FTIR spectroscopy, and DTA/TG thermoanalysis. The appearance of the $CaCO_3$ phase in the XRD patterns, the occurrence of vibration bands characteristic of carbonate functional groups in the FTIR spectra, and the identification of thermodynamic effects of the $CaCO_3$ thermal decomposition during DTA/TG analysis provide evidence for the formation of $CaCO_3$ as a result of the carbonation process.

Additionally, the study will directly contribute towards SDG 9 (Industry, Innovation, and Infrastructure) by developing novel materials and processes; SDG 11 (Sustainable Cities and Communities) through promoting more environmentally friendly construction materials; SDG 12 (Responsible Consumption and Production) through reusing industrial waste materials and turning them into useful substances; and finally SDG 13 (Climate Action) by cutting down on CO_2 emissions. In summary, this study proposes a sustainable and scalable strategy for the carbonation of calcium-containing industrial waste materials. Not only does this study

address important environmental issues, but it also lays the groundwork for future projects in the domain of sustainable building materials.

MATERIALS AND METHODS

Raw materials and sample preparation

Calcium-containing raw materials utilized in the current research consisted of defecate industrial waste that was rich in calcium content ("Khorezm Sugar" LLC), solid calcium-containing waste (hydrated lime, $Ca(OH)_2$), and quicklime (CaO). Before being processed into samples, the defecate and hydrated lime waste was dried, crushed, and mixed with quicklime in a laboratory ball mill. The crushed material was then sieved with a 0.6 mm sieve. In this work, the initial raw materials used for analysis were industrial waste containing high levels of calcium obtained from a sugar manufacturing factory, as depicted in Figure 1. The chemical composition of defecate can be seen in Table 1. The source of hydrated lime waste in this study is "Khorezmshakar" LLC, and it is formed in great amounts during CO_2 generation at the lime-burning shop. A part of the CaO that is produced is used for the purification of juice in sugar manufacturing, and the rest is thrown into the open waste storage sites. The chemical composition of hydrated lime waste is given in Table 2. The quicklime employed in the study was obtained via calcination of limestone mined from the Jamansoy deposit located in the Beruniy district of the Republic of Karakalpakstan at a temperature of 1100 °C. The deposit has considerable resources and is currently being exploited for use as an excellent raw material for manufacturing construction lime, cement, and soda ash. The chemical composition of the Jamansoy limestone was found to be CaO – 55.37 wt.%, MgO – 0.91 wt.%, Al_2O_3 – 0.12 wt.%, Fe_2O_3 – 0.08 wt.%, SiO_2 – 0.84 wt.%, and CO_2 – 43.45 wt.% The chemical composition of the quicklime prepared from the limestone was the following: CaO – 98.0 wt.%, MgO – 0.5 wt.%, SiO_2 – 0.5 wt.%, Al_2O_3 – 0.4 wt.%, Fe_2O_3 – 0.1 wt.%, SO_3 – 0.6 wt.%, and LOI – 0.5 wt.%. Calcium oxide (CaO) was established as the main component.

Taking into account the quantity of CO_2 measured in the experimental samples, the quantity of the secondary calcium carbonate ($CaCO_3$) produced is estimated by applying the following formula [42,43]:

$$CaCo_3^{secondary} = \frac{CO_2 - CO_2^{raw\ material}}{44} \times 100,\% \quad (1)$$

where: CO_2 – the amount of carbon dioxide (CO_2) in the sample, wt.%; $CO_2^{raw\ material}$ – the amount of CO_2 initially present in the raw materials, wt.% (see Table 2); 44 – Mass fraction of carbon dioxide (CO_2) in pure calcium carbonate ($CaCO_3$).

The composition was analyzed using X-ray fluorescence (XRF) spectroscopy, and it was found that CaO (greater than 55 wt.%) was the main constituent of the waste, with trace quantities of MgO, SiO_2, Al_2O_3, and Fe_2O_3. The thermogravimetric analysis (TGA) technique was used to analyze the existence of any volatile elements, to determine the amount of $Ca(OH)_2$, and its ability to carbonate.

From the compositions provided in Table 2, the constituents were mixed homogeneously. Water amounting to 6 wt.% was then gradually incorporated into the dry constituents to form a homogeneous mass. The samples were molded at room temperature and later exposed to a carbonation chamber in which pure CO_2 was passed through them under ambient conditions to accelerate the carbonation process. The samples were kept at the specified pressure for one hour. The carbonated samples were characterized physically and chemically.



a) b)

Figure 1. Appearance of the defecate waste: (a) as-received waste; (b) ground defecate passing through a 0.63 mm sieve

Table 1. Chemical composition of the defecate waste

Sample	Oxide composition (wt.%)							
	CaO	MgO	Fe_2O_3	SiO_2	Al_2O_3	SO_3	R_2O	Loss on ignition (LOI)
F-1	58.80	0.59	0.15	0.59	0.45	1.08	0.06	38.23
F-2	57.90	0.60	0.18	0.62	0.50	1.07	0.07	39.06

Table 2. Chemical composition of hydrated lime waste

CaO	MgO	SiO_2	Al_2O_3	Fe_2O_3	CO_2	SO_3	H_2O	K_2O	Na_2O	P_2O_5	LOI
71.0	0.59	2.17	1.02	0.49	3.73	0.12	20.33	0.16	0.08	0.24	24.06

X-ray fluorescence analysis

The chemical composition of the waste raw material with high calcium concentration, along with carbonated samples, was obtained through X-ray fluorescence (XRF) spectroscopy with the use of a Rigaku X-ray fluorescence spectrometer. These analyses were conducted both under vacuum and in an air atmosphere. The content of calcium in the raw material and the conversion degree of CaO into $CaCO_3$ in the carbonated samples were determined. The vacuum was used to improve the detection limit of the light elements (Si and Al) while the analysis of heavy elements was done under an air atmosphere using a Rh-anode X-ray tube with 50 kV and 40 mA settings. The samples used for analysis were in the form of pressed pellets, while calibration was done based on SRM/NIST standards. The data collection and processing were accomplished through Spectra Plus and Shimadzu XRF Manager software (Figure 2).

As per the findings of the XRF analysis for the sugar industry wastes, the defecate sample has very high amounts of calcium oxide. It was found that there are very high levels of CaO (calcium oxide), having a value of 58.8 wt.%. This clearly shows that the given waste can be a good source of calcium for mineral carbonation and the production of low-carbon building materials. The most prominent peaks in the XRF spectrum are for the Ca-Kα, Ca-Kβ, and S-Kα emission lines (Figure 3).

The highest peaks in the XRF spectrum correspond to the Ca-Kα and Ca-Kβ lines, which means that calcium is dominant in the studied sample. The peaks of Si, Al, Mg, and Fe lines have low intensities; therefore, they can be said to be present in small quantities. All other trace elements are almost absent in the studied sample. From the XRF results, it is clear that the CaO content in the hydrated lime waste (pushonka) is quite high: 70.10 wt.%, which makes this material suitable for mineral carbonation and CO_2 sequestration in the form of calcium carbonate. The low quantities of SiO_2 (1.12 wt.%), Al_2O_3 (0.737 wt.%), MgO (0.688 wt.%), Fe_2O_3 (0.565 wt.%), SO_3 (0.899 wt.%), and K_2O (0.20 wt.%) indicate the calcium dominance in the material. Low concentration of SO_3 can facilitate the stable process of hydration and decrease the negative impacts from sulfate compounds. The low level of alkali oxides and

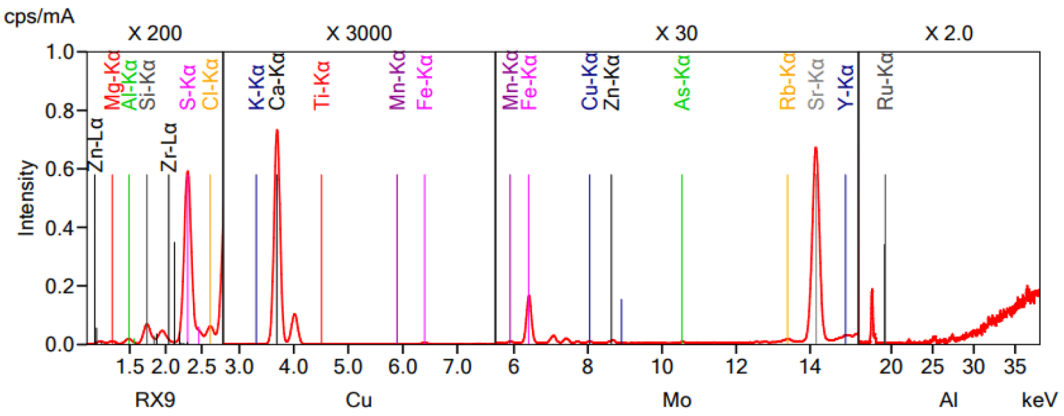


Figure 2. X-ray fluorescence analysis of defecate

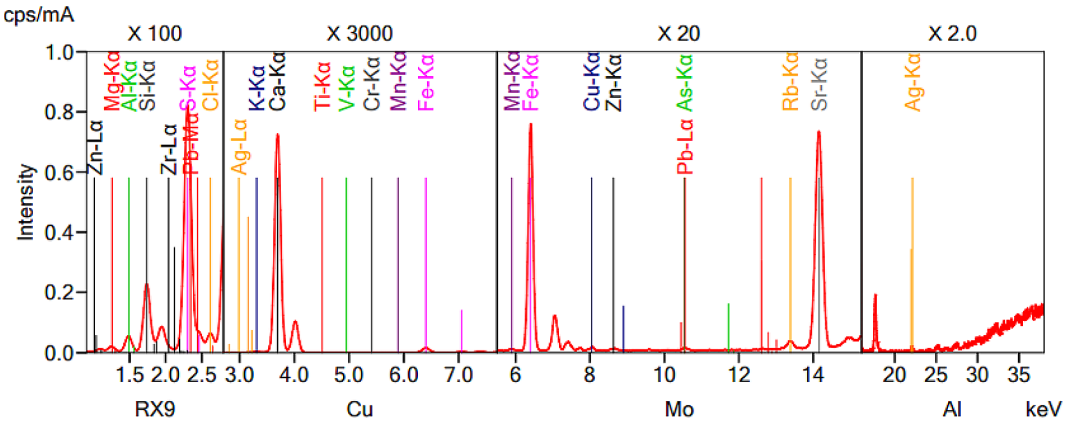


Figure 3. X-ray fluorescence analysis of hydrated lime waste

heavy metals also confirms the environmental safety of the material. In summary, hydrated lime waste (pushonka) proved to be an excellent alternative mineral resource for CO_2 sequestration and the manufacture of low-carbon binders and sustainable construction materials.

Thermal analysis

Thermal analysis of the specimens was done through a NETZSCH STA 449 F3 Jupiter simultaneous thermal analyzer. This test was conducted in nitrogen gas with a gas flow of 50 mL/min over a temperature range of 25–1400 °C at a heating rate of 10 °C/min. About 20 mg of each specimen was used in each test.

Fourier transform infrared spectroscopy

The Fourier transform infrared spectroscopy (FTIR) technique was used to investigate the functional groups and structural alterations of the carbonated samples subjected to the accelerated aging process. FTIR spectra were obtained using the Bruker Tensor 27 FTIR spectrometer within the wavenumber range of 4000 to 250 cm^{-1}, having a spectral resolution of 4 cm^{-1}.

X-ray diffraction analysis

For the characterization of phase compositions of the carbonated samples with an increased rate of reaction, X-ray diffraction (XRD) studies have been performed. The analysis was done on a Rigaku diffractometer utilizing Cu Kα radiation (λ=1.5406 Å). The diffraction data were collected in the 2θ range of 5–80° at room temperature. Phase identification was carried out using the ICDD PDF database.

Scanning electron microscopy and energy-dispersive spectroscopy

The structure and chemical elements of the accelerated carbonated samples were studied using scanning electron microscopy (SEM) associated with energy-dispersive spectroscopy (EDS).

SEM examinations were conducted using an Alpha-type scanning electron microscope with high vacuum, the acceleration voltage being 20 kV and the working distance varying from 5 to 6 mm. The EDS examination was carried out to determine the chemical elements in certain areas of the samples.

RESULTS

Chemical composition of the raw material mixtures

Optimum combination of the accelerated carbonated coating components was achieved through variation in the ratio between defecate, solid waste with a rich amount of calcium (hydrated lime), and quicklime. In total, 10 specimens, referred to as DS-1 through DS-10, were obtained. The amount of quicklime remained unchanged during experiments. Defecate content was varied in the range of 5 to 50 wt.%, and the content of solid waste with a rich amount of calcium (hydrated lime) in the range of 5 to 45 wt.%, with a constant 50 wt.% of quicklime in each specimen. Chemical compositions and phase formation features of the prepared specimens were studied. Compositions of all studied specimens are listed in Table 3.

All of the raw materials were calcium-containing materials that were chosen to obtain $Ca(OH)_2$ and CaO, as these elements are important for calcite formation and carbonate phases. Quicklime was chosen as the main part of the raw materials, and in the presence of 6 wt.% of water, it provided the reactive oxides necessary for the formation of calcium hydroxide. Calcium carbonate in defecate was the main ingredient for the accelerated carbonation and calcite ($CaCO_3$) minerals formation. $Ca(OH)_2$ from solid waste with a high content of calcium was added as an additional calcium material. Thus, this method is the most efficient in terms of using available material and sustainable construction material production from industrial waste. An accelerated carbonation reactor was designed and employed in the accelerated carbonation experiments. The reactor had a temperature control, a CO_2 gas inlet, pressure regulating valves, and a gas distribution system to ensure uniform distribution of CO_2 gas in the reactor. Specimens with a dimension of 50 × 50 × 10 mm were produced based on the compositions from Table 3 and then placed in the accelerated carbonation reactor. The accelerated carbonation was carried out using the following operating conditions: 25 °C temperature, 1.5 bar pressure, 2 $L \cdot min^{-1}$ of CO_2 gas flow rate, and a 1-hour reaction time. The above conditions were chosen after several initial experiments. The level of carbonation was determined through XRD analysis by determining the reduction of $Ca(OH)_2$ and the formation of $CaCO_3$.

Quantitative analysis of carbon dioxide (CO_2) in the obtained samples shows that during the early period of the carbonation process, the amount of the bound CO_2 depends on the physical and mechanical characteristics of the sample preparation, i.e., on its moisture content and the compaction pressure used. The contents of CO_2 determined for the centers and surfaces of the DS-5 and DS-6 samples, prepared at 4.5 MPa compaction pressure, turned out to be almost equal. This is explained by the relatively low density and, thus, higher porosity of the samples under consideration in comparison with the samples compacted at higher pressure values, which made it possible for the CO_2 to diffuse into all parts of the samples. The quantity of CO_2 absorbed from the experimental solutions after 1 h of carbonation was measured using the traditional calcimetric technique. Data on the quantity of absorbed CO_2 is shown in Table 4.

The secondary $CaCO_3$ contents have been tabulated in Table 5. It is clear from the data that the process of forced carbonation is quite efficient, and an accurate estimation of the secondary $CaCO_3$ amount has been carried out. A systematic study of the experimental results has shown that changes in the proportion of components of the raw materials affect the phase composition of the obtained $CaCO_3$ and microstructural formation. Out of 10 studied compositions, the composition DS-5, containing 25 wt.% defecate, 25 wt.% solid calcium-containing waste, and 50 wt.% quicklime, turned out to be the optimal composition following accelerated carbonation. The reason for choosing such a composition was that it facilitates the formation of highly developed marble-like $CaCO_3$ crystalline phases. Thus, the DS-5 sample was chosen as the reference optimal composition for further studies.

Table 3. Composition of the raw material mixtures prepared for accelerated carbonation

Experimental specimens	Pressing moisture content, %	Compaction pressure, MPa	Raw material name va samples, wt.% (mass %)		
			Defecate	Solid calcium-rich waste (Hydrated lime)	Quicklime (CaO)
DS -1	6	5.5	5	45	50
DS -2	6	5.5	10	40	50
DS -3	6	6.5	15	35	50
DS -4	6	6.5	20	30	50
DS -5	6	4.5	25	25	50
DS -6	6	4.5	30	20	50
DS -7	6	5.0	35	15	50
DS -8	6	5.0	40	10	50
DS -9	6	5.0	45	5	50
DS -10	6	5.0	50	-	50

Table 4. Quantitative indicators of CO_2 in the carbonated samples

Experimental samples	DS-1	DS-2	DS-3	DS-4	DS-5	DS-6	DS-7	DS-8	DS-9	DS-10
CO_2 content in the samples, %	23.5	30.0	32.5	38.0	42.5	41.5	39.1	36.0	31.0	27.5

Table 5. Comparative properties of the accelerated carbonated specimens

Experimental specimens	Compressive strength, MPa	Water absorption, %	DTA/TG mass loss (wt.%)	Main XRD phases, formed $CaCO_3$ content, %
DS -1	25.1	7.0	20	45.0 - Calcite ($CaCO_3$)
DS -2	25.5	7.0	25	59.0 - Calcite ($CaCO_3$)
DS -3	26.8	7.6	33	65.0 - Calcite ($CaCO_3$)
DS -4	26.9	4.5	39	78.0 - Calcite ($CaCO_3$)
DS -5	27.5	3.1	45	88.5 - Calcite ($CaCO_3$)
DS -6	27.3	4.8	41	80.1 - Calcite ($CaCO_3$)
DS -7	27.1	4.9	42	80.2 - Calcite ($CaCO_3$)
DS -8	27.0	5.2	39	73.0 - Calcite ($CaCO_3$)
DS -9	26.6	4.8	32	61.0 - Calcite ($CaCO_3$)
DS -10	25.9	4.9	29	54.0 - Calcite ($CaCO_3$)

Compression tests were performed by a universal testing machine (UTM) with a loading rate of 2.4 $kN \cdot s^{-1}$. Moreover, the flexural strength of specimens was determined according to ASTM C348. The choice of DS-5 composition as the optimal one is based on the quantitative assessment of the major physical and mechanical as well as mineralogical parameters of the ten specimens after accelerated carbonation, which are presented in Table 5. The DS-5 sample had the maximum value of compression strength which was 27.5 MPa. Moreover, the XRD results confirmed the formation of the most pronounced crystalline phases for the DS-5 sample following carbonation. Calcite ($CaCO_3$) is the main newly-formed mineral phase. Besides, the wollastonite ($CaSiO_3$) and akermanite ($Ca_2MgSi_2O_7$) phases, which were produced during the reaction of the silicate compounds of the starting raw materials, have been found. The existence of these silicate calcium-magnesium phases facilitates CO_2 sequestration process and increases the density and mechanical properties of the product.

While the mechanical properties of the DS-3 composition showed relative comparability with compressive strength being 26.8 MPa, the XRD analysis suggested that phase composition was not properly developed in the case of carbonation, with a portion of amorphous phase remaining within. On the contrary, the DS-1 and DS-2 compositions have shown mass loss from 20% to 25% along with compressive strengths of lower values. It means that the reaction of calcium compounds in the carbonation process is incomplete. Thus, the optimal composition of the DS-5 sample was chosen as the reference one for all further mineralogical, physico-mechanical, and structural studies. This sample demonstrated efficient carbon dioxide capture, resulting in the formation of $CaCO_3$, which became the major mineral component, and increased the density and mechanical properties of the sample.

X-ray diffraction analysis of the forced carbonated specimen

A marked rise in the intensity of the diffraction lines characteristic of the calcite phase was found in the X-ray diffraction spectrum of the forced carbonated sample. It proves that the reaction of CO_2 with the calcium minerals took place, resulting in the formation of the thermodynamically stable $CaCO_3$ phase. Additionally, the diffraction line at $2\theta = 26.6°$ proves the presence of the quartz phase. Carbonation resulted in an increase in the carbonate phases in the material's mineralogical composition. The diffraction pattern was measured from 5 to 70 degrees 2θ, and it contained several intense peaks, indicating that the sample is crystalline in nature. In amorphous substances, sharp peaks do not usually occur. The strongest diffraction peak exists at an angle of $2\theta = 29.4°$. It shows significantly more intensity

compared to the other diffraction peaks, implying that this is the main crystal structure component of the sample. This diffraction peak belongs to the (104) crystal plane of $CaCO_3$ (Figure 4).

FTIR analysis and discussion of the forced carbonated specimen

The FTIR analysis detected the bands that were characteristic of aluminosilicates and carbonates. The wide band detected at 3442 cm^{-1} corresponds to the vibration of O-H bonds in the water molecules and/or hydroxyl group. The strong absorption bands detected between 1100 and 1150 cm^{-1} belong to the asymmetric vibrations of Si–O–Si and Si–O–Al bonds. These absorption bands found at 1490–1410 cm^{-1} and 876 cm^{-1} correspond to the vibration of CO_3^{2-} groups, showing that calcium carbonate ($CaCO_3$) is formed as a result of the carbonation reaction. The bands less than 700 cm^{-1} are attributed to Si-O-Al bonds and M-O vibrations. The results obtained from FTIR analysis prove the successful carbonation process of the aluminosilicate sample, which proves the changes that were made in the structure due to the presence of CO_2. Moreover, it is clear from the spectrum data that the structure of the aluminosilicate sample was retained, whereas the formation of calcium carbonate caused the changes in the structure and morphology of the material (Figure 5).

Thermogravimetric analysis of the forced carbonated specimen

Thermogravimetric (TG-DSC) study of the forced-carbonated aluminosilicate sample showed several steps of the thermal transformation. The first stage of mass loss up to 200 °C can be explained by physical water evaporation. Mass loss in the 200–600°C range can be explained by the dehydration of hydrated aluminosilicates and the release of structural hydroxyl groups. Massive mass loss with significant endothermal effect in the 700–850 °C range can be related to the decomposition of carbonate phases generated during the fast carbonation process and described by the next chemical reaction: This thermal event confirms the successful mineralization of CO_2 and the formation of stable carbonate compounds within the aluminosilicate matrix. At temperatures above 900 °C, thermal stabilization of the specimen is observed, indicating the formation of thermally stable calcium aluminosilicate phases. The obtained results demonstrate that the forced carbonation process promotes the formation of stable carbonate phases capable of sequestering CO_2 within the aluminosilicate matrix. Furthermore, the produced material exhibits good thermal stability under high-temperature treatment conditions (Figure 6).

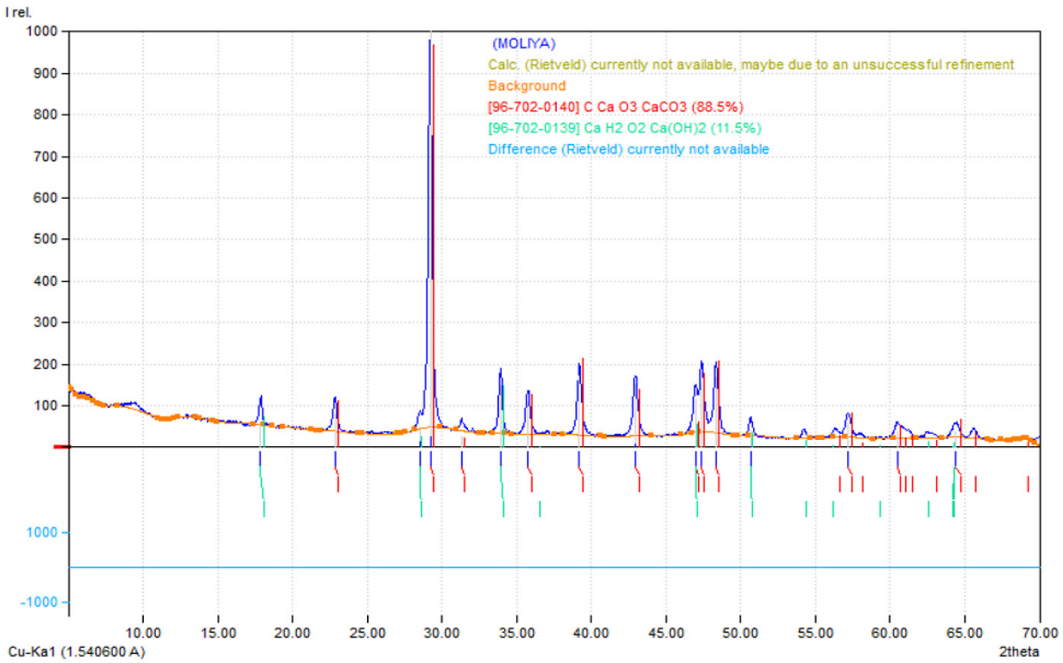


Figure 4. X-ray diffraction (XRD) patterns of the specimens

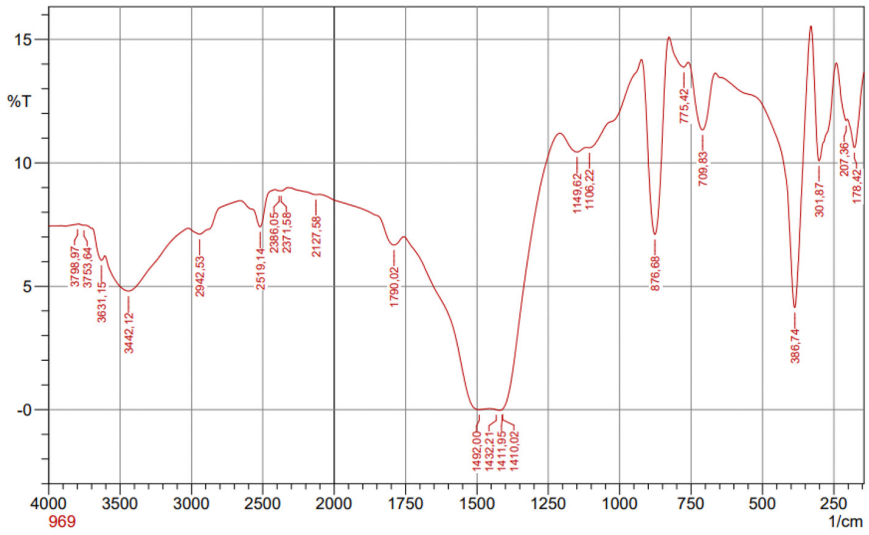


Figure 5. FTIR spectra of the specimens

SEM analysis of the forced carbonated specimen

The morphological studies on the composites were carried out by SEM analysis. EDX analysis was performed to study the distribution of elements. Pore size distribution and total porosity were determined by MIP analysis. The SEM–EDX analysis revealed that the prepared specimen is calcium-rich and possesses a porous microstructure characteristic of the forced-carbonation system. The high concentrations of Ca, C, and O confirm the extensive formation of calcium carbonate phases. The obtained results demonstrate that the calcium-containing components were effectively carbonated during the CO_2 mineral sequestration process, resulting in a denser material structure (Figure 7).

DISCUSSION

In contrast to prior research studies, the current study shows a unique method of carbonation of calcium-rich industrial wastes, combining sugar-industry defecate, hydrated lime residue, and CaO as the reactants of construction materials. Prior studies have focused mostly on single

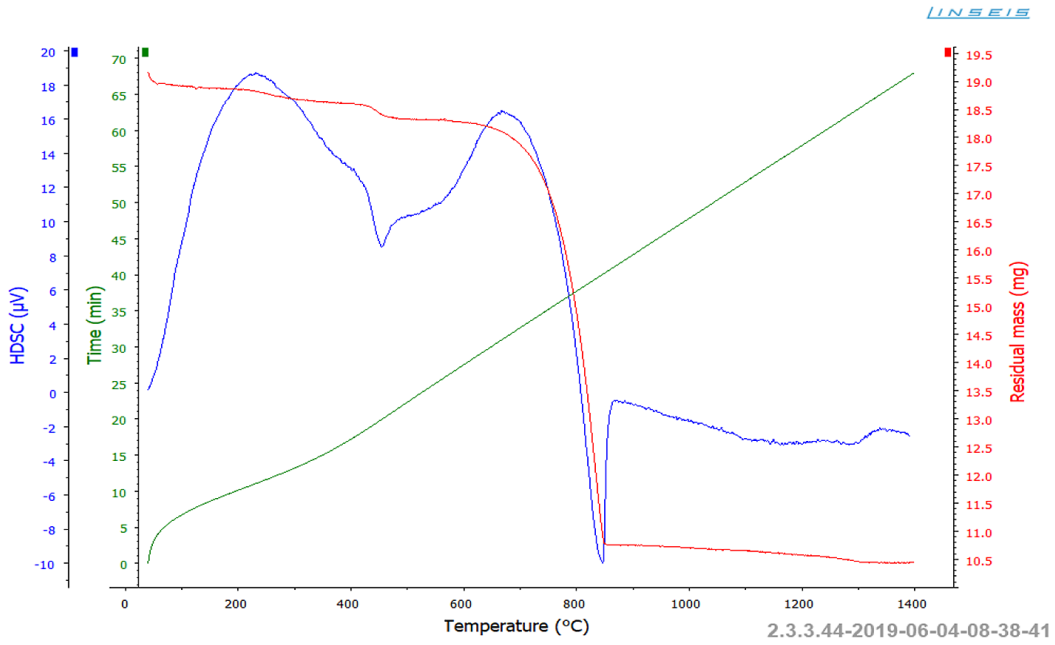


Figure 6. DSC/TG analysis of the specimens

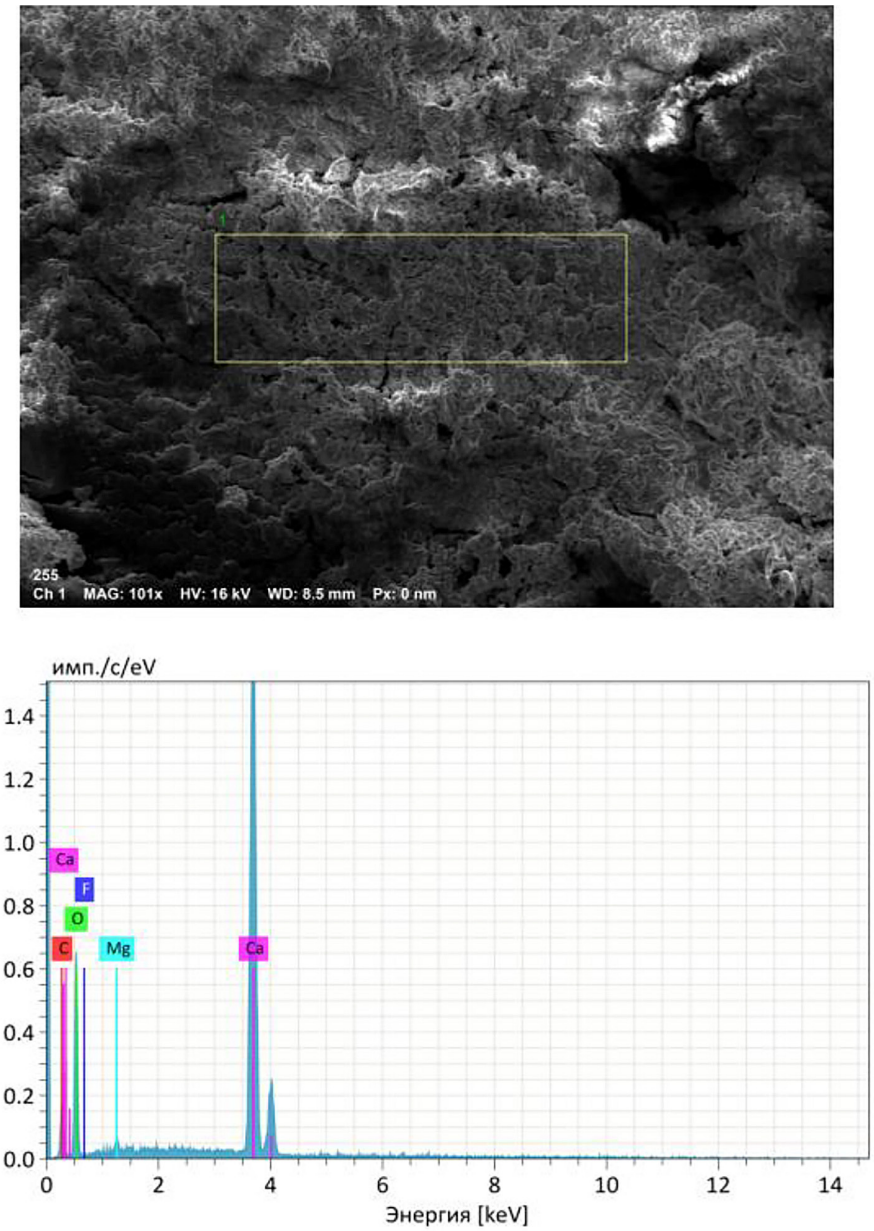


Figure 7. SEM images of the specimens

alkaline wastes such as cement kiln dust, steel slag, calcium carbide slag, or sugar-factory lime waste, while the combination of the use of two calcium-containing wastes from the sugar industry along with CaO as a CO_2 reactive material has been relatively little studied. Bahrami et al. showed that sugar-factory lime waste can be used for the creation of high-performance concrete, proving its potential as a useful secondary source of calcium rather than a waste material; however, their research was focused more on mechanical properties and environmental impact, not on CO_2 mineralization of the waste [44].

Likewise, Bahmani and Mostofinejad found that sugar-factory lime waste could take part in the preparation of sustainable construction materials with a minimum use of cement [45]. However, in the current research, the focus was made not only on the conversion of wastes into useful products but also on their application for CO_2 sequestration. The difference is that in the present case, apart from a process of treatment, carbonation also serves as a process for producing a cementitious mineral binder due to the reactivity of the components of the waste. Indeed, calcium-rich waste materials are characterized by the presence of reactive CaO and/or $Ca(OH)_2$, which allows forming thermodynamically stable $CaCO_3$ in reaction with CO_2, as shown before for cement kiln dust and other

alkaline wastes. It was found that in the current experiment, an increase in the content of defecate up to DS-5 led to an increase in the content of carbonates, whereas its further increase led to a decrease in CO_2 sequestration efficiency. In this regard, the optimum mixture of DS-5 with 25 wt.% defecate, 25 wt.% hydrated lime residue, and 50 wt.% quicklime was able to yield 42.5 wt.% CO_2 content, along with 88.5% $CaCO_3$ content and 27.5 MPa compressive strength, indicates the possibility of simultaneous carbonate formation and structural strength through CO_2 mineralization. It is not appropriate to compare the data from the current research to other studies that report their CO_2 uptake using different units and different waste materials, as it has been emphasized in the literature that CO_2 uptake measurements are highly sensitive to analytical techniques, material condition, moisture content, particle size, and carbonation procedure [46].

Ferrara et al. proved that different measurement techniques can provide CO_2 uptake measurements of carbonated BOF slag at around 13.3–17.0%. In this way, the current work increases the accuracy of the carbonation evaluation through the combined application of calcimetry as well as the data from the XRD, FTIR, and DTA/TG analyses on the formation of carbonates, unlike those based on the simple weight increase and a single method [47]. The well-visible calcite peaks formed upon the carbonation and the behavior of their thermal decomposition independently confirm that the captured CO_2 reacted and formed stable calcium carbonate structures. The fact that calcite is the most stable carbonation product in the calcium-containing waste materials is confirmed by previous studies on mineral-carbonation in calcium-rich industrial wastes. In this regard, the major scientific novelty of the current study is not just the use of calcium-rich material, but the combination of sugar industry waste utilization, mixture optimization, accelerated mineral carbonation, CO_2 quantification, phase analysis, and mechanical property testing in one technological process. With this approach, the DS-5 mixture that is produced appears to provide a promising solution to utilizing local calcium-bearing waste from the sugar industry in the fabrication of construction materials along with sequestration of CO_2 in mineral form, but additional studies are needed [48,49].

CONCLUSIONS

This work examined the possibility of transforming calcium-containing residues obtained in the process of sugar manufacturing into building materials. It was shown that forced carbonation, which is the procedure of accelerated mineralization of calcium-bearing industrial by-products, can substantially enhance their physicochemical characteristics, and thus, allow partially replacing traditional cement-containing building materials with them. Through optimization of the mixture composition, the optimal composition of DS-5, containing 25% defecate, 25% calcium-bearing hydrated lime residue (pushonka), and 50% pure quicklime, was found. The analysis of XRD and FTIR spectra showed that stable polymorphic forms of calcium carbonate, calcite and aragonite, are formed in the course of carbonation. Samples compacted at 25 °C, 6% moisture content, and 4.5 MPa pressure achieved a compressive strength of 27.5 MPa after forced carbonation. As far as the carbonation of solid wastes is concerned, those that contain high amounts of CaO have shown higher degrees of carbonation. However, the phase of minerals containing Ca is also significant. For compaction at a relatively low pressure of 4.5 MPa, a combination of high specific surface area and 6 % moisture content is considered suitable for carbonation. Successful use of sugar industry waste, such as defecate, hydrated lime, and pure quicklime as the main raw materials proves the high value of these substances in producing eco-friendly construction materials. The obtained results create a theoretical and practical foundation for upscaling the technology on a pilot scale and for assessing the strength and durability of the produced construction materials under real conditions of use. Such an approach will be useful for efficient usage of industrial waste, conservation of natural raw materials, reducing CO_2 emissions, and the development of circular economy principles in the construction materials industry.

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