Briquetting lignite-biomass blends to obtain composite solid fuels for combustion purposes

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Abstract. The main aim of this study was to determine the possibility of briquetting lignite-biomass blends to obtain composite solid fuels (CSF) for combustion purposes. Research was conducted at Department of Manufacturing Systems and Department of Thermal Engineering and Environmental Protection, AGH University of Science and Technology to develop a technology for briquettes preparation. The briquetting process was carried out in the cylindrical die and in the roll press. The gravity hopper and new asymmetrical layout briquetting system were used in the roll press and briquettes in the shape of the saddle i.e. without the splitting plane were obtained. Effects of the ratio of biomass to lignite (20/80; 30/70; 40/60 and 50/50), moisture content (9.9%-31.3%), and briquetting unit pressure (32-210MPa) on the strength of the briquettes were investigated. The obtained results showed possibility of briquetting of lignite-biomass blends in the cylindrical die and in the roll presses and the mechanical strength obtained briquettes was high i.e. shatter index >90% and compressive strength >1MPa. All of the produced briquettes were not water resistant. Based on physical and chemical analysis of prepared briquettes it was confirmed that briquettes have good fuel properties for combustion process. Thermal behavior of studied lignite and prepared mixture was investigated by thermogravimetric analysis (TG). The samples were placed in an alumina crucible. C.a. 15 mg of sample was heated from an ambient temperature up to 1000°C at constant two rates: 10°C/min and 40°C/min in a 40 ml/min flow of air. Finally, basic analysis of ash from burning briquettes were carried out and some of the results are presented.

1. Introduction

Production of composite solid fuels (CSF) from energy sources of various origins, mostly low quality, is a way of their rational use and disposal in many regions of the world. The requirements for their properties are varied, depending on the destination, e.g. burning in fireplaces [4, 7-13, 19-21, 23], kitchen ovens [5, 6, 21-23], low power boilers for settlements heating [1-13, 19-23], co-firing in heat and power plants [4, 7, 10, 11, 14-18, 22, 23] and gasification [3, 4, 24] in small and large scale. The idea of such fuel is to obtain a homogeneous mixture of the available raw materials (selected in the desired (required) proportion) and then, most often, by pressure agglomeration, giving it a form of briquette or pellet. The main constituent of CSF is usually low-grade coal or lignite, which is mixed with broadly defined biomass [1-18] or with high quality coal [19, 20]. Such a fuel is also often produced by biomass mixtures of various types [21-24], which can also be pre-applied to appropriate thermal processes [4, 24]. An important and frequent addition to CSF is also the binder: organic,
inorganic, natural or synthetic [4, 6, 10-13, 19, 20, 24, 25]. This alternative approach to the traditional use of natural primary energy carriers naturally occurring in the form of solid minerals, primarily coal, is due to a number of different causes and reasons. Production of CSF is a way of obtaining fuel with stable performance parameters, which is very important for its potential customers [3, 17, 19, 23]. The use of wastes in such fuel makes it easier to manage [17-20], and the addition of biomass allows to increase its so-called energy density and makes it more environment-friendly (reduction of harmful substances, especially SO₂, NOₓ, CO, HCl) [1-5] and stable as opposed to pure biomass [21, 24], utility parameters. Furthermore, the addition of different additives to the mixture composition has an impact on the improvement of the energy, mechanical and utility properties of the fuel produced [1-4, 19, 20, 24, 25].

In general, CSF has better energy parameters, higher density, higher calorific value (especially per volume unit) and lower moisture content as compared to the raw materials and different kind of materials can be used to make mixtures for briquettes and pellets [1-25].

The process of pressure agglomeration can be carried out in different devices [3-5, 11, 14, 16, 21, 23] thus the outcome and process itself is influenced by many factors (compressibility and compatibility of materials, parameters of the agglomeration process, shape of the forming tools and others) [4, 11-14, 16, 21]. For this reason, the industrial implementation of these processes is usually preceded by experimental research.

The aim of this study was to determine the possibility of briquetting lignite-biomass blends to obtain CSF for combustion purposes. The briquetting process was carried out in the cylindrical die and in the roll press. Effects of the ratio of biomass to lignite, moisture content, and briquetting unit pressure on the strength of the briquettes were investigated. Next, the combustion process using thermal analysis of obtained briquettes was examined and finally, basic analysis of ash from burning briquettes were carried out.

2. Experimental – materials and methods

The lignite from Bełchatów-Szczerców quarry with a grain size of 0-2 mm was used for the tests along site with oat straw and cellulose fibers mixed in the ratio of 19:1. The oat straw was fragmented to a size of <50 mm in a rotary mill with a multi-spin head and then ground in a hammer mill equipped with a 4 mm sieve. Dried and chopped pulp fibers were waste from the paper industry. Selected properties of raw materials for testing are presented in Tables 1-3.

| Table 1. Analysis of Belchatów-Szczerców lignite. |
|-----------------------------------------------|
| LHV  | Moisture | HHV  | Ash  | Sulphur | Nitrogen | Carbon | Hydrogen |
| Q<sub>ar</sub> | W<sub>ar</sub> | Q<sub>ad</sub> | A<sub>ad</sub> | S<sub>ad</sub> | N<sub>ad</sub> | C<sub>ad</sub> | H<sub>ad</sub> |
| kJ/kg | % | kJ/kg | % | % | % | % | % |
| 8,091 | 51.16 | 15413 | 20.74 | 0.75 | 0.54 | 39.6 | 3.25 |

Notes: t – total; ar – as received; ad – air dried

| Table 2. Sieve analysis of Belchatów-Szczerców lignite. |
|-----------------------------------------------|
| Particle size [mm] | 0.2 | 0.5 | 1 | 2 | 2.5 |
| Undersize [%]      | 39  | 65.83 | 83.1 | 94.0 | 100 |
Table 3. Analysis of biomass (oat straw and cellulose fibers) used in these studies.

|         | HHV $^*$ | Ash $^*$ | Sulphur $^*$ | Carbon $^*$ | Hydrogen $^*$ | Chlorine $^*$ |
|---------|----------|----------|--------------|-------------|---------------|--------------|
| oat straw | 16870    | 5.6      | 0.06         | 43.0        | 5.4           | 0.12         |
| cellulose fibers | 15030    | 9.4      | 0.05         | 39.6        | 5.43          | 0.57         |

Notes: $^*$ total; $^*$ air dried

The lignite composition was homogenized chemically and physically in a mixer with a heated body, followed by the addition of biomass to the mixture. The mass share of biomass relative to lignite was 20, 30, 40 or 50%, respectively. Simultaneous mixing and drying resulted in mixtures in which the bulk water content was 31.3% to 9.9%. Mixing time was determined experimentally. The moisture was determined by weight of mixtures. The samples were dried in an electric drier at 105°C to constant weight, controlling the loss of analytical balance. The average moisture content of the three samples was found to be representative.

The material was briquetted in cylindrical die and in the roll press with set moisture levels of the mixture. Closed matrix with internal diameter of 20 mm was filled each time with a sample of 8g material, from which briquettes were formed on an universal pressure machine with a pressure of 32-210 MPa. The stroke speed was 10 mm/min. Each of the briquettes removed from the matrix was seeded for 7 days prior to testing its quality indicators.

For a continuous briquetting process laboratory roll press with asymmetrical layout briquetting system was used [10, 26, 27]. This kind of forming system allows to make briquettes in the shape of the saddle i.e. without the splitting plane. The gravity hopper was used to feed material into gap between the counter-rotating rolls. The amount of fine-grained material was 5÷7 kg. The sample was united at 0,1÷0,4 m/s of tangential velocity corresponding to rotational speed 4,25÷17,0 rpm. The initial value of the interspace between the rolls during briquetting process was ~1,0mm. After leaving the cavities the briquettes were stored in a container for 7 days from which samples were taken at random (in batches 10 pcs. each) to determine parameters characterising their mechanical properties. Some mechanical strength tests of the briquettes were done directly after briquetting.

The compressive strength (CS) of each briquette was measured by using the universal tensile testing machines (ZDM-10 and ZWICK 1120). A single briquette was placed between two flat, parallel plates which have facial areas greater than the projected area of the briquette. An increasing load was applied at a constant rate 5 mm/min, until the test briquette failed by cracking. CS was calculated dividing the load of the fracture point by cross-sectional area of plane of fracture [28]. Each time 10 randomly sampled briquettes from each batch were tested. CS was calculated as an average of ten measurements.

Additionally, in radial direction, compressive strength (specific crushing strength) of a cylindrical briquet was determined as the crushing force normalized by the cross-sectional area (d × h) [29].

The drop shatter test (according modified PN-ISO 616) was determined by dropping briquettes in batches 10 each from a height of 2m onto a steel plate. This test was performed three times, each time sieving the broken mass at a sieve where, size of sieve was determined to 2/3 of average calculated from two maximum briquette dimensions measured in mutually perpendicular directions. The shatter index was calculated as an average of three measurements [17].

Thermogravimetric analysis was carried out using a Mettle Toledo TGA/SDTA 851 apparatus. The TGA instrument was calibrated with indium, zinc and aluminum. Its accuracy is equal to 10$^{-6}$ g. For the thermogravimetric analysis (TG) the samples were placed in an alumina crucible. C.a. 15 mg of sample was heated from an ambient temperature up to 1000°C at a constant two rates: 10°C/min and 40°C/min in a 40 ml/min flow of air. The thermal results (TGA) were presented in the form of TG and
DTG curves, respectively, where: TG - thermogravimetry presents the weight loss of studied samples in contrast to the initial mass under increasing temperature and DTG - differential thermogravimetry is based on the rate of weight loss and DTG profiles make it possible to know, the weight loss which is taking place at a temperature during the combustion process.

Samples of ash, obtained after combusted briquettes, were averaged and weighed down, before being a subject of physical and chemical properties.

The relative density was determined by the pycnometric method, a Gay-Lussac pycnometer measuring 50 cm³, at a constant temperature of 21°C. Bulk density measurement was performed by weight, using a 50cm³ vessel.

The ash moisture content was determined by weight, by drying in an electric drier, weighed on an analytical weighing scale. Drying was carried out at 110 ± 2°C to constant weight (about 24h) and then their moisture content was determined on the basis of the weight loss. The average humidity in three weights is representative.

The content of flammables was determined by weight method by heating the weights in an electric resistance furnace at 900°C in an oxidizing atmosphere for 4 hours. The unburnt coal (roasting loss - LOI) content was determined based on the weight loss. The average content of flammable parts in three weights was considered representative. The roasting losses were determined for the starting material, so it was the sum of the burned organic parts and moisture of the sample, which was taken into account in further calculations.

Grain size distribution was done by sieving fraction less than 0,25mm. The residue was subjected to the laser analyzer Malvern Mastersizer 2000.

The study of changes in the resistivity of the dust layer as a function of temperature in the range of 40-250°C was made using a Wahlco instrument - a reference device for this type of measurement [30].

Microscopic examination of ash samples was carried out using the JEOL JSM 5400 scanning electron microscope, which cooperates with the EDS x-ray microarray, LINK ISIS 300 (Oxford Instrument), during which full chemical analysis was performed. The co-operation of the analyzer with an electronic scanning microscope ensures a complete quantitative analysis of the chemical composition of the test surface in a small area and in a very short time. The analysis was carried out on unmodified dust samples, using EDS, under low vacuum conditions (10⁻⁴ Tor). Prior to analysis, the samples were coated with a thin layer of carbon to ensure that the load was drained during the measurements, making it impossible to assess the carbon content. The presented analysis of the ash composition is the average result of at least three measurements made at different points of the sample. The results of the chemical composition analysis of the dust generated by the combustion of CSF are presented in the form of oxide composition, expressed in atomic, weight and molar percentages.

3. Results and discussion

3.1. Results of briquetting

The first briquetting experiments were carried out in the cylindrical die. It was stated that the best results, i.e. mechanical strength of the briquettes, have been obtained from a 70/30 blend ratio of lignite and biomass [11]. Therefore, the 70/30 blends of lignite and biomass at different moisture contents were used for the next tests in the cylindrical die.

Relations between axial compressive strength/radial compressive strength and unit pressure for cylindrical briquettes at various moisture contents are presented on Figure 1. The general character of changes in briquette strength in the axial and radial direction depending on the unit briquetting pressure is the same. It was found that with the reduction of the moisture content of the briquette mix, the briquette strength in both mutually perpendicular directions increased. The nature of compressive strength (CS) was found to be linear [29], and the direction factor was termed compactibility. The higher value of this coefficient expresses the ability of the material to form a higher strength CS agglomerate at a lower briquetting pressure. The highest values of CS changes from 0.26 MPa to
3.03 MPa corresponded to a briquette pressure of 210 MPa for mixtures with a moisture content of 27.9% to 18.6%, respectively.

Compressive strength of the cylindrical briquettes measured in axial direction (ACS) is approx. ten times greater than CS measured in radial direction. Merging above the unit pressure of 140 MPa causes only slight changes to the ACS [31]. Within the range of moisture content of the blends, the ACS value for the 210 MPa pressure ranged from 4.47 to 33.39 MPa. The maximum value of ACS = 33.39 MPa and CS = 3.03 MPa had briquettes with a moisture content of 18.6%.

Changes in the shatter index of cylindrical briquettes with different humidity from the briquetting unit pressure are similar (Figure 2). Shatter index is increasing in the range of unit pressures of 31-140 MPa and above 140 MPa the value of this index changes only slightly. Briquettes with moisture content of 18.6%, 21% and 25.5% obtained with a pressure of 31 MPa have a shatter index higher by about 31%, while pressures above 140 MPa are about 9% higher than briquettes with a moisture content of 27.9%.

![Figure 1](image1.png)

**Figure 1.** Relation between axial compressive strength/radial compressive strength and unit pressure of briquetting for cylindrical briquette made of lignite/biomass blend (ratio 70/30) at various moisture contents.

![Figure 2](image2.png)

**Figure 2.** Relation between shatter index and unit pressure of briquetting for cylindrical briquette made of lignite/biomass blend (ratio 70/30) at various moisture contents.

During the briquetting in the roll press following parameters were measured: the influence of rolls speed, mass share of biomass and moisture content on the briquette density, shatter index immediately
After the test, compression strength after 7 days, unit pressure in the cavity and torque on the roll shaft [10]. It has been found that the roll speed and the moisture of the mixture determine the compressive strength of the briquettes. In each case (with each test compound) with an increase in the rotational speed of the rollers, a significant decrease in the quality of the briquette product was observed. The dried briquettes (moisture content 9.9%) had the lowest compressive strength of 0.41 MPa. For briquettes with a moisture content of 24.4% at rotational speed of the rolls 4,25 rpm. (mass fraction of lignite-biomass 50/50) the value of this strength was highest and amounted to 1.28 MPa. The unit pressure of the briquetting in the roll press is the resultant parameter. To make comparison with briquettes in a cylindrical die, the compressive strength of briquettes made in roll press is also shown in a function of unit pressure (Figure 3). The shatter index was dependent primarily on the moisture content of the mixture and to a small extent on its composition and rotational speed of the rollers. The highest value of shatter index was obtained for briquettes with a moisture content of at least 21.9%, their relative strength was up to 93.8%. The strength of briquettes dried to a moisture content of 9.9% was definitely lower, their shatter index was up to 57%. It has been found that in order to obtain a high mechanical strength of briquettes in a roll press, the moisture content of the blends should be within 20%-30% [10]. The briquettes obtained in the cylindrical die and in the roll press are presented in Figure 4.

![Figure 3](image)

**Figure 3.** Some examples of relations between compressive strength and unit pressure of briquetting for briquettes made in roll press at different moisture content, lignite/biomass ratio and rolls speed.

![Figure 4](image)

**Figure 4.** Photo of briquettes made in the cylindrical die (left) and in the roll press (right).

All of the produced briquettes were not water resistant. The time to completely disintegration the briquettes after their immersion in water has not exceeded 15 minutes.
3.2. Thermal analysis

Combustion characteristics of lignite and biomass and lignite briquette (blends) have been studied using thermal techniques (TG/DTG). The thermogravimetric analysis has a lot of advantages in combustion studies because it gives the information, the temperatures at which combustion starts and ends, the maximum reactivity and ash amount [32-36]. The influence of addition of biomass to lignite and the heating rate has been investigated. Figures 5 and 6 show the mass change (%) TG curves and DTG curves for lignite and lignite and biomass briquette under combustion conditions.

Generally, the combustion process of lignite could be divided into three main stages (Figure 5). The initial decrease in the weight loss was attributed to removal of moisture content, it took place up to 200°C. The mass loss was more than 30%, what confirmed the high moisture content in lignite. The main stage of combustion process was in 200–600°C temperature range where the degradation of organic compounds took place. Based on DTG curves it was be observed that maximum mass loss was at c.a. 400°C, and the second smaller one at c.a. 500°C. The last stage could be defined above 600°C where no significant weight loss and thermal effect were found. Taking into account the heating rate of studied process some small differences in temperature ranges, the shape and the character of TG and DTG curves could be observed. The TG curve (for 40°C/min) was moved into higher temperatures and the mass loss in main stage was faster. The amount of solid residue increased with the increase of heating rate.

The addition of biomass to lignite had changed the TG and DTG profiles during the combustion process. The lignite and biomass briquette had characterised less amount of moisture content (during the briquetting process some amount of water could release). The maximum mass loss took place c.a. 320°C. For heating rate of 40°C/min the combustion process was moved slightly into higher temperatures. The amount of solid residue was 20%.
3.3. Ash analysis

Results of physicochemical properties, chemical composition and morphological properties determined by microscopic fly ash analysis, after combusted of lignite-biomass (70/30) briquettes, are presented in Tables 4-6.

Physicochemical properties (especially ash density and combustible parts content), granulometric composition and electrical parameters are factors that have a significant effect on the efficiency of the dust extraction process [30]. The physicochemical properties of fly ash are shown in Table 4.

| Relative density | Bulk density | Relative moisture | Content of flammable parts |
|------------------|-------------|------------------|---------------------------|
| \( g/cm^3 \)     | \( g/cm^3 \) | % mas.           | % mas.                    |
| 2.40             | 0.3932      | 1.15             | 3.31                      |

The mass fraction of unburned coal (roasting loss) in the fly ash studied at the level of 3.31% is comparable to the calcite fly ash burns generated during the combustion of lignite in pulverized furnaces. The relative and bulk density of the investigated ash is close to that of other ashes produced from the combustion of different types of carbon and carbon-biomass mixtures [30].

The cumulative and population cumulative curve of the ash fly ash sample is shown in Figure 7. The characteristics of the particle distribution are: median \( D_{50}=100.75 \, \mu m \), moda \( D=148.26 \, \mu m \), \( D_{10}=8.64 \, \mu m \) and \( D_{90}=243.05 \, \mu m \). The grain sizes are in the range of 38-447 \( \mu m \). The presence of about 10% of grains above 250\( \mu m \) may be due to the elongated shape of the biomass grains, a portion of which, during the sieve analysis, has been lost through a sieve with a mesh size of 0.25 mm.

| Particle size distribution of fly ash sample from the combustion of briquettes made from lignite-biomass blend. |

Ash in its composition include magnesium, aluminum, silicon, phosphorus, sulfur, potassium, calcium and iron (Tab. 5). Sulfur is most likely in the form of sulphates. The sulfur content is relatively high and amounts to approx. 19% at. The most oxalic constituent is calcium oxide, about 46% by weight. In addition, approximately 17% of alumina and silicon oxide are present. Relatively high iron oxide (hematite) is also about 14% by weight (Table 6). The sum of the basic oxides (\( SiO_2 + Al_2O_3 + CaO + Fe_2O_3 \)) is 94.88%. No sulfur was considered in the oxide analysis because it is not present in the form of oxide. The morphology of the fly ash resulting from combustion of the 70/30 lignite/biomass blend briquettes is shown in Figure 8 at a magnification of 350× and 3000×.

| Table 5. Elemental composition of fly ash, in atomic %. |
|----------------------------------------------------------|
| Element | Mg | Al | Si | P | S | K | Ca | Fe |
| Content | 2.78 | 15.75 | 13.86 | 0.92 | 18.96 | 1.26 | 38.58 | 7.89 |
Table 6. Chemical composition of the fly ash, % by mass and % by mol.

| Oxide   | MgO   | Al₂O₃ | SiO₂ | P₂O₅ | K₂O  | CaO   | Fe₂O₃ |
|---------|-------|-------|------|------|------|-------|-------|
| Content, % wag. | 2.44  | 17.36 | 17.79 | 1.40 | 1.28 | 46.21 | 13.52 |
| Content, % mol.   | 4.14  | 11.67 | 20.29 | 0.68 | 0.93 | 56.49 | 5.80  |

Figure 8. SEM photographs of fly ash samples from the combustion of lignite-biomass blend in magnification: 350× and 3000×.

The fly ash image is characteristic of the residue after combustion of biomass-like organic components. Particles vary in size from very fine particles of the order of 20 μm to large ones of 200 μm. Visible agglomerates of porous and partially melted structures. The shape of the particles is elongated and there are no spherical forms characteristic of the ashes coming from combustion, e.g. stone coals. The structure is fibrous with high porosity with holes that can be formed by the release of gases (e.g. CO₂). EDS spectra of the composition of the test sample are shown in Figure 9.

Figure 9. EDS spectrum of elemental fly ash composition.

Knowledge of the ash resistivity value enables to decide whether it can be separated with the electrostatic method, which is significant for the appropriate selection of the exploitative parameters of the ESPs. The resistivity of the ash ranging from 10¹⁰-10¹¹ Ωcm is considered optimal due to its dedusting properties in the ESPs. The characteristics of the changes in resistivity of the analysed ash in the temperature function (Figure 10) indicates that the analysed ash is highly resistive (≥10¹² Ωcm).
The specific morphology of the lignite, resulting in bonding of water in its structure, has a beneficial impact on the electrostatic separation of the ashes obtained from the lignite combustion. Steam that is released in the combustion process reduces the resistivity of the ash contained in the fumes. The fumes from the combustion process can be, therefore, successfully dedusted with the ESPs.

4. Conclusions
Experimental studies have shown that the CSF in high mechanical quality (shatter index>90% and compressive strength >1MPa) can be obtained from the selected components using cylindrical die and roll press. The compressive strength and the shatter index of briquettes produced in the cylindrical die increased with decreasing moisture and greater unit pressure. It was found that the rotational speed of the rolls, the moisture content of the mixture and its composition determine the compressive strength of briquettes during briquetting in the roll press. The shatter index of the briquettes was dependent primarily on the moisture content of the mixture and to a small extent on its composition and rotational speed of the rolls. The produced briquettes were not water resistant.

Combustion lignite and biomass-lignite briquette was illustrated by thermal decomposition behaviour by TGA analysis. The combustion of these fuels confirmed the high amount of moisture in lignite. The increase of heating rate was shifted to higher temperatures the TG curves thereby combustion process.

In co-firing coal and biomass, physical and chemical characteristics of used fuels have an influence on the characteristics of ash. The main component of fly ash is calcium oxide (CaO) 46.21%, but in a large proportion there are also aluminosilicates (SiO2+Al2O3) 35%. Such ash can be a component of the cement, an additive in the concrete composition and be the fundamental component of the binders used in the grounds stabilisation processes, base reinforcement and other works connected with roads construction. The analysed ash does not contain toxic substances and it can be used as a mineral fertiliser or a soil deacidification substance (Fe2O3+CaO+MgO+K2O+P2O5 = ~65%).

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5. References
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