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10.2298/HEMIND220211011J

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Abstract

This paper presents the results of experiments performed to determine how the quantity of the binder (bentonite) and the parameters of the laboratory roll press affect the quality of the briquettes obtained from limestone powder. These experiments aim to examine the conditions in which limestone briquettes are formed and to determine their use for agricultural purposes. During the experiments various mass fractions of bentonite was added to limestone (from 1 to 10 %), while the force of roll press drums ranged from 2 to 25 kN. The briquettes have been tested by applying scanning electron microscopy (SEM), differential thermal and thermo-gravimetric (DTA/TG) analyses, X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR). Bentonite distribution was found to be uniform within the compact briquette structure. Formation of new compounds was not evidenced. The experiments also demonstrated that during briquetting, limestone properties remain unchanged, the changes are only physical, water solubility is not reduced, mechanical properties (impact resistance, compressive strength and abrasion resistance) are satisfactory by the transport and storage terms if the binder mass fraction is over 5% and the briquetting force exceeds 10kN and finally there is no loss due to wind dispersal during application. The only downside of the "green" briquettes obtained is the time required for their complete disintegration if totally immersed in water.

Keywords: bentonite; briquette roll press; green briquette; briquette properties.

Available on-line at the Journal web address: http://www.ache.org.rs/HI/

ORIGINAL SCIENTIFIC PAPER

UDC: 552.541:622.788:005.935.33

Hem. Ind. **76 (2)** 97-107 (2022)

1. INTRODUCTION

Limestone, a salt of calcium and carbonic acid, is a naturally occurring mineral [1]. It appears throughout the world and it is crucial raw material used in various industries [2]. As reported, there are approximately 300 various applications of limestone and further on, processed limestone is a base for numerous other products [3]. One of them is the limestone use for agricultural purpose to adjust the soil acidity.

In low pH environments, abutment of heavy metals is dissolved and released to the soil, which can be toxic to plants [4-5]. Acidity of the soil (pH) has an impact on availability of most heavy metals [6]. Liming efficiently improved soil chemical composition [7]. Limestone is commonly applied for treatment of acidic soils in agriculture and forestry to boost yield and product quality [8]. Soil acidity reduction and higher levels of ex-changeable Ca and Mg was achieved during a ten-year soil liming field experiment, by treating the soil with powdered limestone and finally resulting in higher green grain yield [9-11].

Calcium-oxide (CaO) is a compound with greatest impact on the limestone quality [3]. By adding CaO into soil, the heavy metal-induced toxicity is significantly reduced, and the yield is increased. After treating the soil with limestone, calcium reacts with carbon dioxide and water from the soil, depositing Ca and Mg carbonates. Further on, this triggers a reaction with the acidic colloidal complexes, where Ca and Mg are replacing hydrogen and Al. These reactions produce

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Paper received: 11 February 20212 Paper accepted: 9 May 2022; Paper published: xx May 2022.
https://doi.org/10.2298/HEMIND220211011J

emission of carbon dioxide and higher pH value of the soil to a satisfactory level [12]. By increasing the soil pH metal mobility is reduced so that limestone can be designated as a stabilization agent [13-17].

In agricultural application, limestone should be sufficiently fine as to be dissolved by the effects of weathering and spread evenly on the soil. However, problems arise during transportation and handling when this powdery material is easily dissipated and dispersed by wind from land surface. Thus, it is necessary to consolidate the powdered limestone, to meet both requirements. Common approaches are pelletizing and briquetting, resulting in suitable material size, easy for transport, handling, and use [18]. Pelletized nutrition is already used for application of urea. Experiments with urea briquettes pointed to significantly ($p < 0.05$) increased grain yields, nitrogen uptake, and nitrogen recovery compared to broadcast urea [19].

Briquettes can be produced by high load compaction, during which a bulk solid is transformed into a new structure with physical properties different from the original ones [20]. It is desired to control agglomeration of powdered materials in order to improve physical properties such as density, homogeneity, strength, compressive strength, shape, appearance, etc., and increase product quality [18]. The required values are determined by standards, norms or needs and demands of individual customers. Which method of agglomeration will be chosen depends on the type of material and on the target properties of the agglomerate; pelletizing (agglomeration by rotation), briquetting (agglomeration by pressure) or sintering (agglomeration by thermal treatment) [18,21].

The technologically simplest methods for limestone briquette formation have been the matter of extensive studies and presented in numerous literary sources [18,21,22]. The most important parameter for agglomeration strength is adhesion force between the particles that form the agglomerate [24] and different adhesion mechanisms have been studied [25]. Since the adhesion forces may differ, with sufficiently large separating forces (*e.g*. elastic reactive forces, current or frictional or impact forces) the formation of agglomerates is possible only when these, adhesion forces, are sufficiently greater than the separating ones [26]. Therefore, briquettes are dried to achieve the necessary strength. Usually, the quality of produced briquettes is in relation to their application (testing properties are shape, size, strength, porosity, and surface area) [27].

The main aim of this work was to examine conditions for production of limestone briquettes with the addition of bentonite as a binder and to determine adequacy of their use for agricultural purposes. Tests were conducted on the assumption that bentonite, as a highly viscous binder will not produce negative effects on the soil quality as confirmed in literature [26,28]. Briquetting conditions were investigated with the goal to form briquettes of the same structure and solubility as the initial crushed limestone, and that will not be prone to dispersal and loss during application, while exhibiting mechanical properties adequate for transport and storage. In addition, the tests aimed at production of “green” briquettes. The term ‘’green’’ briquettes is used to indicate that the resulting material does not contain ingredients that are harmful to the environment, i.e., the resulting briquettes should be eco-friendly.

2. MATERIALS AND METHODS

2. 1. Briquette production

For experimental tests, a limestone sample was taken from the lithothamnium limestone deposit at the Dobrilovic Site near Loznica (Serbia). Bentonite, composed predominantly of the mineral smectite and originating from the Sipovo Deposit (Bosnia and Herzegovina) was used as a binder.

A series of detailed tests were performed with crushed lithothamnion limestone to examine the briquette forming conditions. The tests were conducted in three phases:

- Phase 1 - forming briquettes with different binder mass fractions and applying different briquetting forces,
- Phase 2 – determining limestone structural changes when transformed into briquettes,
- Phase 3 – examining other limestone properties essential for liming acid soils. Each testing phase was conditioned by the positive results of the previous phase.

Briquetting was performed with the B050 Laboratory Roller Press (Komarek, US) with specifications as follows: roll diameter - 100 mm, roll width - 38 mm, roll speed – variable from 0 to 7,5 rpm, roll separating force – variable up to 50 kN, feed screw speed – variable up to 137 rpm, variable throughput up to 20 kg h^{-1}, total power installed – 1.4 kW.

The briquetting process was conducted by applying different forces (in the range of 2, 5, 10, 15, 20 and 25 kN) and different binder contents (in the range of 1; 2.5; 5 and 10 %), while keeping constant the other process and press parameters. The initial amount of each sample (limestone) was 500.0 g. Different amounts of binder (bentonite) were added, in the range of 1; 2.5; 5 and 10 % related to the initial amount of the limestone. The roller press itself contains a vessel with a mixer that serves to uniformly distribute the binder within the material. Limestone and the binder are first added to that vessel which provided mixing until the mixture was completely homogenized. During the homogenization process, water was continuously added up to a maximum of 10 % related to the total sample amount. Then the homogenized sample was transferred *via* a screw conveyor to the press section in which the sample was briquetted. Once the so-called "green" briquettes were withdrawn from the press they were left in air for the next 24 h. Following this method, 24 sets of briquettes were made each with different (combined) characteristics (the briquetting force and the binder content).

Briquette groups obtained under extreme conditions were selected for characterization: briquettes formed with the lowest binder content (1 %) and at applying the lowest force (2 kN) and briquettes formed with the highest binder content (10 %) and at applying the highest force (25 kN).

2. 2. Briquette characterization

2. 2. 1. Thermal analysis

The STA 409 EP (Netzsch, Germany) was used for differential thermal and thermo gravimetric analysis (DTA, TG). The samples were thermally treated in air atmosphere, in a temperature range 20 to 1000 °C, at a heating rate of 10 °C min^{-1}. The sample α-Al_2O_3 was used as reference sample.

2. 2. 2. Fourier transform infrared spectroscopy

Infrared spectroscopic tests were performed with the Nicolet IS-50 (Thermo-Fisher Scientific, US). The attenuated total reflectance (atr) technique was used for measurements in the range between 4000 and 400 cm^{-1} with 32 scans at a resolution of 4. After completed measurements it was necessary to make two corrections: the atmospheric correction for the removal of gas signals (CO_2 and H_2O) and automatic baseline correction.

2. 2. 3. X-ray diffraction analysis

A PW1710 X-Ray Diffractometer (Philips, Netherlands) with curved-graphite monochromator and scintillation counter was used to monitor the phase composition of the samples applying X-ray diffraction analysis.

2. 2. 4. Scanning electron microscope analyses

Scanning electron microscope (SEM) analyses were performed with the JSM-6610LV Scanning Electron Microscope (Jeol, Japan) equipped with INCA Energy Dispersive X-Ray Spectrometer (EDS).

2. 2. 5. Impact resistance

Impact resistance was tested on a set of 10 briquettes each having a total weight of 100 g. Impact resistance testing was performed by 25 consecutive releases of the briquette sample from the height of 457 mm on a steel plate 9-mm thick, after which the briquette sample was sieved on a 2-mm mesh screen and the mass of screen undersize was weighed [29]. This means that the mass of broken briquette particles lower than 2 mm in size is weighted. The results are presented as a mass percentage with respect to the initial sample mass and should not exceed 10 % of the total initial weight.

2. 2. 6. Compressive strength

Compressive strength was tested on a group (set) of 10 pellets on a laboratory press (Tonindustrie, Germany), to determine the maximum pressure that the pellet can withstand without breaking. These tests were performed according to the procedure of the company Mars Minerals (US), which is a designer and manufacturer of agglomeration equipment and systems and has been the pioneer in this field [29], which recommended that pellets should withstand a minimum of 0.5 kg / pellet, considered satisfactory for further manipulation.

2. 2. 7. Abrasion resistance

Abrasion resistance was tested by sieving a group of pellets weighing 100 g each on a mechanical laboratory sieving device Analysette 3 (Fritsch, Germany) using a sieve of appropriate mesh size (depending on the tested size class) for a period of 5 min. After that, the mass of the screen undersize that arise due to mechanical abrasion is determined, which should not exceed 5% of the total mass of the sample.

2. 2. 8. Disintegration

Briquette disintegration (complete disintegration) time when immersed in water at room temperature was tested by placing 3 briquette samples from each group into water. The disintegration is determined visually.

2. 2. 9. Limestone solubility

Solubility tests were performed to determine the solubility of various limestone products in water and to determine the possible impact of agglomeration processes on the solubility process. The following were used in the tests: analytical balance, platinum cups, water bath, laboratory oven and hotplate. The experiment is performed by dissolving 6.0000 g (weight) of the sample in 100 cm^3 of hot distilled water and cooking for 1 min. When the suspension has cooled, the solution was separated from the precipitate by squeezing through a filter paper (blue tape). Two tests were performed for each sample. Platinum cups are dried at 105 °C in an oven and cooled in a desiccator for 1 h and measured to constant weight (m_1). 50 cm^3 of the solution is poured into a platinum cup (which mass is stable), evaporated in a water bath, and then the platinum cup is dried in an oven at 105 °C, cooled for 1 h in a desiccator and measured on the analytical balance to constant mass (m_2). Thus, the difference in two measured mass values presents the sample fraction soluble in water. After the measurement, the mass percentage of the fraction soluble in water (S_{H2O}) is determined according to the equation (1):

$$S_{H_2O} = \frac{2(m_2 - m_1))}{6000} 100 \quad (1)$$

where m_1 and m_2 are masses expressed in g.

2. 2. 10. Wind dispersal

Tests of the influence of wind on the distribution of materials of 2 different limestone products were performed, *i.e.* limestone powder and briquettes.

Three 100-g samples taken from each group (limestone powder and limestone briquette) were, at different wind speeds of 1, 3 and 5 m s^{-1} uniformly discharged from a height of 60 cm to a 50×70 cm container, for a period of 4 minutes per each test. Wind was simulated by the axial fan type A.B.V.E - 3.5 (capacity 3600 m^3 h^{-1}, 780 rpm) (Tvornica ventilacijskih uređaja, Croatia) and wind speed was measured using anemometer CFM Metal Vane Anemometer SN: Q617422 (Extech, US). After every test the weight of the material from the container was measured. During each test the weather elements were also measured: temperature and humidity were measured by using the sensor ST-321S (Standard Instruments, China) and air pressure was measured by using Barolux A 9896 (R. Fuess Berlin-Steglitz, Germany).

3. RESULTS AND DISCUSSION

Limestone in the form of briquettes, used for testing is shown in Figure 1.

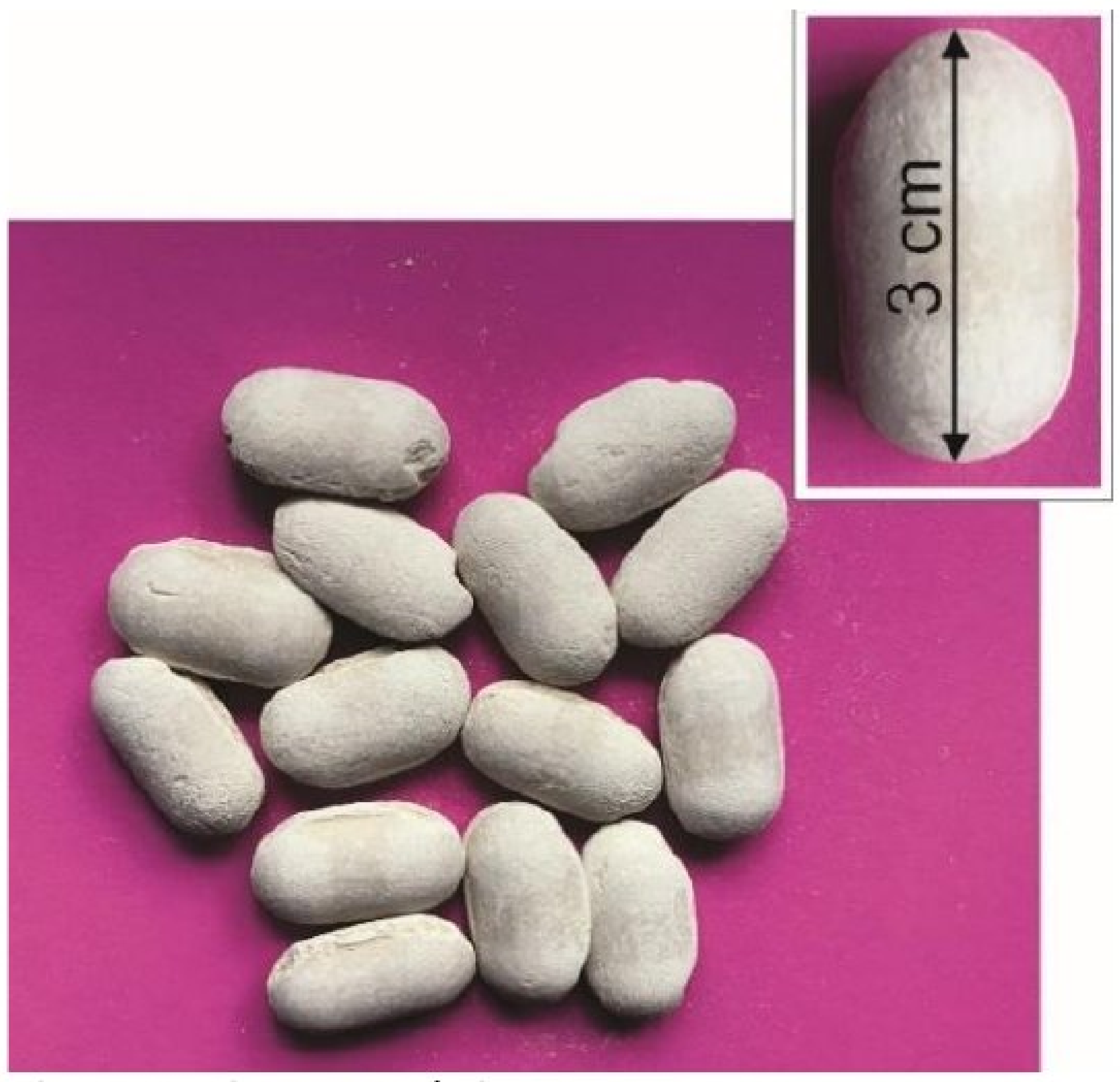

Figure 1. Limestone briquettes

To produce a usable briquette, limestone must not undergo any changes that might disturb its structure or hinder its ability to raise the alkalinity of soil. For characterization, we selected the briquette groups obtained under extreme conditions – briquettes formed with the lowest binder content and by applying the lowest force (1 % of bentonite at 2 kN) and on briquettes formed with the highest binder content at the highest force (10 % of bentonite at 25 kN). The briquettes were analysed by applying SEM, DTA/TG and FTIR analyses to determine possible changes in the structure.

The results of scanning electron microscopy (SEM) analysis are shown in Figure 2, which comparatively presents the SEM micrographs of samples at different magnifications. Despite the large difference in the binder mass fraction, the SEM micrographs do not reveal any difference between the two sample groups. This can be explained by the fact that the initial material is fine-grained, and the sample is well homogenized with the binder.

DTA curves of briquettes with different bentonite contents are shown in Figure 3a, which presents typical limestone peaks without major displacements. With the higher bentonite content, the peak intensity typical for limestone gradually decrease. Due to the low bentonite content and considerably lower peak intensity, typical bentonite peaks were not detected. Also, results of TG analyses (Figure 3b) indicate negligible difference in mass as compared to the initial limestone sample. The reason for this finding is a relatively low mass fraction of bentonite. From the TG diagram it can be observe that there is insignificant mass loss in the temperature range up to 100 °C, which corresponds to the water loss. These results indicated that after the drying process the remaining moisture content in briquettes is around 1 wt.%.

The results shown in Figure 3 are indicating that the application of bentonite as a binder in limestone briquetting at various mass fractions does not have an impact on the briquette thermal properties in comparison to the initial limestone sample.

Figure 4 shows the results of the infrared spectroscopy analysis (FTIR).

As shown in Figure 4 in the infrared spectra of briquette samples, in addition to the spectral lines characteristic for limestone, with unchanged intensities and positions, it is possible to observe bands typical for bentonite at 993 cm^{-1} whose intensity increases with higher bentonite content. The absence of either new spectral lines or major displacements of positions typical for spectral bands in the spectra of the analyzed samples (Fig. 4), indicate that the structural properties of the initial samples have not changed [30].

The results of all shown tests are confirming sufficient homogenization of bentonite and limestone, without any impact of the process (briquetting) and binder on limestone structural properties. These facts confirm that the briquettes can be equally successful for liming acid soils as limestone powder or limestone lumps and can be used interchangeably.

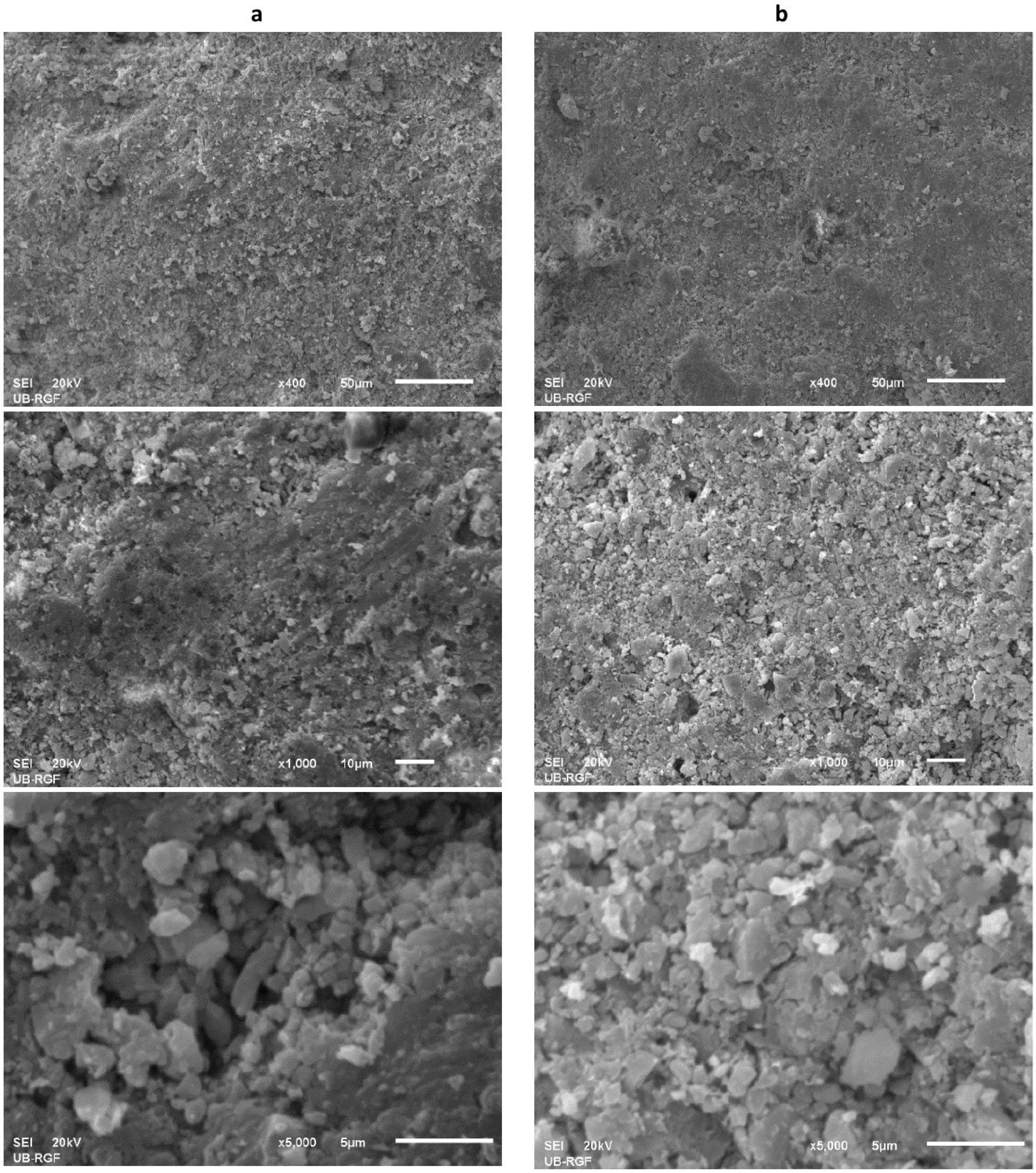

Figure 2. SEM micrographs of briquette samples: (a) with the lowest binder content (1 %) at the minimum briquetting force (2 kN) and (b) with the highest binder content (10 %) at the maximum briquetting force (25 kN), at different magnifications

For the agricultural use, it is important that briquettes retain the initial solubility of crushed limestone. Soluble content of powdered limestone and briquettes with 1 % of the binder obtained at 2 kN force was 0.12 %, while it was 0.128 % for briquettes with 10 % binder obtained at 25 kN force. According to these results, limestone maintained excellent solubility, which was not disturbed in the briquetting process.

For briquette handling it is important to determine how it behaves when fully submerged in water. This is determined by measuring the time needed to obtain the complete disintegration of the briquette. From the results presented in Figure 5, it is evident that the time of disintegration is longest at the lowest binder content. Therefore, the swelling property of bentonite reduces the strength of the links between limestone grains.

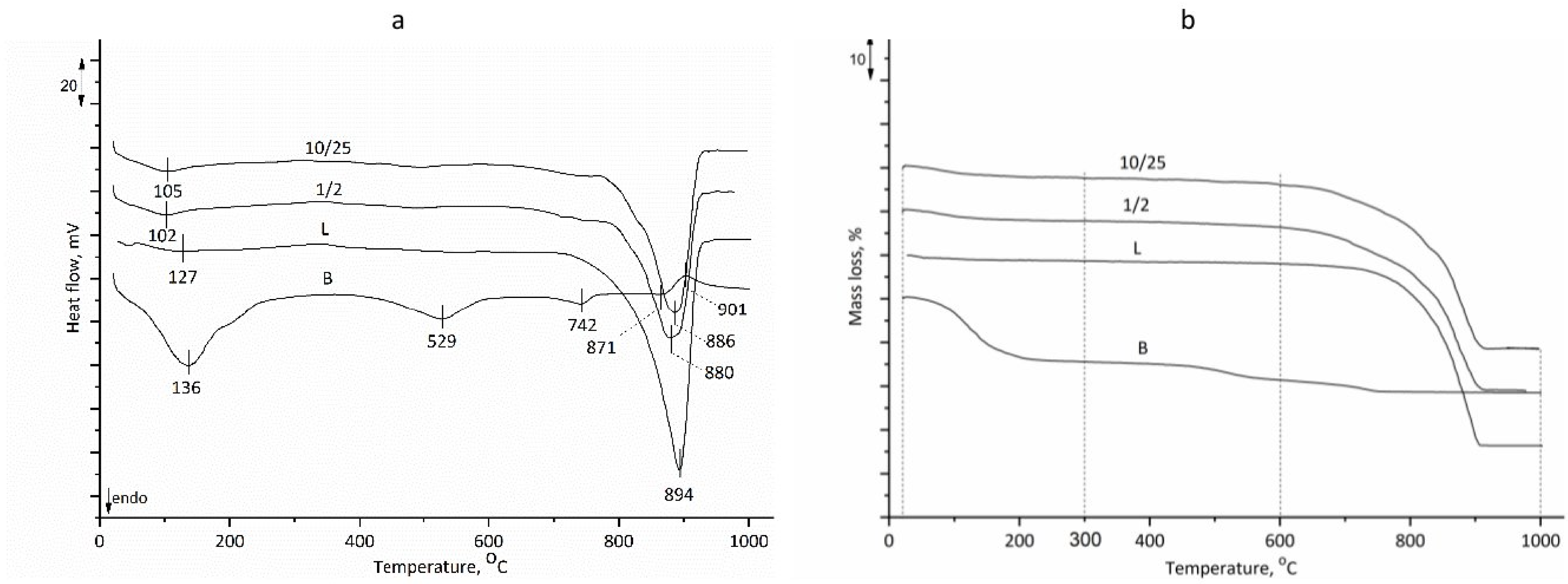

Figure 3. DTA (a) and TG (b) results for: bentonite (B), limestone (L) and briquette samples 10/25 (produced with 10 % binder under 25 kN force) and 1/2 (produced with 1 % binder under 2 kN force)

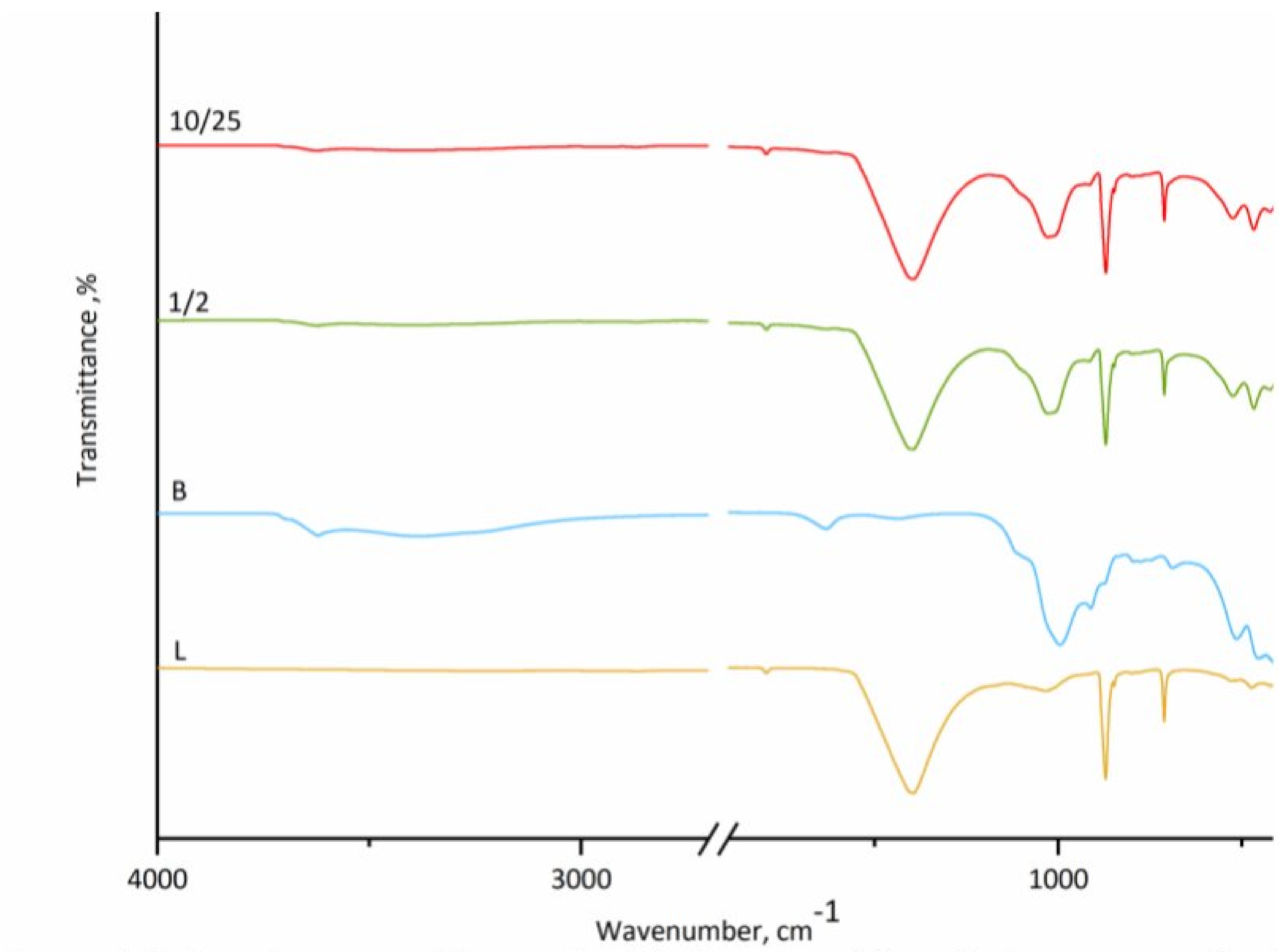

Figure 4. Infrared spectra of bentonite (B), limestone (L) and briquette samples 10/25 (produced with 10 % binder under 25 kN force) and 1/2 (produced with 1 % binder under 2 kN force)

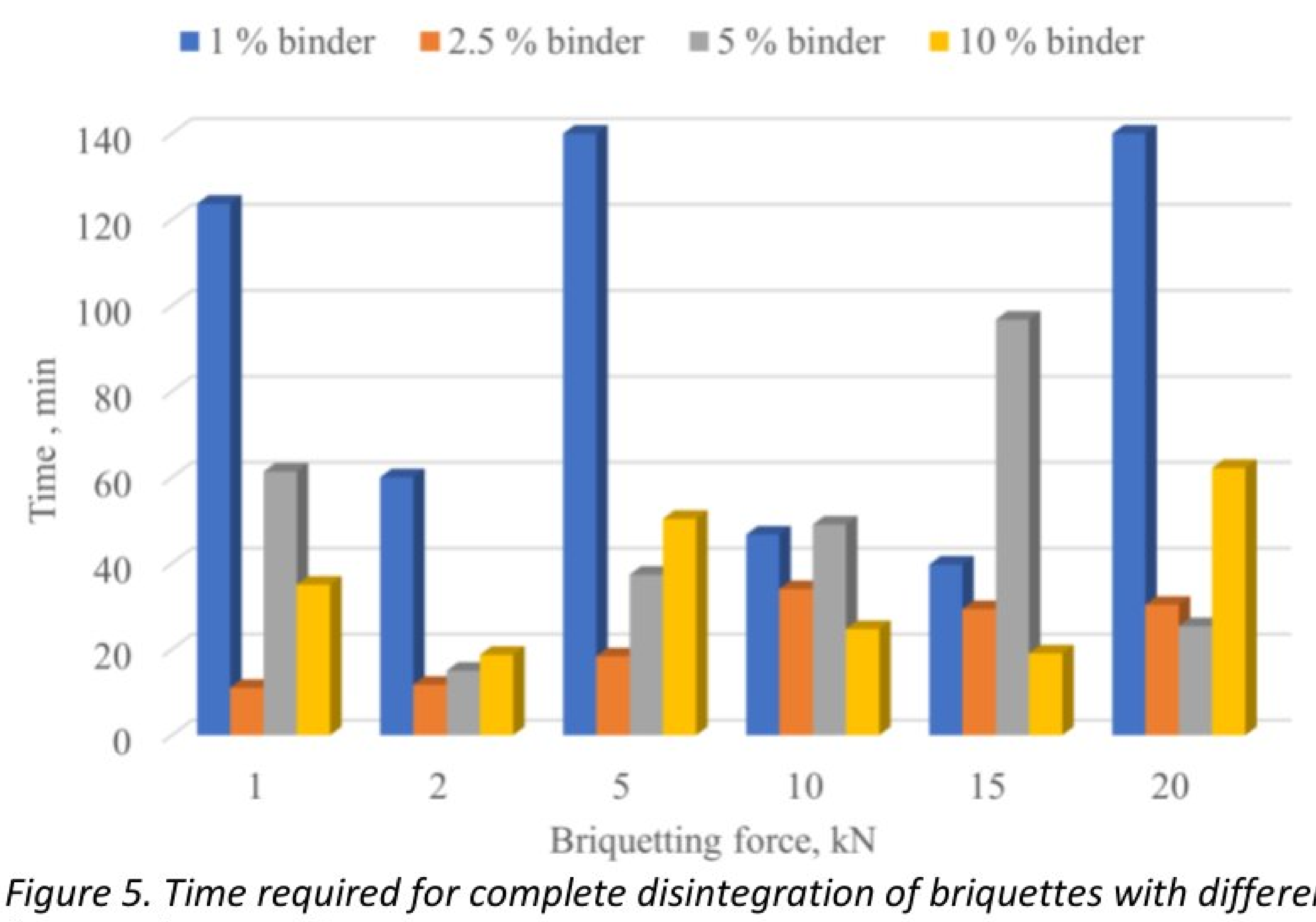

Figure 5. Time required for complete disintegration of briquettes with different binder contents obtained at different briquetting forces submerged in water

The results of mechanical tests of "green" briquettes are shown in Figure 6. Standard mechanical properties were tested such as: impact resistance, compressive strength, and abrasion resistance [29]. According to authors dealing with briquette quality issue mechanical durability is main indicator of briquette mechanical quality [14,21,27,31]. The company Mars Minerals (USA), proficient in agglomeration of limestone, has provided the literature data that was used as a baseline for determining the fulfillment of conditions [29].

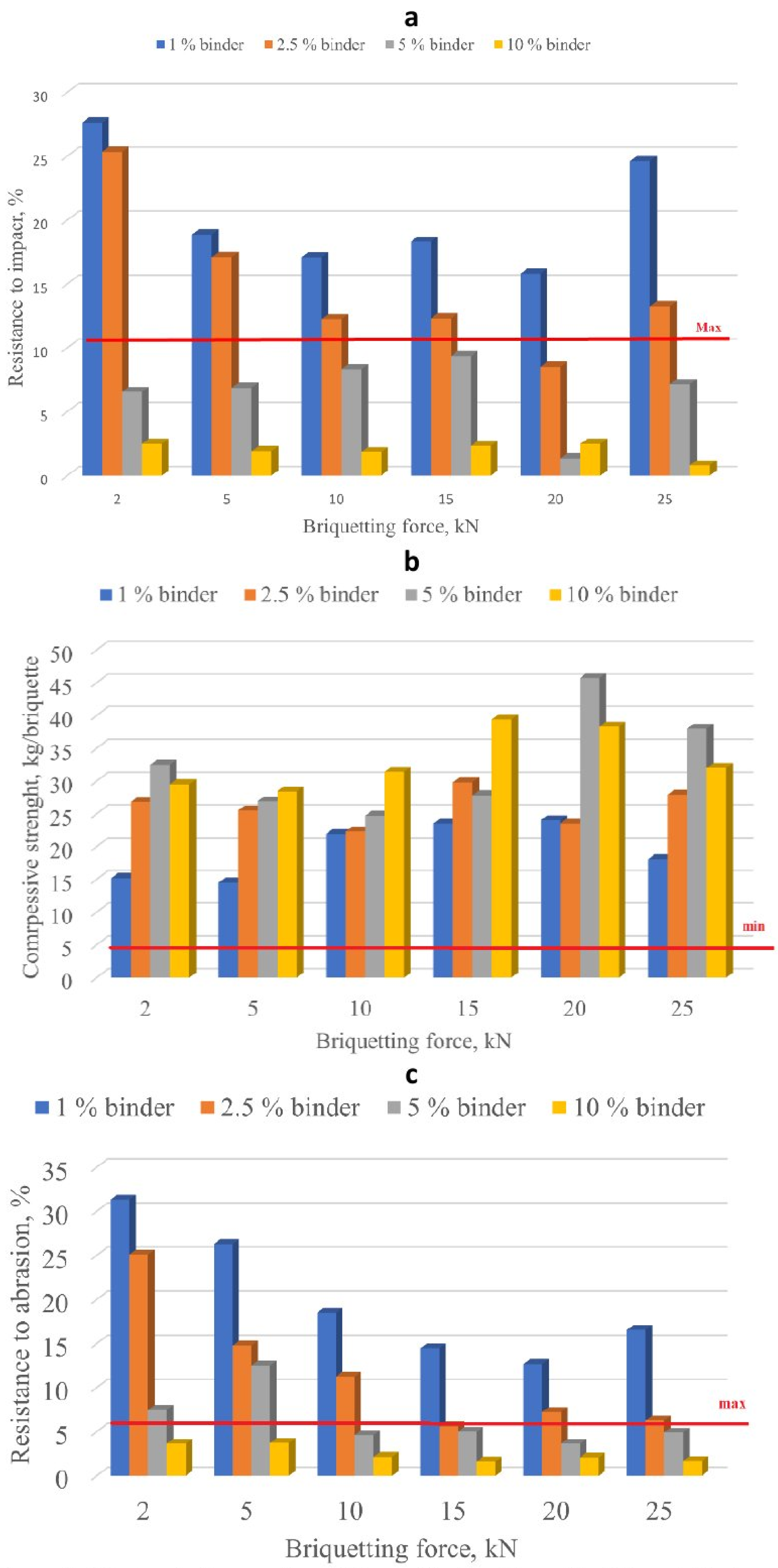

Figure 6. Mechanical properties of "green" briquettes with different binder contents and obtained under different briquetting forces: (a) impact resistance, (b) compressive strength according to the procedure in [29], (c) resistance to abrasion

The obtained mechanical properties of the "green" briquettes (Fig. 6) indicate that the impact resistance depends more on the binder mass content than on the briquetting force. The binder mass content is a relevant parameter for achieving suitable briquette abrasion resistance, but the force at which the briquette is formed is equally important. Briquettes with 1 and 2.5 % binder contents show satisfactory resistance to compression, but unsatisfactory resistance to abrasion and impact. Briquettes with 5 % binder obtained at briquetting forces above 10 kN and briquettes with 10% binder meet all resistance criteria.

The overall results show that the briquettes demonstrate better mechanical properties if the binder mass fraction is higher and generally if formed at higher briquetting forces. Separately, the best results are achieved with a 10 % binder mass content at 15 kN briquetting force, however from the practical point of view, the most economical briquetting procedure implies the use of 5 % binder content at the force of 10 kN. Considering that herein "green" briquettes were the object of investigation, it can only be assumed that after drying, even the briquettes with 2.5 % binder content, formed at the briquetting force ranging from 10 to 15 kN will be acceptable, since it is reasonable to expect that drying will increase the briquette resistance to impact and abrasion.

However, regarding the disintegration time in water, only the samples with low binder mass content showed satisfactory properties, irrespective of the briquetting force applied.

Table 1 shows results obtained by examining the effects of wind on material dispersal for briquettes and limestone powder for comparison. Air temperature was 20 °C, air humidity was 35 % and air pressure was 1001 mbar in the moment of testing. Tests at higher wind speeds than 5 m s^{-1} were not performed since it is considered unreasonable to apply limestone in stronger winds.

Table 1. Test results for determining the effects of wind on material dispersal

Product	Limestone powder			Limestone briquette[1]		
Wind speed, m/s	1	3	5	1	3	5
The weight of the sample before the test, g	1000	1000	1000	1000	1000	1000
The weight of the sample after the test, g	640	470	370	1000	1000	1000
Weight loss, %	36	53	63	0	0	0

[1]Briquette sample was the 50-50 mixture of 10/25 and 1/2 samples

Briquettes have also shown superior properties in wind dispersal tests, as expected, as compared to the powdered material, which was significantly lost at the increasing wind speed (for 36-63 %, Table 4), while it did not induce briquette dispersal. The obtained results showed that it is possible to produce stable "green" briquettes with different binder mass fractions and applying different briquetting forces.

5. CONCLUSIONS

This paper presents the results observed during briquette formation from lithothamnium limestone by adding bentonite. The criteria for determining the quality of "green" briquettes are defined as: unchanged structure and function of limestone, unchanged water solubility, mechanical properties follow the recommendations of the company Mars Minerals and there is no loss during application to the soil. The conducted tests have shown that during briquetting, limestone properties remain unchanged, and all the changes are limited to physical characteristics. According to this we can conclude that during the agglomeration process the binding of the starting material occurred due to the physical and not chemical adsorption. This fact helps us, from the scientific point of view, to better understand the processes that occur during the agglomeration of limestone with the bentonite as a binding agent. Additionally, water solubility is not reduced, the briquettes have satisfactory mechanical properties when binder mass fraction is over 5 % and the briquetting force exceeds 10 kN and finally there is no loss due to wind dispersal during application.

The only downside of so produced "green" briquettes is related to the time required for their complete disintegration if totally immersed in water, (*e.g.* if there is a flood in the limestone briquette warehouse). In such conditions the bentonite starts swelling and briquettes with generally good mechanical properties, quickly lose their shape and disintegrate completely. Given the results, it is evident that the briquetting process should be completed by introducing drying, which would transform the "green" briquettes into hardened briquettes that should have even better mechanical properties.

Acknowledgements: The research presented in this paper was performed with the financial support of the Ministry of Education, Science and Technological Development of the Republic of Serbia, within the funding of the scientific research work, according to the contract with the registration number 451-03-68/2022-14/200023

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Korelacija uslova dobijanja i kvaliteta briketa krečnjaka za upotrebu u kalcizaciji kiselih zemljišta

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(Naučni rad)

Izvod

U radu su prikazani rezultati eksperimenata koji su sprovedeni da bi se utvrdilo kako količina veziva (bentonita) i parametri laboratorijske valjkaste (rol) prese utiču na kvalitet briketa dobijenih od krečnjačkog praha. Ovi eksperimenti imaju za cilj ispitivanje uslova u kojima se formiraju briketi krečnjaka i utvrđivanje mogućnosti njihove upotrebe u poljoprivredi. U toku eksperimenata krečnjaku su dodavani različiti maseni udeli bentonita (od 1 do 10 %), dok se sila valjaka prese kretala od 2 do 25 kN. Briketi su analizirani primenom skenirajuće elektronske mikroskopje (SEM), diferencijalno termičkih analiza (DTA/TG) i infracrvene spektroskopije sa Furijeovom (Fourier) transformacijom (FTIR). Utvrđena je ravnomerna distribucija bentonita, kao i kompaktnost briketa, bez stvaranja novih jedinjenja. Eksperimenti su takođe pokazali da tokom briketiranja svojstva krečnjaka ostaju nepromenjena, promene su samo fizičke, rastvorljivost u vodi nije smanjena, mehanička svojstva (otpornost na udar, čvrstoća na pritisak i otpornost na abraziju) su zadovoljavajuća prema uslovima transporta i skladištenja, ako je maseni udeo veziva preko 5 % i sila briketiranja prelazi 10 kN i, konačno, nema gubitaka usled raspršivanja vetrom tokom nanošenja. Jedini nedostatak dobijenih „zelenih“ briketa je vreme koje je potrebno za njihovu potpunu dezintegraciju ako su potopljeni u vodu.

Ključne reči: bentonit; briketiranje; rol-briket presa; zeleni briket; osobine briketa