

POTENTIAL USE OF LOCAL LIGNITE FOR SEMI-COKE PRODUCTION THROUGH LABORATORY-SCALE CARBONIZATION AND PROCESS PARAMETER OPTIMIZATION

Shahid Hussain Dahri¹, Fahad Irfan Siddiqui², Safiullah Memon³, Ansar Ahmed Memon^{*4}

^{1,2,3,*4}Department of Mining Engineering, Mehran University of Engineering and Technology, Jamshoro, Sindh, Pakistan

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Corresponding Author: *

Ansar Ahmed Memon

Abstract

Pakistan holds an estimated 185 billion tonnes of coal, of which more than 99 % is Sindh lignite characterised by high moisture, ash, sulphur, and volatile-matter (VM) contents. These properties limit its direct use in cement plants and steel mills, which instead rely on costly imported coal. This study evaluates the upgrading of Lakhra lignite into a smokeless, carbon-dense semi-coke through low-temperature carbonization. A custom air-tight iron carbonization chamber coupled with a steel cyclone for tar and gas separation was designed and fabricated for use inside a temperature-controlled crucible furnace. Eighteen carbonization runs were carried out on three size fractions ($-25/+10$, $-10/+5$ and $-5/+2$ mm) at three temperatures (400, 600 and 750 °C) for two carbonization times (2 and 4 h). Raw feed and carbonized products (36 samples) were characterised by proximate, sulphur and gross calorific value (GCV) analysis. Carbonization reduced VM from an average of 57.6 % to as low as 0.4 to 6.7 %, raised fixed carbon (FC) from 32.1 % to 84 to 95 %, and increased GCV from 10,771 to about 13,000 Btu/lb. Treatment at 750 °C lowered sulphur from 8.3 % to 6.6 %. Balancing product quality against energy consumption, the optimum conditions were a $-10/+5$ mm fraction carbonized at 600 °C for 2 h, producing a semi-coke comparable to commercially reported products. A preliminary cost analysis indicates a favourable margin against imported coal.

I. INTRODUCTION

Global energy demand continues to rise with population growth; world population is projected to approach ten billion by 2040, and primary power demand is expected to reach roughly forty-two quadrillion BTU by the same year [1]. For more than two centuries, close to 90 % of energy for power and transport has come from non-renewable fossil fuels, and at present consumption rates conventional oil reserves may be exhausted within half a century [2]. Coal therefore retains a central role in the energy mix, supplying close to 40 % of global electricity, but its combustion

releases particulate matter and toxic species owing to inherent impurities [3].

Devolatilization of low-rank coals improves combustion efficiency and reduces particulate emissions, because volatile matter is a primary precursor of such emissions and can be removed by carbonization, leading to cleaner coal utilisation [4], [5]. Lignite is widely used for power generation because of its high reactivity and abundant volatile matter, but its high moisture (typically 10 to 50 %) lowers plant efficiency, raises transport costs and promotes spontaneous combustion; drying and devolatilization are therefore required before use [6]. Coal is also the

feedstock for metallurgical coke, conventionally produced by carbonizing premium coking coals at temperatures up to about 1200 °C. Because such coals are scarce and expensive, with prices that peaked near US\$400/tonne in 2011, and there is growing interest in substituting carbon products derived from low-rank coals for part of the blast-furnace charge [7].

Pakistan is endowed with about 185 billion tonnes of coal, of which 184 billion tonnes (over 99 %) is Sindh lignite, sufficient in principle to generate 40,000 MW for centuries [10]. Despite this, cement and steel industries import higher-grade coal because indigenous reserves carry high moisture, ash, sulphur and volatile matter, causing inefficient combustion, noxious emissions and equipment fouling [8], [9]. Upgrading by carbonization, while varying particle size, temperature and carbonization time, offers a route to enrich fixed carbon and remove volatiles, making local lignite attractive to these industries. The objectives of this study were to design and fabricate a laboratory-scale carbonization test setup capable of operating in an inert atmosphere, and to determine the optimum temperature, particle-size and carbonization-time ranges for carbonizing local Lakhra lignite into semi-coke.

II. BACKGROUND AND RELATED WORK

Low-temperature carbonization (LTC) of brown coals yields valuable coke-oven gas (COG), tar and semi-coke, and has long been preferred industrially; consequently, moisture content, temperature and dehydration during LTC have been studied extensively [11], [12]. Moisture in lignite occurs as inherent and external water; hydrophilic oxygen-bearing functional groups form hydrogen bonds with water molecules and adsorb them on coal surfaces, raising moisture content [6].

Steel manufacturing depends heavily on coal: the World Coal Association reports that roughly 70 % of global steel output is coal-dependent [13]. The depletion of hard coking coals has driven development of technologies to produce semi-coke from lower-rank coals; Nippon Steel and Sumitomo Metal established dry charging processes such as Coal Moisture Control (CMC)

and the Dry-cleaned and Agglomerated Pre-compaction System (DAPS), increasing the proportion of semi-soft coking coal in the blend and reducing coke-making costs [14]. More recently, high-strength, low-reactivity coke has been produced from low-grade carbonaceous materials by depositing pyrolysis tar onto the char, broadening the range of feedstocks suitable for coke making [15]. Under CMC pretreatment, lignite moisture was reduced to about 6 % and high-quality coke obtained [12]. The carbonization kiln, together with the burning and coke chambers, governs heat transfer, so temperature distribution in the coking chamber has been examined in detail [16].

Mollah et al. combined Victorian brown coal with a coal-derived binder to form briquettes; although the products had adequate compressive strength, they remained too volatile for blast-furnace use [7]. Binderless briquetting has likewise been reviewed as a route to densify lignite into stronger, more transportable fuel briquettes without added binder [17]. In a laboratory-scale rotary-kiln study, lignite was carbonized at 550 to 750 °C for 15 to 120 min across several size fractions; temperature was found to control the surface area of the carbonate, which expanded roughly threefold relative to the raw coal, with about 60 min being optimal for surface development [18].

Carbonization performs best in the absence of oxygen, since oxidation lowers carbonization rate, coke yield and coke quality [19], [20], [21], [22], [23]. The product slate depends on process type: low-temperature coke-making (around 700 °C) yields semi-coke with less COG and ammonia but more tar, the COG having a high calorific value and low hydrogen content, whereas high-temperature processing produces more COG with little tar and a lower-calorific, hydrogen-rich gas [24]. In the presence of alkaline compounds the micropore volume of the resulting carbon increases during pyrolysis, accompanied by CO and CO₂ evolution indicative of gasification [25].

A. Types of Carbonization

On the basis of operating temperature, carbonization is classified as low-temperature carbonization (LTC, 500 to 700 °C) and high-

temperature carbonization (HTC, 900 to 1200 °C). LTC gives a coke yield of about 75 to 80 % retaining 5 to 15 % VM; the product is mechanically weak and unsuitable as a metallurgical fuel but burns smokelessly, making it an excellent domestic fuel, and its by-product gas has a high calorific value. HTC gives a 65 to 75 % yield with only 1 to 3 % VM, producing a hard, porous, strong coke suitable for metallurgical use, with a higher-volume but lower-calorific gas.

B. Products of Carbonization

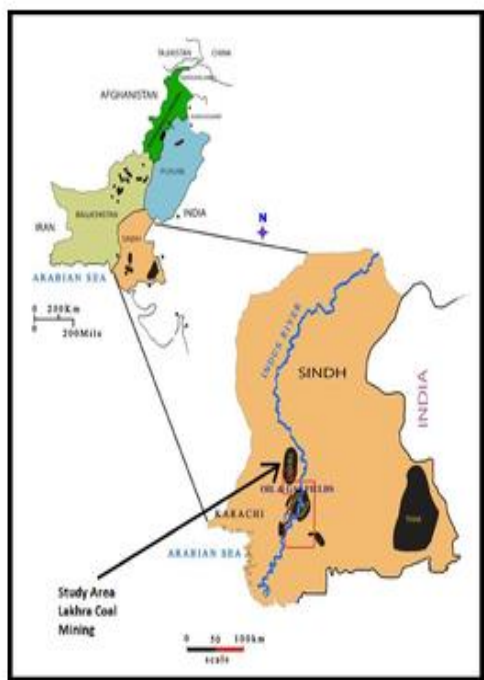
Coke-oven gas, mainly carbon monoxide, hydrogen and methane with some carbon dioxide and nitrogen, is a valuable by-product; about one tonne of coke yields roughly 360 m³ of COG [26], [27]. China alone produces about 70 billion Nm³ of COG annually, only a fraction of which is used as fuel, so its release without utilisation represents a significant waste and pollution source [28]. In carbonization, around 70 % of the coal mass converts to solid coke and the remaining 30 % to gaseous and liquid (tar) by-products [29]. Coal tar, accounting for about 2.5 to 4 % of the feed, is a feedstock for aromatic chemicals; more than 90 %

of compounds such as anthracene, pyrene and phenol are recovered from it [30], [31], [32], [33].

III. SITE LOCATION AND GEOLOGY

Coal samples were obtained from mines of the Indus Coal Company operating in the Lakhra coalfield, selected for its accessibility and proximity to the university. Lakhra coal, first reported in 1853, is classified as lignite with a calorific value of about 5,219 to 13,555 Btu/lb; the field is fully developed and holds mineable reserves of roughly 146 million tonnes, its suitability for power generation having been confirmed by an independent feasibility study.

The field forms a doubly plunging anticline, the Lakhra Anticline, with a north-easterly axis, gentle folding and strata dipping at about 7°. A set of faults parallel to the axis dips 70 to 80° with a small down-throw. The principal seams are Dhanwari, Lailian and Kath; the Lailian seam, persistent across the area, is about 3 m thick, while the overall average seam thickness is about 1.5 m, with overburden of 50 to 150 m above the first mineable seam.



(a) Location of Lakhra coalfield, Sindh



(b) Geological map of the field

Fig. 1. Study area: (a) location of the Lakhra coalfield in Sindh, Pakistan, and (b) geological map of the field.

IV. MATERIALS AND METHODS

A. Sample Preparation

About 15 kg of run-of-mine coal was received in bags and handled to preserve its as-received condition. Lump coal was crushed manually and split with a Jones riffle sample splitter to separate coarse and fine fractions. The riffled coal was then screened on a mechanical sieve shaker into three size fractions: $-25/+10$ mm, $-10/+5$ mm and $-5/+2$ mm. For proximate and ultimate analysis, sub-samples were pulverised in a mortar and passed through a $250\text{-}\mu\text{m}$ sieve as required by the analysers.

B. Equipment

Sample preparation employed a mechanical sieve shaker and a temperature-controlled crucible furnace (Fig. 2). The furnace provided the controlled, oxygen-deficient environment required for carbonization. Proximate, sulphur and calorific analyses were performed on a LECO TGA-701 thermogravimetric analyser, a LECO SC-832 sulphur and carbon analyser and a LECO AC-500 isoperibol calorimeter, respectively (Fig. 3) [34].



(a) Mechanical sieve shaker



(b) Crucible furnace

Fig. 2. Sample-preparation and carbonization equipment: (a) mechanical sieve shaker; (b) temperature-controlled crucible furnace.



(a) TGA-701



(b) SC-832



(c) AC-500

Fig. 3. Analytical instruments used for characterisation: (a) LECO TGA-701 (proximate analysis); (b) LECO SC-832 (sulphur/carbon); (c) LECO AC-500 (gross calorific value).

C. Carbonization Test Setup

A bespoke carbonization unit was designed to satisfy the inert-atmosphere requirement of the process. An air-tight cylindrical chamber was machined from iron, chosen for its stability up to the 750 °C target and sized to fit the inner dimensions of the crucible furnace. The chamber

was connected by pipe to a copper cyclone that separated condensable tar from non-condensable gases: tar drained from the lower cone into a collection pan, while gases discharged from the upper outlet and were vented outside the laboratory by an exhaust fan. The complete assembly is shown in Fig. 4.



(a) Prototype carbonization chamber



(b) Carbonization test in furnace



(c) Fluids (tar) exhaust assembly



(d) Cyclone separator

Fig. 4. Fabricated laboratory-scale carbonization setup: (a) air-tight prototype chamber; (b) chamber heated inside the crucible furnace during a test; (c) fluids (tar) exhaust assembly; (d) copper cyclone separator.

D. Carbonization Procedure and Test Matrix

In each run a weighed charge was sealed in the chamber, which was then placed in the furnace and connected to the cyclone and exhaust train, with a beaker positioned to collect tar. The furnace was heated to the set point; once reached, the soak time was started and held for the required interval, after which the furnace was switched off and allowed to cool to ambient temperature before the chamber was opened and the product weighed and labelled. Eighteen carbonization runs were

performed, covering three size fractions, three temperatures (400, 600, 750 °C) and two carbonization times (2 and 4 h). In total, 36 samples (18 raw feeds and 18 products) were characterised.

E. Performance Indicators

The solid product yield (recovery) of each run was computed as the ratio of product mass to feed mass:

$$\text{Recovery (\%)} = (m_{\text{product}} / m_{\text{feed}}) \times 100 \quad (1)$$

The corresponding devolatilization mass loss is the complement of the recovery:

$$\text{Mass loss (\%)} = 100 - \text{Recovery (\%)} \quad (2)$$

The relative improvement in energy density achieved by carbonization was evaluated from the gross calorific values of product and feed:

$$\Delta \text{GCV (\%)} = [(\text{GCV}_{\text{product}} - \text{GCV}_{\text{feed}}) / \text{GCV}_{\text{feed}}] \times 100 \quad (3)$$

V. RESULTS AND DISCUSSION

A. Physical Changes and Product Recovery

Raw lignite was moist, hard and grey, whereas the carbonized products were drier, more brittle and darker, with 40 to 60 % mass loss. The recovery decreased with increasing severity, falling from about 53 % at 400 °C/2 h to about 40 % at 750 °C/4 h, consistent with progressive devolatilization (Table I).

TABLE I

REPRESENTATIVE CARBONIZATION RUNS AND SOLID PRODUCT RECOVERY

Run	Feed (g)	Temp. (°C)	Time (h)	Product (g)	Recovery (%)
1	60	400	2	32.0	53.3
2	60	400	4	28.0	46.7
3	60	600	2	31.0	51.7
4	60	600	4	26.0	43.3
5	60	750	2	24.5	40.8
6	60	750	4	24.0	40.0
9	60	600	2	30.0	50.0
12	60	750	4	24.0	40.0
18	60	750	4	24.0	40.0

B. Effect of Carbonization on Coal Quality

Averaged over all 18 raw samples, the feed contained 57.6 % VM, 32.1 % FC, 10.8 % ash and 8.3 % sulphur, with a GCV of 10,771 Btu/lb. Carbonization dramatically restructured this composition. For the optimum product (−10/+5

mm, 600 °C, 2 h) VM fell to 6.7 %, FC rose to 84.4 %, and GCV increased to about 12,654 Btu/lb (Fig. 5). The large reduction in VM and the corresponding enrichment in fixed carbon confirm effective devolatilization.

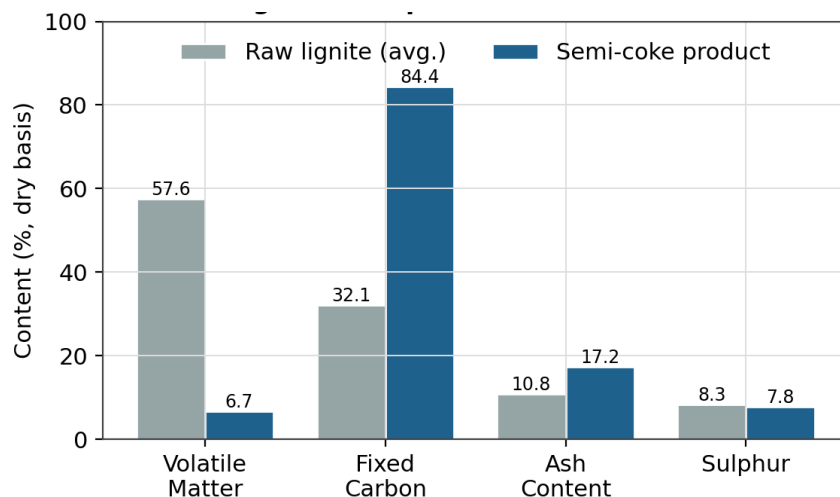


Fig. 5. Comparison of average raw lignite and the optimum semi-coke product (dry basis)

C. Effect of Temperature

Temperature was the dominant variable. Increasing it from 400 to 750 °C reduced VM from 26.0 % to 1.3 % and raised FC from 68.2 % to 88.9 % (Fig. 6). Ash increased from 13.7 % to 18.8 %, reflecting concentration of mineral matter as combustible volatiles were driven off. Importantly, higher temperature lowered sulphur from 12.2 % at 400 °C to 6.6 % at 750 °C, while GCV declined modestly from 13,190 to 12,524

Btu/lb (Fig. 7). Thus 750 °C gives the most complete devolatilization and the greatest sulphur removal, but at the cost of slightly reduced calorific value and higher energy input. A comparable trend has been reported for lignite pyrolysis, where raising the final temperature to about 920 °C reduced volatile matter by roughly 95 % while enlarging the pore structure of the resulting semi-coke [35].

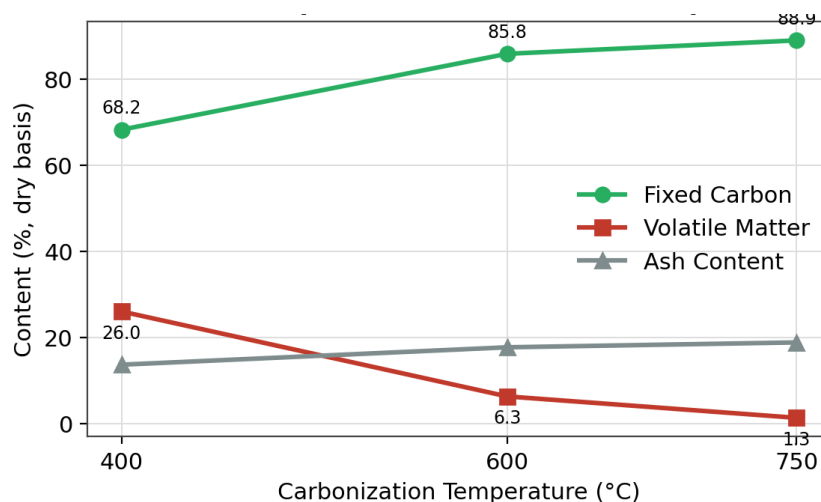


Fig. 6. Effect of carbonization temperature on volatile matter, fixed carbon and ash content.

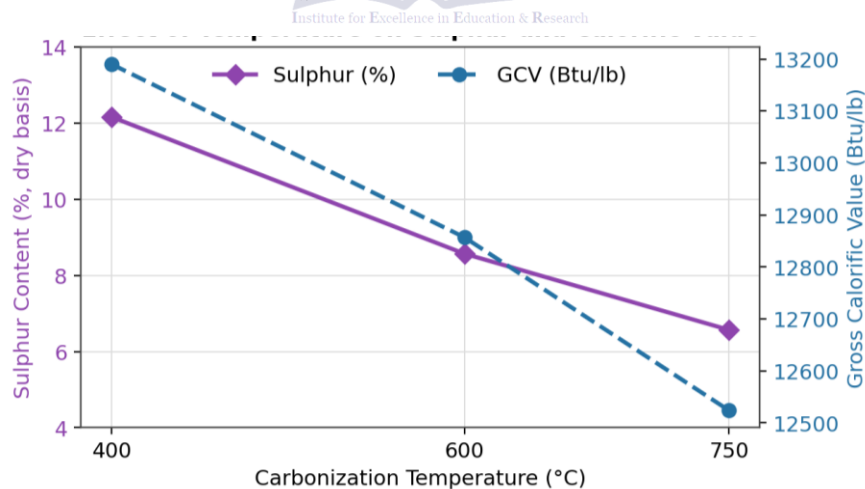


Fig. 7. Effect of carbonization temperature on sulphur content and gross calorific value.

D. Effect of Particle Size

Particle size exerted a comparatively minor influence on VM and FC, which followed similar trends across all fractions (Fig. 8). The intermediate -10/+5 mm fraction gave the best

balance, combining the highest fixed-carbon and calorific values with the lowest ash, attributable to more uniform heat penetration than the coarse fraction and less mineral-rich fines than the finest fraction. Sulphur was largely insensitive to size.

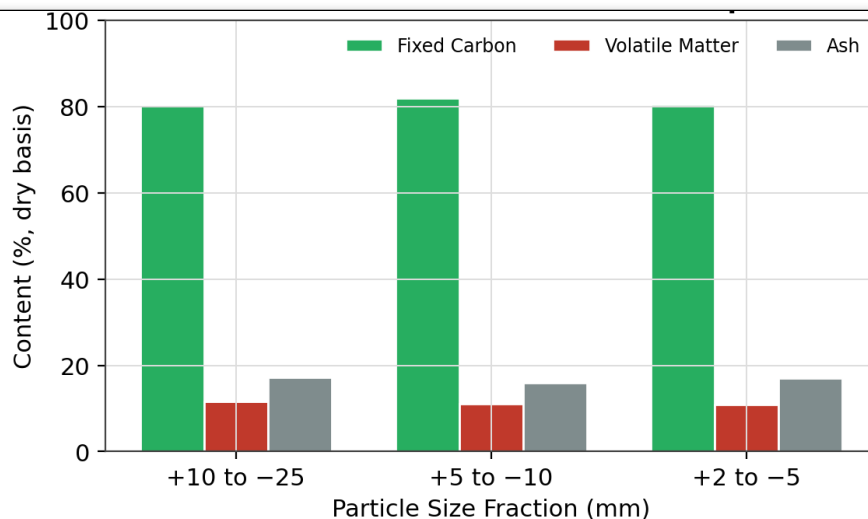


Fig. 8. Effect of particle-size fraction on the proximate composition of the products.

E. Effect of Carbonization Time

Extending the carbonization time from 2 to 4 h further reduced VM (12.3 % to 10.2 %) and raised FC (77.4 % to 84.6 %), but the incremental gain in fixed carbon was only about 7 percentage points

for a doubling of furnace time (Fig. 9). Gross calorific value was essentially unchanged. Because the additional energy expenditure outweighs the marginal quality benefit, the shorter 2-h soak is preferable on economic grounds.

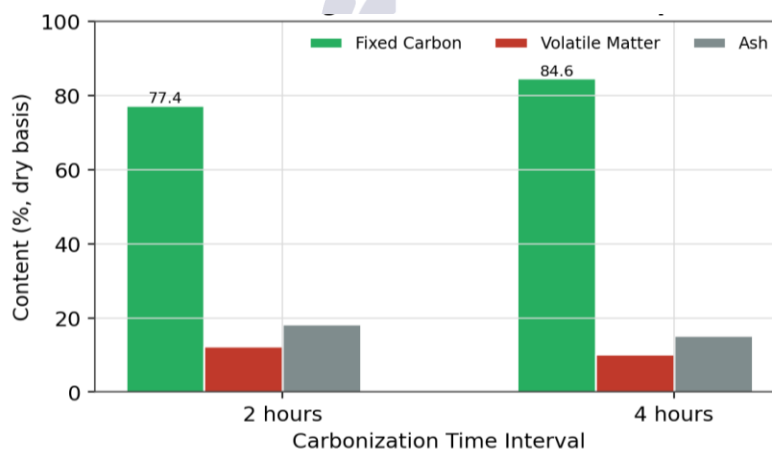


Fig. 9. Effect of carbonization time on the proximate composition of the products.

F. Optimum Conditions

Averaging each parameter (Table II) identifies the trade-off. Although 750 °C maximises fixed carbon and sulphur removal, raising the temperature a further 150 °C above 600 °C yields only about 3 % more fixed carbon, an

unfavourable return on energy. Likewise, the −10/+5 mm fraction and the 2-h soak give the best quality-to-energy balance. The recommended conditions are therefore a −10/+5 mm feed carbonized at 600 °C for 2 h.

TABLE II

AVERAGE EFFECT OF TEMPERATURE, SIZE AND TIME ON PRODUCT QUALITY

Variable	Level	VM (%)	Ash (%)	FC (%)	S (%)	GCV (Btu/lb)
Temp. (°C)	400	26.03	13.67	68.20	12.16	13,190
	600	6.31	17.71	85.82	8.57	12,857
	750	1.34	18.84	88.93	6.57	12,524
Size (mm)	−25/+10	11.70	17.27	80.38	8.91	12,846
	−10/+5	11.07	15.87	81.96	8.75	12,987
	−5/+2	10.92	17.08	80.61	9.63	12,738
Time (h)	2	12.30	18.26	77.36	9.17	12,895
	4	10.16	15.22	84.61	9.02	12,820

G. Benchmarking and Economic Consideration

The optimum product compares well with semi-coke values reported in the literature and by commercial suppliers; its fixed-carbon content (about 84 %) matches or exceeds several benchmarks, although its ash is higher owing to the mineral-rich nature of Lakhra lignite (Fig. 10). On cost, at an indicative recovery of 40 % about

2.5 tonnes of Lakhra lignite (around PKR 13,750) are required per tonne of semi-coke, against an average imported-coal price near PKR 24,900 per tonne. The resulting margin of roughly PKR 11,000 per tonne is sufficient to absorb carbonization costs while remaining free of foreign-exchange exposure, supporting the economic case for local processing.

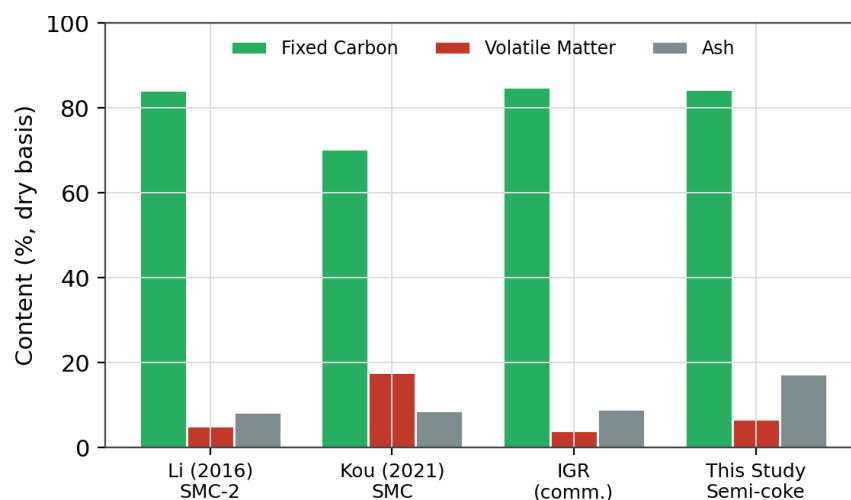


Fig. 10. Benchmarking of the optimum product against reported and commercial semi-coke compositions.

VI. CONCLUSIONS

A laboratory-scale carbonization unit, an air-tight iron chamber with a cyclone for tar/gas separation, was successfully designed, fabricated and operated to upgrade Lakhra lignite to semi-coke. Across 18 runs, temperature, particle size and carbonization time all influenced product quality, with temperature being dominant. Carbonization cut volatile matter from an average

of 57.6 % to as little as 0.4 to 6.7 %, raised fixed carbon from 32.1 % to 84 to 95 %, and increased gross calorific value to about 13,000 Btu/lb, while higher temperatures reduced sulphur from 8.3 % to 6.6 %. The −10/+5 mm fraction gave the best results among the sizes tested. Although 750 °C and a 4-h soak maximised fixed carbon, the marginal gains did not justify the extra energy; balancing quality and energy use, the optimum

conditions were a $-10/+5$ mm feed carbonized at 600 °C for 2 h. The resulting smokeless, carbon-dense semi-coke is comparable to commercial products and economically attractive relative to imported coal.

VII. RECOMMENDATIONS

Future work should characterise the carbonization by-products (coke-oven gas and tar) and their potential applications, so that the full resource value of indigenous lignite can be realised. Small-scale carbonization plants could be installed at the Lakhra field to manufacture this high-energy-density, smokeless fuel for fuel-intensive industries. Because Lakhra lignite is comparatively high in sulphur, similar studies are recommended on lower-sulphur Sindh deposits; in particular, Thar lignite (sulphur below about 1 %) is expected to yield a superior semi-coke.

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