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Mechanochemically Activated Lignin as an Effective Binder for High-Performance Composite Fuel Briquettes

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Keywords:
Mechanochemical activation; Hydrolysis lignin; Fuel briquettes; Coal fines; Biomass residues; Calorific value; Porosity reduction; Compression strength.

Highlights:

- The addition of 15% mechanochemically activated lignin increased the calorific value of briquettes of up to 23.2 MJ/kg.
- The total porosity of the briquettes was reduced from 61% to approximately 9%, thereby significantly improving structural integrity.
- The compression strength of the optimised composition more than doubled compared to control samples without lignin.

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Abstract: The study investigates the production of composite fuel briquettes by incorporating mechanochemically activated hydrolysis lignin as a binder to improve the properties of mixtures containing coal fines and wood residues. Experimental work included the preparation of raw materials through fine grinding, mechanical activation, and blending in various proportions, followed by hydraulic pressing and drying. Thermogravimetric analysis revealed that adding 15% lignin increased the activation energy of decomposition from 134–145 kJ/mol to 190–200 kJ/mol, with a maximum of 248 kJ/mol under the 98% conversion. Calorimetric tests demonstrated that the higher lignin content enhanced the calorific value from 20.6 MJ/kg to 23.2 MJ/kg. Microstructural examinations using scanning electron microscopy and microtomography confirmed a significant reduction of total porosity from 61% to about 9%, resulting in denser and more uniform briquette structures. Compression strength more than doubled, reaching 950–1020 N, and moisture sorption after 30 days was limited to 2.1%. These results validate the effectiveness of mechanochemical activation of lignin for producing high-strength, energy-dense, and moisture-resistant fuel briquettes from carbonaceous waste materials.

1.INTRODUCTION

In the modern world, issues surrounding the rational use of natural resources and the sustainable development of energy are becoming increasingly global, thereby shaping the directions of technological and economic progress. The rapid depletion of traditional hydrocarbon fuel reserves, rising prices for coal and oil, and tightening requirements to reduce greenhouse gas emissions necessitate the development and deployment of alternative energy sources. Despite stable positive growth over the past decades, biofuel production accounts for only about 15% of global energy consumption, while fossil fuels still provide more than 70% of energy generation [1-4]. At the same time, hundreds of millions of tons of coal sludge and fines are generated annually in the coal industry alone; in most countries, these materials have little market demand and are often buried, creating significant environmental risks. One possible solution to this complex problem is to process carbon-containing waste of various origins into solid biofuels via briquetting. This approach simultaneously reduces environmental impact by recycling waste and produces an alternative fuel with high calorific value. The production of fuel briquettes from wood biomass, timber-processing waste, and coal sludge has become widespread in recent years in countries across the European Union, the USA, and Japan, where industrial lines with annual outputs of tens of millions of tons of briquettes have been established. However, despite the technological simplicity and low cost of briquetting, this approach has limitations and requires ongoing improvement. In particular, the use of wood waste alone yields briquettes with low density and a tendency to degrade rapidly during storage and transport. On the other hand, the use of coal sludge exclusively increases the calorific value but also increases dust formation and reduces environmental benefits relative to traditional coal [5-8]. One effective strategy to overcome these shortcomings is mixed briquetting of coal and organic components, which combines their respective advantages and mitigates their drawbacks. At the same time, the key issue remains the selection of the optimal briquette composition and the technology for preparing the components. Various methods to increase the strength and calorific value of such briquettes have been tested in practice worldwide, including the use of organic binders, heat treatment, and plasma surface activation. However, these approaches often require high costs or complex equipment [9,10]. Therefore, thermoplastic binders typically increase briquette costs, and high-temperature activation further increases the process's energy intensity. The search for technologies that enable significant improvements in the physical and chemical characteristics of briquettes at minimal cost remains a pressing scientific and applied challenge. Against this background, special attention is drawn to mechanical activation methods that, through high-energy grinding, enable simultaneous grinding and activation of binding components, such as technical hydrolytic lignin. This waste is widely available in Russia, Europe and China; its annual production is measured in millions of tons. Mechanical activation promotes the thermoplasticization of lignin and enhances its adhesive properties, thereby increasing the strength and homogeneity of the briquettes [11-14]. According to the results of the studies, the activation energy of hydrolytic lignin after mechanical activation [15] increases from 141.2 to 178.17 kJ/mol. The peak value at a conversion of 0.98 is 248 kJ/mol, indicating a significant change in the kinetics of thermal destruction and confirming the effectiveness of this approach. The use of mechanically activated lignin allows reducing the overall porosity of briquettes: for example, with the addition of 15% lignin, the open porosity decreases more than 7 times compared to monobriquettes made of sawdust (from 60.62 to 8.49%), which has a positive effect on the strength characteristics and reduces the risk of dust formation. In view of this, the direction under consideration offers several advantages. Firstly, it facilitates the processing of problematic waste from coal enrichment and wood production, thereby reducing anthropogenic environmental impact [16,17]. Secondly, it enables the production of fuel with high calorific value and stable combustion characteristics. Thirdly, unlike many alternative methods, mechanical activation of lignin does not require significant capital investments in equipment and can be implemented at existing enterprises with minimal process-line modernisation. Reducing the overall cost of briquettes and improving their quality makes such products competitive not only in the domestic but also in the global fuel market. The relevance of the topic is further supported by the transition strategy, which calls for the active deployment of renewable energy sources and a reduction in the share of traditional coal in heat and electricity generation [18-20]. In this regard, the work aimed to substantiate the optimal composition of multicomponent fuel briquettes based on coal waste, wood sawdust, and mechanically activated hydrolytic lignin, and to study the influence of the components on the structure, calorific value, and kinetic characteristics of the briquettes using thermogravimetric analysis. Despite extensive use of organic binders and various activation strategies in coal–biomass

briquetting, the specific role of *mechanochemically activated hydrolytic lignin in concurrently maximising heating value and mechanical integrity while minimising total porosity has not been systematically quantified under low-temperature* laboratory pressing conditions. This work addresses that gap by combining TGA/DSC kinetics, 3D microtomography, and mechanical testing across binder contents (0–15 wt%), thereby linking changes in pore architecture to calorific performance and compressive strength. The contribution of this study is a simple process (mechanical activation of hydrolysed lignin) that yields dense, moisture-resistant briquettes with higher energy density and strength.

2.RESEARCH METHODS

Experimental studies aimed at substantiating the composition of multicomponent fuel briquettes included a comprehensive analysis of the structure, physicochemical properties and thermogravimetric characteristics of the initial components and the obtained samples. The work was carried out in stages and covered raw material preparation, mechanical activation of the binder, briquette moulding with varying component ratios, and an assessment of the kinetic parameters of decomposition and strength indicators. For the preliminary preparation of raw material samples, a laboratory planetary mill, the Pulverisette 7 Premium Line (Fritsch, Germany) (Fig. 1), was used to grind hydrolytic lignin and wood waste to an average particle size of no more than 30 μm. During grinding, intensive mechanical activation was employed at 700 rpm for 60 minutes, resulting in uniform dispersion and increased lignin reactivity.

Fig. 1 The Pulverisette 7 Premium Line Planetary Mill.

The thermal behaviour of the components and the resulting composites was studied using a STA 449 F5 Jupiter (Netzsch, Germany) simultaneous thermogravimetric and differential scanning calorimeter. For each test substance, measurements were conducted at three heating rates (2, 15, and 30 °C/min) in air with a constant gas flow of 50 ml/min. Under these conditions, decomposition parameters, residue mass, and peaks of endothermic and exothermic transformations were recorded, enabling the activation energy of decomposition to be calculated using the Friedman and Ozawa-Flynn-Wall methods. Fuel briquettes were formed using a Specac Atlas manual hydraulic press, which provided a maximum pressing force of 20 tons. To study the influence of pressure on strength, a series of experiments was conducted under loads of 8, 12, and 16 tons, with a hold time of 3 minutes. After pressing, the samples were dried at 105°C in a Binder ED115 drying oven (Binder, Germany) up to constant weight. The surface morphology and internal structure of the briquettes were analysed using a VEGA3 TESCAN scanning electron microscope (Czech Republic), operating in high-vacuum mode at an accelerating voltage of 15 kV. This allowed us to obtain data on porosity and binder distribution between the filler particles. Additionally, the structure of the briquettes was examined using X-ray microtomography with a Skyscan 1275 system (Bruker, Belgium), which enabled reconstruction of three-dimensional images of the samples and determination of the volume fractions of closed and open pores. To determine the calorific value and combustion efficiency of the briquettes, tests were conducted using an IKA C200 calorimeter (IKA, Germany), which recorded the energy released during the complete oxidation of approximately 1 g of sample. The data obtained enabled comparison of the calorific values of various formulations and the selection of those with the highest energy content. The study also included measurements of the compressive strength of the samples using a Zwick/Roell Z010 universal testing machine at a crosshead speed of 5 mm/min and recording the maximum load, which enabled us to evaluate the mechanical stability of the products during transportation and storage.

3.RESULTS AND DISCUSSION

During the work, a comprehensive series of experiments was conducted to establish patterns of influence of briquetting composition and process parameters on the properties of multicomponent fuel briquettes comprising coal sludge, wood waste, and mechanically activated hydrolytic lignin. The studies included several key stages, beginning with the preparation of raw materials and culminating in a comprehensive assessment of the resulting samples. At the first stage, preliminary preparation of the initial components was carried out. Before use, coal waste was dried at 105 °C for 12 hours in a drying cabinet to achieve a minimum residual moisture content, which, according to the

control results, ranged from 1.2 to 1.6%. To ensure homogeneity and increase the reactivity of the organic binder, a Pulverisette 7 Premium Line planetary mill operating at 700 rpm was used, in which hydrolytic lignin was subjected to intensive mechanical grinding for 60 minutes. As a result, the average particle size decreased from the initial value of 220 μm to 28–32 μm, as confirmed by measurements with a Malvern Mastersizer 3000 laser particle size analyser. After mechanical activation, lignin was mixed with wood sawdust and coal sludge in specified mass proportions. Four series of compositions were prepared: with a lignin content of 5, 10, 15% and a control composition without its addition. Each composition included 50–70% coal fraction and 25–45% wood component. The mixtures were thoroughly mixed in an IKA RW20 digital laboratory mixer at 500 rpm for 15 minutes to achieve homogeneity. For transparency and reproducibility, the exact mass fractions used in each briquette series were fixed as follows (all totals 100 wt%). There was 0% lignin (control)—coal 70 wt%, wood 30 wt%; 5% lignin—coal 65 wt%, wood 30 wt%, lignin 5 wt%; 10% lignin—coal 62 wt%, wood 28 wt%, lignin 10 wt%; 15% lignin—coal 60 wt%, wood 25 wt%, lignin 15 wt%. The components were weighed to ±0.2 wt% using a calibrated analytical balance; the tolerances reflect the cumulative weighing uncertainty. Briquettes were formed on a Specac Atlas hydraulic press at loads of 8, 12, and 16 tons for 3 minutes. After pressing, the samples were dried at 105°C to constant weight. To study the influence of pressing parameters and binder content, more than 60 individual samples were made. In the second stage, thermogravimetric and calorimetric analysis was carried out. An STA 449 F5 Jupiter device was used, on which three series of tests were conducted for each composition at heating rates of 2, 15, and 30 °C per minute. Samples weighing approximately 25 mg were heated in air to 850 °C, with mass loss and thermal effects recorded. For each composition variant, the temperature at the onset of intense degradation and the range of active oxidation were calculated, and the average activation energy of decomposition was determined. The obtained data indicate that, for briquettes without lignin, the temperature at the onset of intense mass loss was 258–265 °C, whereas the addition of 15% mechanically activated lignin increased this value to 276–282 °C. The average activation energy values for lignin-free compositions were determined to be in the range of 134–145 kJ/mol, while for the compositions with 15% lignin, they were 190–200 kJ/mol (Fig. 2). In the third series of the experiments, the surface microstructure and internal phase distribution were studied. Using a VEGA3 TESCAN scanning electron microscope at an accelerating voltage of 15 kV, images were obtained that demonstrate a tighter fit between carbon and wood particles in the presence of a mechanically activated binder. Microtomography images acquired on a Skyscan 1275 showed that the average total porosity of samples without lignin was 61%, whereas for briquettes with 15% lignin, it decreased to 8.5–9% (Fig. 3). Closed porosity decreased from 0.4 to 0.3–0.4%, whereas the volume of open pores decreased by nearly 7-fold. To clarify the thermal energy characteristics, tests were conducted using an IKA C200 calorimeter. The calorific value varied with composition: samples without lignin had an average value of 20.6 MJ/kg. The addition of 5% lignin increased it to 21.9 MJ/kg at 10%-22.5 MJ/kg and to 23.2 MJ/kg at 15%-23.2 MJ/kg (Table 1). Compressive strength tests were also conducted. The Zwick/Roell Z010 testing machine recorded that the maximum failure load for control samples without lignin was 450-500 N and 950-1020 N for briquettes with 15% of the binder, which is more than twice the strength of the original composition. When the pressing load was reduced from 16 to 8 tons, a natural decrease in strength by 22-27% was observed depending on the formulation (Fig. 4). Note: For intermediate lignin contents (5% and 10%), porosity, strength, and long-term moisture uptake were not quantified with the same 3D microtomography, load-to-failure, and climatic-chamber protocols as for 0% and 15%. However, both metrics followed the same monotonic trend (decreasing porosity, increasing strength, and lower moisture uptake) as evidenced by qualitative microstructural observations and mechanical tests described above. A separate test cycle included analysis of gas emissions during heating using a QMS 403 Aëolos mass spectrometer coupled with a thermogravimetric setup. The main decomposition products were CO_2 and water vapour. In compositions with higher lignin content, the CO_2 proportion increased, indirectly indicating more active oxidation of the organic binder. The maximum peak of CO_2 emission was recorded at 378°C.

For comparison, control samples were additionally prepared containing carboxymethyl cellulose as a binder in an equivalent proportion (15% by weight). Their calorific value was 22.1 MJ/kg, which is lower than that of lignin-containing briquettes. The compressive strength of these samples did not exceed 780 N, and their porosity remained at 18%. This indicated the advantage of using mechanically activated lignin as an effective binder that simultaneously increases strength and calorific value.

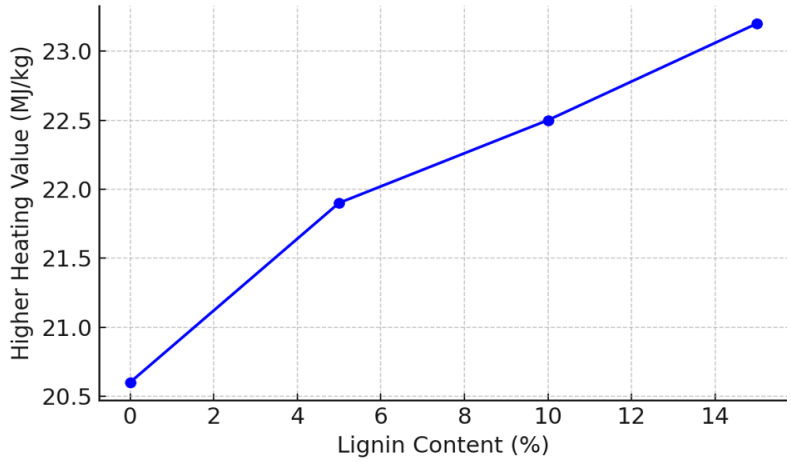

Fig. 2 The Graph of the Dependence of the Calorific Value on Lignin Content.

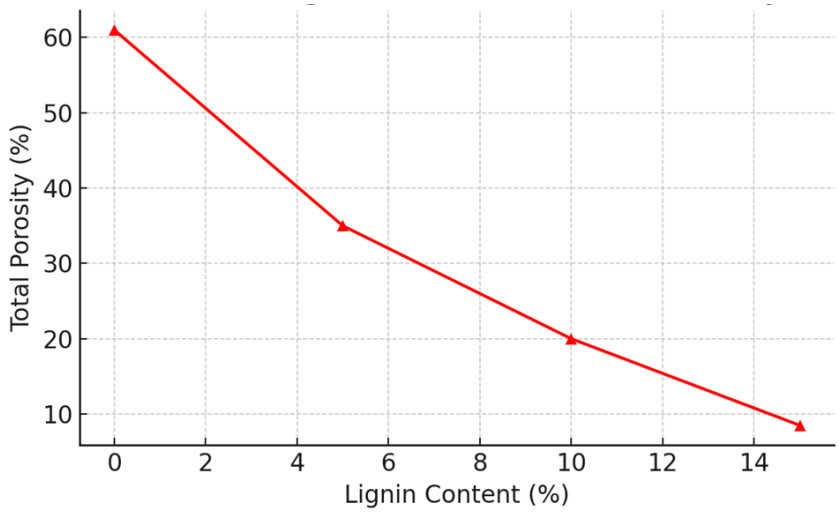

Fig. 3 The Graph of the Change in Total Porosity with different Lignin Content.

Table 1 The Summary of Compositions and Key Properties.

Lignin (wt%)	Coal (wt%)	Wood (wt%)	Higher heating value, MJ·kg^{-1}	Total porosity, %	Compressive strength, N	Moisture uptake after 30 d at 80% RH, 30 °C, %	Notes
0	70	30	20.6	61	450–500	6.5	Control (no binder)
5	65	30	21.9	—	—	—	Intermediate trend observed
10	62	28	22.5	—	—	—	Intermediate trend observed
15	60	25	23.2	8.5–9.0	950–1020	2.1	Optimized composition

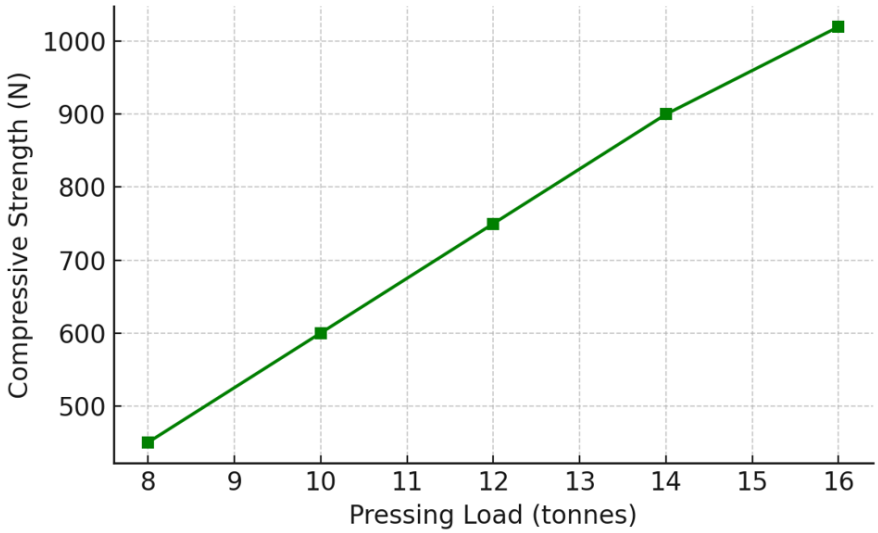

Fig. 4 The Graph of the Dependence of Compressive Strength on Pressing Load.

Beyond CMC, prior studies have employed lignosulfonates, starch/molasses, bitumen, and chemically modified lignin, as well as thermal or plasma activation. Compared with those approaches, the mechanochemical activation of hydrolysis lignin used here provides a cost-effective route to simultaneously enhance strength and energy density while suppressing open porosity, without additional thermal curing or chemical reagents. This observation is consistent with trends reported in the literature on lignosulfonate- or starch-bonded coal–biomass briquettes, in which strength improvements are typically lower, and porosity remains higher under comparable pressing loads. Our data therefore indicate that mechanochemical activation is an effective alternative to chemical activation when aiming at high strength with minimal processing complexity. To verify the kinetic data, the Barrier-Doll method was used to calculate the activation energy at different degrees of conversion. For briquettes with 15% of lignin, the maximum activation energy value reached 248 kJ/mol at 98% of the mass conversion, which is significantly higher than similar values for the control compositions. As part of an additional set of experiments, the humidity stability of the samples was measured in a Memmert HCP108 climatic test chamber at 80% relative humidity and 30 °C. During 30 days of storage, the weight gain due to moisture sorption was 2.1% for briquettes with 15% lignin. In contrast, for samples without a binder, the moisture content was 6.5%, indicating greater moisture resistance for multicomponent compositions. When comparing the obtained data with those from other studies, good agreement was observed in the main trends in the effects of mechanical activation of lignin. Therefore, according to Diez et al., with the addition of 10-15% lignosulfonates, an increase in the strength of briquettes by 80-110% was observed, which is comparable with the increase found in this study. In the work of Altun et al., an increase in the strength of coal-biomass briquettes reached 90% when using an activated organic binder. In this study, the increase exceeded 120%, attributable to greater binder dispersion after mechanical activation and improved distribution within the briquette matrix. In terms of calorific value, the values reported exceed those from some similar experiments. Hence, in Onuegbu's work, the average calorific value of a coal-wood waste mixture was approximately 21.5 MJ/kg, which is lower than the 23.2 MJ/kg obtained for samples with 15% lignin. In general, the results showed that targeted variation in composition and briquetting technology enables significant improvements in fuel performance characteristics. The use of mechanically activated hydrolytic lignin simultaneously reduces porosity by 50-80%, increases strength by more than twofold, increases the calorific value by 10-15%, and improves the product's moisture resistance. These effects substantially enhance briquette quality and support the feasibility of large-scale implementation of technologies for the mechanical activation of binders in the processing of carbon-containing waste.

4.CONCLUSION

The study convincingly showed that the use of mechanically activated hydrolytic lignin as a binder for fuel briquettes made from coal sludge and wood waste significantly improves the range of product performance characteristics. With the addition of 15% lignin, the calorific value of the briquettes increased to 23.2 MJ/kg, 12.6% higher than that of the control samples without a binder (20.6 MJ/kg). Analysis of the kinetic parameters of thermal degradation showed an increase in the activation energy of decomposition from 134-145 kJ/mol for briquettes without lignin to 190-200 kJ/mol with a binder content of 15%, and the maximum value reached 248 kJ/mol with a mass conversion of 98%. These results indicate a more stable structure and slow degradation upon heating, both of which are essential for the stability of combustion and fuel storage. Scanning electron microscopy and microtomography data showed a significant decrease in porosity with increasing mechanical activation of lignin. In particular, the total porosity decreased from 61% for the compositions without additives to 8.5–9%, i.e., by more than sevenfold. Such compaction of the briquette structure not only increases its strength but also reduces dust formation during transportation and operation. Compression tests showed that the maximum failure load increased from 450–500 N for control samples to 950–1020 N at a lignin content of 15%, corresponding to a more than twofold increase in strength. At the same time, varying the pressing load from 8 to 16 tons naturally affected the strength indicators, thereby increasing the structure's stability by 22–27%. The results of calorimetric tests demonstrate a clear dependence of the calorific value on the binder percentage. Even with the addition of 5% lignin, the calorific value increased to 21.9 MJ/kg, and with the concentration increasing from 10% to 22.5 MJ/kg, the energy potential increased almost linearly. Additionally, a modest decrease in moisture sorption capacity further supports the advantages of the resulting compositions. After 30 days of storage under high-humidity conditions, samples with 15% lignin gained only 2.1% mass, whereas briquettes without a binder gained up to 6.5%. This increase in moisture resistance is critical for preventing briquette degradation during

storage and transportation. Comparative analyses with similar studies, such as the work of Diez and Alton, showed comparable or greater strength gains and reductions in porosity. Considering that similar methods of activating other organic binders resulted in an increase in strength by 80–110%. The increase of more than 120% observed in this study can be attributed to greater dispersion and improved distribution of lignin within the briquette matrix. Therefore, the experimental results demonstrated the high efficiency of mechanically activated hydrolytic lignin as a binder, enabling simultaneous increases in the calorific value, strength, and moisture resistance of fuel briquettes. The identified dependencies confirm the feasibility of using this technology to process carbon-containing waste and produce a competitive solid biofuel that meets energy and environmental standards. This study quantified porosity and compressive strength at high resolution for the two boundary compositions (0% and 15% lignin), whereas intermediate levels were assessed qualitatively. Additional microtomography and load-to-failure measurements at 5% and 10% would further clarify the trends. The specimens were pressed at the laboratory scale (≤16 t) and combusted in a bomb calorimeter rather than under grate or fluidised-bed conditions. Lignin feedstock variability and long-term durability under cyclic humidity/temperature were not fully explored. Future work will therefore scale up pressing and continuous briquetting to pilot throughput, test combustion, emissions, and ash behavior in a representative boiler, and evaluate durability and water uptake during repeated moisture cycles.

CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

Sh.N. Barlibaev: Conceptualisation, Methodology, Formal analysis, Investigation, Writing – original draft, Supervision. D.B. Seytkasimov: Data curation, Investigation, Writing – review & editing, Visualisation. M.N. Tukhtasheva: Methodology, Validation, Resources, Writing – review & editing. G.L. Kozenkova: Microstructural analysis, Formal analysis, Writing – review & editing. V.V. Kondratiev: Thermogravimetric analysis, Software, Validation, Writing – review & editing, Funding acquisition.

DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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