



Evaluation of the mechanical properties of wood-derived charcoal briquettes for use as a reductant

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Dates:

Received: 29 Aug. 2019

Revised: 16 Mar. 2021

Accepted: 19 Mar. 2021

Published: May 2021

How to cite:

Makgobebele, N.W., Mbaya, R.K.K., Bunt, J.R., Leokaoko, N.T., and Neomagus, H.W.J.P. 2021

Evaluation of the mechanical properties of wood-derived charcoal briquettes for use as a reductant.

Journal of the Southern African Institute of Mining and Metallurgy, vol. 121, no. 5, pp. 187–192.

DOI ID:

<http://dx.doi.org/10.17159/2411-9717/903/2021>

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Synopsis

Silicon Smelters consumes more than 80000 t/a of wood-derived charcoal as carbonaceous reductant in the production of silicon metal. More than 10% of this material is discarded as fines (<6 mm) generated due to abrasion during processing. Charcoal fine residues (<650 µm) and polyvinyl alcohol (PVA) binder were used in this study to produce mechanically strong charcoal briquettes for metallurgical application as carbonaceous reductant. The PVA binder was added in mass percentages of 1, 3, and 5 wt% to the charcoal fines. The compressive strength, abrasion resistance index (ARI), drop shatter resistance (SRI), and water resistance index (WRI) were measured as functions of curing for up to 7 days under atmospheric conditions, and the results compared with metallurgical grade coarse charcoal. The ash content of the produced briquettes was found to be high (6.6–8.0%) compared with the coarse charcoal (1–3%). The 3 and 5 wt% PVA-bound briquettes were found to be the strongest, with compressive strengths of 40 and 115 kg/cm² respectively, with WRI values of 75 and 73% respectively. The produced briquettes were found to have lower ARI and SRI values compared to the coarse charcoal. Future work should include beneficiation of the fine charcoal discards prior to briquetting, and an increase in binder addition to above 6 wt% to improve the ARI and SRIn.

Keywords

wood charcoal, fines, briquettes, reductant, polyvinyl alcohol binder, compressive strength, water resistance, curing time.

Introduction

In 2015, South Africa's production and processing of wood-derived charcoal resulted in the generation of more than 5.2 Mt of charcoal fines (<6 mm) due to abrasion and attrition during transport and handling, which were discarded as a residue (FAO, 2017). Coarse charcoal (CC) with particle sizes (6–60 mm) is widely used in the metallurgical industry as a carbonaceous reductant because of its low level of impurities compared to coal, and ease of handling. More than 10% of the charcoal fines less than 6 mm in size is discarded into stockpiles as residue. In this research, different polyvinyl alcohol (PVA) binder solution concentrations were used with charcoal fines to produce charcoal briquettes for metallurgical utilization. The mechanical properties of the briquettes produced were evaluated in relation to coarse charcoal.

Literature review

Wood-derived charcoal is a carbonaceous product with low ash, moisture, and volatile contents, obtained by a pyrolysis process at temperatures ranging between 400 and 600°C (Basu, 2010). With the exception of particle size and moisture differences, the charcoal fines have similar characteristics as the coarse charcoal. Beneficiation of charcoal residue gives briquettes a charcoal-like appearance; hence the terms 'charcoal briquettes' or 'biocoal' (McDougall, 1991). Briquetting of charcoal fines is the process of converting the low-density pulverized charcoal matter from the biomass material to high-density and energy-concentrated charcoal briquettes, often with the aid of a suitable chemical binder material (Zubairu and Gana, 2014). Briquetting technology has been used successfully in many countries to amalgamate loose biomass into hard solids of regular shapes such as briquettes, pellets, or cubes, depending on the densification equipment employed.

Charcoal briquette properties depend on the type of wood from which the charcoal is derived, as well as the carbonization process used (Sahajwalla, 2004). The earliest industrial use of charcoal was as carbonaceous reductant for iron smelting to reduce iron oxide to metallic iron. With the development of the chemical industry and increasing legislative requirements for the preservation of the environment, the application of charcoal for purification of industrial wastes has grown markedly. Some metallurgical

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applications of charcoal in various industries are smelting and sintering of iron ores, production of ferrosilicon and silicon metal, case hardening of steel, as a purification agent in smelting non-ferrous metals, a fuel in foundry cupolas, and electrodes (FAO, 1985). The earliest coal briquettes were made in hand-filled brick moulds using clay and cow dung as binders. These briquettes had poor mechanical properties (compressive strength, abrasion resistance, shatter resistance, and water resistance) which made them unsuitable for transportation over long distances (Venter and Naude, 2015). The acceptable compressive strength for coal briquettes in the industry is 3.8 kg/cm² (Richards, 1990). Increased strength reduces absorption of atmospheric moisture and thus increases the briquette durability. Different parameters affect the compaction pressure, *e.g.* feedstock, temperature of the pressing chamber, dimensions (length and diameter), the shape of the pressing chamber, and the compacting procedure (Kaur, Roy, and Kundu, 2017).

Briquettes undergo various degrees of mechanical degradation during conveying over short distances and long-distance haulage. This arises from breakage due to compression and abrasion while the briquettes are in contact with each other and the walls of the transportation vehicle. The falling of the briquettes during conveyer belt transfers, from chutes to bins, and off trucks onto the ground may also enhance their degradation. Impact-shatter is defined as breaking violently into pieces from a sudden impact. Impact-shatter forces are encountered when briquettes drop during stockpiling, at conveyor transfer points, and from bins and chutes. The measurement of this parameter can be used to indicate the extent to which briquettes will remain intact during handling, transportation, and storage. Water resistance shows the resistance of solid fuel briquettes to moisture or water penetration during transport or storage. High water resistance is a desirable quality which enables briquettes to remain impermeable to water for a long period before losing their integrity (Prasityousil and Muenjina, 2013).

Materials and method

Charcoal fines discard (<6 mm) from Silicon Smelters (Pty) Ltd (Polokwane, South Africa) and polyvinyl alcohol (CH₂CHOH)_n (PVA 17-99) from Chem System (Pty) Ltd (Kempton Park, South Africa) were used in this study. The produced charcoal briquettes were characterized and compared with the metallurgical-grade coarse charcoal used as a reductant in electric-arc furnaces.

Charcoal fines preparation

The charcoal fines were milled in a stainless steel ball mill. The particle size distribution was determined using a Malvern Mastersizer 2000, and 90% of the milled sample was found to be below 650 μm. The milled charcoal was air-dried and a representative sample was taken for proximate and ultimate analyses.

Proximate and ultimate analyses

The inherent moisture content was determined by mass loss. The air-dried charcoal sample was placed in an oven which was thermostatically controlled with forced air ventilation, maintaining a temperature of 105°C for 3 hours. The ash and volatile matter contents on an air-dried basis (adb) were determined by means of the ISO 1171 (2010) and ISO 562 (2010) standards using a muffle furnace. The percentage of fixed carbon (FC) was calculated as the difference between 100% and

the sum of the percentages of inherent moisture (IM), ash, and volatile matter (VM) contents on an air-dry basis using Equation [1].

$$FC_{adb} = 100 - (\%IM + \%Ash + \%VM) \quad [1]$$

The ultimate analysis was determined by Bureau Veritas based on the ISO 12902 standard to determine the percentage carbon, hydrogen, and nitrogen. Sulphur was determined using the (ISO 19576 (2006) standard. The oxygen content was calculated by difference, using Equation [2]:

$$\%O = (100 - \%C - \%H - \%N - \%S) \quad [2]$$

The ash composition was determined based on the ASTM D4326-04 92004 standards by X-ray fluorescence (XRF) using fusion beads. Table I shows the obtained proximate, ultimate, and ash composition analyses of the discard charcoal fines.

The proximate and ash analyses indicate an ash content of 7.7 wt% and a high silica content in the ash, of 72 wt%.

Polyvinyl alcohol (PVA) binder preparation

The 1, 3, and 5 wt% PVA solutions were prepared using a Labotec 105 magnetic heater stirrer, glass beaker, and thermocouple. Three 100 g aqueous solutions were prepared containing 1, 3, and 5 g of PVA powder. Water, in a beaker, was placed on the heated stirrer and heated to a maximum temperature of 92–98°C, at a heating rate of 2°C/min and stirring rate of 100 r/min. The fully solubilized solution was then allowed to cool to a temperature less than 30°C. The PVA solutions produced were then sealed in bottles and stored in an air-conditioned laboratory.

Charcoal briquetting

A 25 g aliquot of PVA solution was gradually dosed into 475 g of fine charcoal and homogenized using a pestle and mortar, to give a total mixture mass of 500 g. To produce the binderless and the PVA-bound briquettes, a mixture of charcoal fines with PVA solution was fed into a single-die mould machine to produce a compacted cylindrical agglomerate of size 13 × 10 mm. The fines

Table I

Proximate, ultimate and ash analyses of the charcoal fines

Proximate analysis, wt % (adb)	
Inherent moisture	15.6
Ash	7.7
Volatile matter	21.0
Fixed carbon (by difference)	55.7
Ultimate analysis, wt % (adb)	
Carbon	52.2
Hydrogen	2.0
Nitrogen	0.6
Oxygen (by difference)	21.9
Total sulphur	0.1
Ash Composition, %wt	
SiO ₂	71.8
Al ₂ O ₃	8.5
CaO	6.5
Fe ₂ O ₃	4.4
Na ₂ O	2.5
K ₂ O	2.2
MgO	0.8
SO ₃	1.9
TiO ₂	0.4
P ₂ O ₅	0.3

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were compacted with a Chatillon Amatek TCD200 press (Figure 1), with a force of 1000 N at a constant speed of 120 mm/min to produce high-density briquettes. The briquettes were stored in an air-conditioned room for further proximate and physical analyses. Charcoal lumps of dimensions 13 × 10 mm were selected from the coarse sample (+6 –60 mm) for comparison with the briquettes.

Mechanical analysis

The coarse charcoal, binderless, and PVA-bound briquettes were tested for compressive strength (CS) (kg/cm²), drop shatter resistance (SRI), abrasion resistance (ARI), and water resistance (WRI) as a function of curing time. The charcoal briquettes were cured in an air-conditioned laboratory at 24°C, 40% relative humidity, and tested on days zero (as produced), 3, 5, and 7.

Drop shatter resistance index

The drop shatter index is a measurement of the particle's resistance to mechanical impact, which mainly happens during transportation and handling of the briquettes. Weighed samples, each containing 20 particles of coarse or agglomerated charcoal, were dropped twice from a 2 m height. For each sample, the shattered pieces were screened using a 2 mm aperture screen and weighed to determine the mass of the retained sample, which was recorded as the final weight. Percentage mass loss of charcoal and briquettes was determined from the difference between the initial mass and final mass. The %SRI was calculated using Equation [3] (Ajiboye *et al.*, 2016):

$$\% SRI = 100 - \% \text{ Mass loss} \quad [3]$$



Figure 1—Chatillon Amatek press

Compressive strength

A Chatillon Amatek DFE II instrument was used to measure the compressive strength of the coarse charcoal and charcoal briquettes. A sample was placed on the flat horizontal surface of the instrument, and slowly pressed between two parallel flat metal plates with facial areas greater than the projected area of the sample (20 and 50 mm diameters for the lower and upper plates, respectively). The cross-sectional area (A_c) of the particle was determined and the maximum length of the peak was horizontally displayed on the instrument as the fractural load (F_i) as the plate was vertically compressing the particle. The compressive strength of the sample was calculated using Equation [4] (Mangena *et al.*, 2004):

$$\text{Compressive strength} = \frac{F_i(N)}{A_c(m^2)} \quad [4]$$

Abrasion resistance index

ARI was measured by tumbling five weighed particles of charcoal or charcoal briquettes in a cylindrical drum of 70 × 35 mm ID at 50 r/min for 2 minutes. During tumbling, the cylindrical briquettes were abraded along the edges and became pillow-shaped. The load was then screened with a 2 mm aperture screen and the mass of the retained sample determined. Percentage mass loss of charcoal and briquettes was determined from the difference between the initial mass and final mass. The %ARI was calculated using Equation [5] (Venter and Naude, 2015):

$$\% ARI = 100 - \% \text{ Mass loss} \quad [5]$$

Water resistance index

The method for WRI determination was described in a study by Mangena *et al.* (2004). A single weighed sample was submerged in a beaker of cold water for 2 hours and inspected for disintegration, weighed, and then dried at atmospheric conditions for more than 6 hours with repeated weighing until no further significant mass loss was observed. The difference between initial and final mass of the particle sample was used to calculate the percentage water absorbed, and the WRI was calculated using Equation [6]:

$$WRI = 100 - \% \text{ Water absorbed} \quad [6]$$

Results and discussion

Proximate analysis

Table II shows the results for proximate analysis of the metallurgical grade, coarse charcoal (CC), and charcoal briquettes with mass concentrations of zero (binderless briquettes), 1, 3, and 5 wt% PVA binder. It was observed that the metallurgical-grade coarse charcoal had a low ash yield (below 3%), with a fixed carbon content of 66%. The PVA-bound briquettes, on the

Table II

Proximate analysis of charcoal briquettes (adb)

Specification	CC	0%wt	1%wt	3%wt	5%wt
Moisture	12.2	15.6	10.9	7.0	10.2
Ash	2.5	7.7	8.0	7.7	6.6
Volatile matter	19.2	21.0	22.8	22.0	20.6
Fixed carbon (by difference)	66.1	55.7	58.3	63.3	62.6

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other hand, yielded more ash (ranging between 6.5 and 8%) due to the high silica content of the ash, which was found to be 72%. The percentage fixed carbon for the produced charcoal briquettes with PVA ranged between 56 and 63%, while the volatile matter ranged between 20 and 23%. According to (Narjord (2017) these char characteristics are acceptable for metallurgical utilization in the production of steel. Table II indicates an increase in fixed carbon content with the addition of PVA.

Briquette mechanical analysis

Figure 2 shows the moisture content as a function of curing time for coarse charcoal, binderless, and 1, 3, and 5 wt% PVA-bound briquettes.

For the binderless briquette, the moisture content reduced from 14.7% on day zero to 4.1% on day 7, while in that curing period the 1 wt% PVA-bound briquette experienced an inherent moisture reduction of 4.4%. The 3 and 5 wt% PVA-bound briquettes had inherent moisture contents of 7.0% and 10.2%, respectively on day zero, which decreased to 4.0% on day 7. It was observed that moisture of the charcoal briquettes decreased with curing time to less than 4.5%. The mechanical properties of the CC, 0, 1, 3 and 5 wt% PVA briquettes as a function of curing time in relation with the decrease in moisture are depicted in Figures 3, 4, and 5.

Compressive strength

Figure 3 shows the briquette compressive strength as a function of curing time.

The binderless and 1 wt% PVA-bound briquettes failed to meet the mean compressive strength of 31 kg/cm² obtained for the coarse charcoal. It appears that briquettes produced with less than 3 wt% PVA were not amenable to compression, even if they were naturally dried for an extended period. This was probably due to weaker cohesion and adhesion forces between the low concentration PVA binder and the charcoal fines. According

to Rousset *et al.* (2011), the mean compressive strength for charcoal ranges between 10 and 80 kg/cm², with an ash content in the range of 2–5%. When the ash content ranged between 1 and 1.5%, the charcoal compressive strength was between 50 and 100 kg/cm². Both the 3 and 5 wt% PVA-bound briquettes attained the minimum required mean compressive strength after 3 days of natural curing, which ranged between 28 to 40 kg/cm² for the 3 wt% PVA-bound briquettes, and 64 to 115 kg/cm² for the 5 wt% PVA-bound briquettes.

Abrasion resistance

Figure 4 shows the briquette abrasion resistance as a function of curing time. The obtained results were compared with the coarse charcoal lumps.

It can be observed that the ARI for binderless briquettes continues to decrease with curing time, from 22% to 3% between days zero and 7. An initial 50% decrease in the ARI, from 20% to 10%, was observed in the 1 wt% PVA-bound briquettes between days zero and 3, followed by an increase to 15%. The decrease in ARI for both the binderless briquettes and the 1 wt% PVA-bound briquettes could be attributed to the low adhesion forces between charcoal fines and PVA binder at zero or low binder concentrations. For the 3 and 5 wt% briquettes the ARI increased to 80 and 84%, respectively, after 3 days of curing. Between 3 and 7 days of curing, the average ARI for both the 3 and 5 wt% PVA-bound briquettes was 82% with a standard deviation of 0.3. The ARI obtained for the coarse charcoal was 96%, which was not attained by the manufactured briquettes. According to Mangena (2001), a 25% fines content may be tolerated, therefore 75% ARI was chosen in this study as the minimum target value for abrasion resistance.

Drop shatter resistance

Figure 5 shows the effect of briquette curing time on SRI, with the SRI of coarse charcoal for comparison.

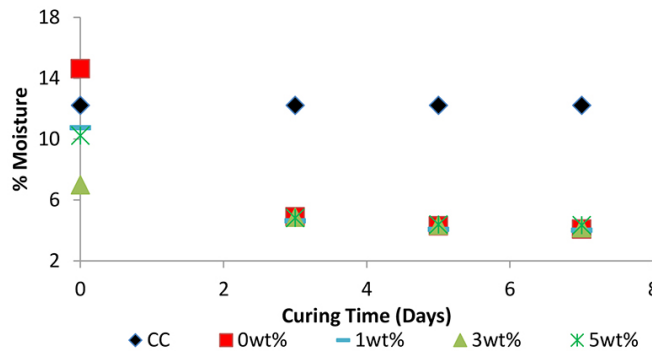


Figure 2—Percentage moisture as a function of curing time

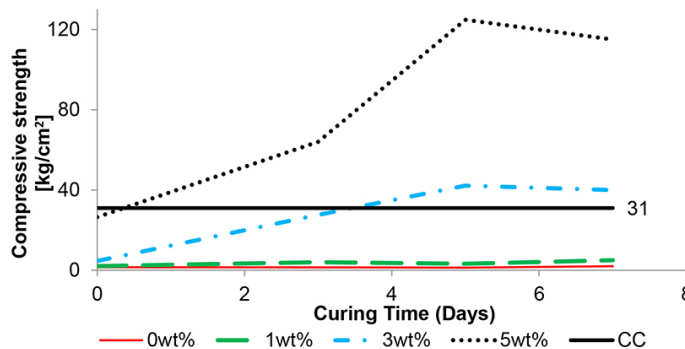


Figure 3—Compressive strength as a function of curing time

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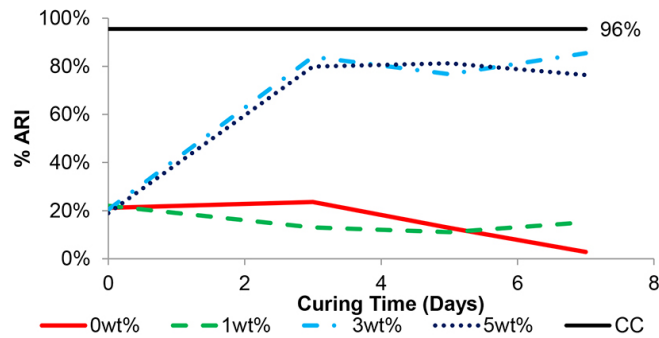


Figure 4—Abrasion resistance as a function of curing time

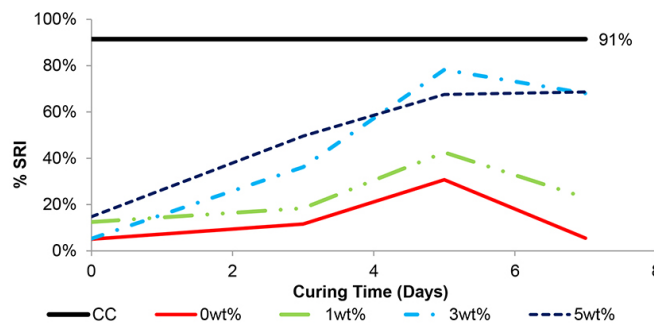


Figure 5—Shatter resistance as a function of curing time

In the first five days of curing, the SRI increased for all briquettes. It was observed that from days 5 to 7 of curing, a continuous decrease in SRIs of the charcoal briquettes resulted, with the briquettes becoming more brittle and breaking easily. The lower SRI for the binderless briquettes and the 1 wt% PVA-bound briquettes (SRI 5 and 23%, respectively) were obtained at inherent moisture contents of less than 4.4%, after 7 days of curing. The results obtained for both the 3 and 5 wt% PVA-bound briquettes (averaging 70%) were low compared to the shatter resistance of 91% for the coarse charcoal.

Water resistance

Table III shows the water resistance index (WRI) results obtained for all briquettes, along with the coarse charcoal, submerged in water for more than 2 hours.

The binderless briquettes and the 1 wt% PVA-bound briquettes disintegrated within a few seconds in the presence of water, which proved that the briquettes were not water resistant. Both the 3 and 5 wt% PVA-bound briquettes yielded a water resistance index between 73 and 75%. According to (Richards (1990), a WRI greater than 95% should be obtainable after 2 hours. Although lower than the suggested minimum acceptable WRI proposed by Richards, the WRI results obtained for the 3 and 5 wt% PVA-bound briquettes were higher than for the coarse charcoal. This was attributed to the high concentration of PVA binder on the surface of the briquettes. The wet compressive strength was also tested over a 9-day period at 3-day intervals, in order to determine the effect of PVA binder on the briquettes under rainy conditions. The results are shown in Figure 6.

It was observed that the wet compressive strength increased with curing time for the first 5 days. The insignificant increase in wet compressive strength observed for the coarse charcoal (from 20 to 32 kg/cm² over the 9-day period) was attributed to the charcoal's large surface area and high porosity. On day zero, the wet compressive strength for the 3 wt% briquette was

Sample	% Water absorbed	% WRI
CC	42	58
0%wt	100	0
1%wt	100	0
3%wt	25	75
5%wt	27	73

2 kg/cm², increasing to 36 kg/cm² due to natural curing for 5 days. After day 5, the compressive strength of the 3 wt% PVA-bound briquettes decreased to 32 kg/cm². The compressive strength of 5 wt% PVA-bound briquettes, with an initial value of 12 kg/cm², increased significantly to 83 kg/cm², but subsequently decreased to 78 kg/cm² after 7 days of curing. The results indicate that the rainy seasons will have a greater effect on the mechanical properties of the lower concentration PVA-bound briquettes, which will therefore require care in handling and storage on site. Table IV shows a summary of the mechanical properties for the binderless and PVA-bound briquettes compared to the coarse charcoal after 7 days of curing.

From Table IV it is clear that the mechanical properties of the binderless and 1 wt% PVA-bound briquettes are inferior to those of the coarse charcoal (CC). On the other hand, the 3 and 5 wt% PVA-bound briquettes showed superior performance compared to the raw charcoal in terms of compressive strength and water resistance. The abrasion resistance and shatter resistance of the 3 and 5 wt% PVA bound-briquettes were slightly inferior to the coarse charcoal. The increase in binder concentration may have improved these results, to produce agglomerates comparable to coarse charcoal.

Conclusions

This study showed that charcoal briquettes produced from wood

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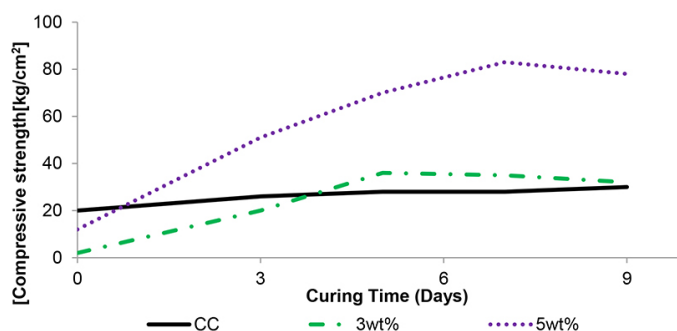


Figure 6—Wet compressive strength as a function of curing time

Table IV

Mechanical properties as a function of curing time (days)

	CC	0wt%	1wt%	3wt%	5wt%
CS(kg/cm ²)	31	2	5	40	115
ARI, %	96	3	15	85	76
SRI, %	91	5	23	68	69
WRI, %	58	0	0	75	73

charcoal fines with a PVA binder concentration of 3 wt% and higher have suitable compressive strength and water resistance properties for metallurgical application as a carbonaceous reductant. The binderless and the low concentration (1 wt%) PVA-bound briquettes, on the other hand, exhibited poor mechanical properties. The mechanical properties of the briquettes with 3 wt% PVA binder improved with curing time, with the compressive strength increasing from 5 to 42 kg/cm² after 5 days of curing. The 5 wt% PVA-bound briquette yielded the highest compressive strength, which increased from 26 to 125 kg/cm² after 5 days of curing. This is more than a 300% increase in compressive strength compared to the coarse charcoal at 31 kg/cm². The 3 and 5 wt% PVA-bound briquettes yielded water resistance indices of 75 and 73%, respectively, compared with the benchmark of 58% set by the coarse charcoal.

The results show that the charcoal fines will require an appropriate storage facility to avoid contamination. The 3 and 5 wt% PVA-bound briquettes will also require a storage facility in order to avoid water damage during prolonged rainy periods. Based on the proximate and physical analyses obtained for the 3 and 5 wt% PVA briquettes, in comparison to the coarse charcoal, the results show that the briquettes can be used as a carbonaceous reductant for metallurgical applications. Future work should include an increase in binder concentration to above 6 wt% to improve the ARI and SRI. Beneficiation to remove extraneous sand/debris from the discarded charcoal fines should also be investigated to reduce the ash content before briquetting.

Acknowledgement

The information presented in this paper is based on research which is financially supported by the South African Research Chairs Initiative (SARChI) of the Department of Science and Technology and National Research Foundation of South Africa (Coal Research Chair Grant No. 86880). Any opinion, finding, or conclusion or recommendation expressed in this material is that of the author(s) and the NRF does not accept any liability in this regard. Facets of the study have been presented at the North West

University, Potchefstroom campus and the Tshwane University of Technology.

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