



Attempts to produce blast furnace coke from Victorian brown coal. 2. Hot briquetting, air curing and higher carbonization temperature



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HIGHLIGHTS

- Hot briquetting and air curing caused significant increase in compressive strength.
- Hot briquetting reduced the reactivity.
- High carbonization temperature reduced reactivity and surface area.
- The proportion of graphitic structure remained small.
- Compressive strength and reactivity remained higher than those of a typical BF coke.

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ABSTRACT

Attempts have been made to obtain a substitute for blast furnace (BF) coke from a Victorian brown coal mixed with a tar derived from brown coal under conditions sufficiently mild to be economical. Changes to the procedure used in a previous attempt include hot briquetting of the coal–tar mixture at 150 °C rather than ambient, the addition of air curing at 200 °C and carbonization at higher temperature, 1200 °C, rather than 900–950 °C. All these changes led to a decrease in reactivity and increase in compressive strength. In addition, the higher carbonization temperature led to a decrease in surface area. Thus the combined changes led to an increase in strength and decrease in reactivity and surface area. This is the first time a carbonized product has been prepared from brown coal in good yield and of lower reactivity than brown coal char when compared with BF coke. However, the reactivity and surface area remain too high for the product to be used as a substitute for BF coke.

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1. Introduction

The most popular method of producing iron from its ores is to react the ore with carbon, usually coke, in a blast furnace (BF). Coke is a hard, large-pored carbonaceous product, formed by pyrolyzing coking coals, which, when heated, become plastic and then coalesce into a coherent mass of lumpy coke [1,2]. The coke acts as fuel source and the reducing agent in a blast furnace and also provides the permeable support for the burden of iron ore. Its properties of high mechanical strength and low reactivity [3] towards BF gas are necessary for an efficient BF operation [4]. Coking coals will become increasingly harder (and more expensive) to obtain if the anticipated growth in global steel demand [5] occurs and the more easily mined coking coal becomes depleted [6]. Victorian brown coal (VBC) can be mined by open cut and, so, is cheap. It also

contains little inorganic material, which is a favourable factor for iron production in a blast furnace [7–9]. At present, the cost of coking coal is about USD112–116/tonne [10] compared to about USD35/tonne [11,12] for brown coal dried by conventional methods. The price of dried brown coal would be expected to fall when innovative methods of drying [13] are adopted. However, in its as-mined condition VBC does not form coke when pyrolyzed, only producing a char which is too reactive towards BF gas to be used in a BF.

In our earlier work it was shown that products obtained from VBC by ambient temperature briquetting of a mixture of VBC and tar obtained from VBC and subsequent carbonization had a high compressive strength, but too high a reactivity in CO₂ to be used as BF coke [14,15]. Hot briquetting of a mixture of coal and char both derived from higher rank, but non-coking, coals can convert them to materials which when carbonized are of sufficiently high strength and low reactivity to be used in a BF [16]. More generally, hot briquetting low grade, but higher rank, coals can give carbonized products of increased strength and decreased reactivity,

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which would make them more suitable for metallurgical usage [17]. This work has encouraged the use of hot briquetting in studies aimed at obtaining a coke substitute from low rank coals (brown coal or lignite). However, to date, few of these studies have considered the reactivity of the final product as well as strength. In East Germany after 1945, carbonized products were obtained from hot briquetted brown coal with suitable reactivity, strength and porosity to be used in a BF [18,19], but the production process was complex and gave low yields, and is no longer used [20]. Higgins and Kennedy [21–28] attempted to make a BF coke substitute from hot briquetted VBC, but the product, though of sufficient strength, was too reactive. Hot briquetted acid washed VBC after carbonization gave a strong product, but the reactivity of this material was not compared with that of a conventional BF coke [29].

Other studies of BF coke substitutes from hot briquetted brown coals have not considered reactivity but only strength. Recently, French and Reeves [30] patented a process in which low rank coals were briquetted at low temperature (<66 °C) and subsequently carbonized without added binder to give a product of comparable compressive strength and bulk density to BF coke. However, again, no information was given on reactivity in CO₂ or strength of the product after exposure to CO₂ at high temperature (~1000 °C). Mori et al. [31] briquetted VBC at a high force and high temperature, up to 230 °C, without binder, and again obtained a product after carbonization of high compressive strength. They examined the effect of changes in the briquetting conditions and carbonization temperature on the strength, bulk density and microscopic structure of the products and related their results to earlier work on the properties of low rank coals. They did not examine the reactivity of their products in CO₂ at high temperature.

Therefore, none of these prior studies has indicated how the reactivity of a BF coke substitute from brown coals could be reduced economically while giving a product of sufficient strength. This suggested that it might be useful to attempt to produce a BF coke from hot briquetted VBC–binder mixtures, being careful to measure, at least approximately, many of the properties that are important for a BF coke and studying how these properties vary with production conditions. Since air curing has often been found to be beneficial in improving the strength of carbonized materials [32–34], its effects on the properties of hot briquetted samples have also been examined in the present study. In this initial screening study, the crucially important reactivity, as well as the compressive strength, of the products are determined by methods suitable for small samples and these properties are then related to other characteristics of the final carbonized products, such as bulk density [35], surface area [36] and microscopic structure [31,37]. In addition, the extent of graphitisation [38,39] was estimated by Raman spectroscopy.

2. Materials and methods

2.1. Materials

VBC was obtained from an open cut mine located in Loy Yang, Victoria, Australia and milled to <3 mm. VBC tar (referred to below as tar), which was a by-product of char production, was sourced from Australian Char Pty Ltd., Morwell, Victoria. The tar was soluble in tetrahydrofuran (THF) and dichloromethane [14] and about 72% (v/v) boiled below 320 °C [40]. A BF coke sample was obtained from Nippon Steel Corporation, Japan. THF of Liquid Chromatography grade was sourced from Merck Australia, Kilsyth, Victoria.

2.2. Analyses of materials

The moisture content of VBC and BF coke was determined as the loss in weight when an as-received sample was heated in a flow of

N₂ at 105 °C for 4 h. For ashing, samples of VBC, tar and coke were dried at 105 °C in a flow of N₂ for 4 h to determine the moisture content, and then heated in air at 815 °C for 2 h. The VBC was analyzed for volatile matter by HRL Technology Pty Ltd., Victoria. Analyses for C, H, N and S were carried out by HRL Technology (VBC) and by Campbell Microanalytical Laboratory, University of Otago, New Zealand (tar, coke). O was obtained by difference. (For VBC, O was obtained using the mineral and inorganic contents of the coal rather than the ash yield.) The analyses of coal, tar and BF coke are shown in Table 1. The standard deviations of the ash yields, based on duplicate determinations, were ±0.02 wt%db (VBC) and ±0.7 wt%db (coke). The uncertainties in the VBC analyses were ±0.3 wt%db for C, ±0.1 wt%db for H, ±0.05 wt%db for N and ±0.03 wt%db for S and those for coke and tar analyses were ±0.15 wt%db for C, ±0.06 wt%db for H, ±0.04 wt%db for N and ±0.1 wt%db for S. The composition of the ash of VBC is given in Table 2.

2.3. Binder impregnation and briquetting the mixture

Prior to impregnation with binder, VBC was dried at 105 °C in a flow of N₂ and ground to <0.15 mm. Tar was dissolved in THF, then mixed with ground VBC at ambient temperature so as to incorporate 0–15 wt% tar. The THF was recovered by rotary evaporation. The mixture was dried under a flow of N₂ at 105 °C, ground to <0.15 mm and dried again as before to 1–2 wt% moisture content, as determined by an OHAUS MB45 halogen moisture analyser. A tar-THF solution rather than tar was used to promote penetration of tar into coal pores.

About 1.3 g of coal–tar mixture was placed into a 6.5 mm radius die set at room temperature. Then the die was heated to 150 °C by a heating element attached to it at a rate of 10 °C/min. After confirmation that the contents had reached the required temperature, force was applied using an INSTRON 5569 series Mechanical Tester at a rate of 10 kN/min until the force reached 20 kN and the force held for 10 or 30 min. The force was then released and the die was cooled to ambient temperature to recover the briquette, which was weighed. The radius and thickness of the briquettes were about 6.5 and 8.0 mm respectively.

2.4. Air curing

The briquettes were heated in a flow of air (300 mL/min) at 200 °C for 2 h [32,33,41] and then cooled to ambient temperature

Table 1
Analyses of materials.

Analysis ^a	VBC	Tar	Coke
<i>Proximate</i>			
Moisture (wt%,ar)	60.0	–	3.0
Ash (wt%db)	3.25	0.0	12.0
Volatile matter (wt%db)	49.4	–	–
Fixed carbon (wt%db)	47.4	–	–
<i>Ultimate (wt%db)</i>			
Carbon	65.7	74.0	86.2
Hydrogen	4.70	9.36	0.4
Nitrogen	0.60	0.62	1.01
Sulphur	0.66	0.40	0.4
Oxygen (by diff.)	26.1	15.6	0.03
Atomic H/C ratio	0.85	1.51	0.06

^a The uncertainties in the values are given in Section 2.2.

Table 2
Ash composition of VBC (wt% of total ash).

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	K ₂ O	MgO	Na ₂ O	CaO	SO ₃	P ₂ O ₅
56.5	19.2	2.3	8.0	0.2	2.4	4.1	1.0	5.4	0.2

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