Development of carbon composite iron ore slime briquettes for using in ironmaking

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Abstract : Iron ore slime is a micro-fine of size below 20 μ m in majority and rich in alumina and silica. It is neither suitable for pelletization nor sintering. Jhama coal is not also used in the iron making because of its poor caking property and thermal stability of lump. Therefore, the composite fluxed briquettes in a combination of these two materials were made to use this in low shaft furnace or mini blast furnace wherein, Jhama coal will be acting as a reducing agent and heat source, which may replace the use of costly metallurgical coke in the subsequent iron making process. The present work develops a process for making composite briquettes with slime and Jhama coal with a fluxing agent without any external binder or very little amount of binder to replace the fluxing agent. The developed briquettes provide sufficient strength of up to 104 kg/cm², 70% reducibility index, 28% reduction degradation index, and good metallization after reduction which shows its suitability for low shaft furnace or mini blast furnace.

Key Words: C- composite briquettes, Jhama coal, slime, iron making, mini blast furnace

1. INTRODUCTION

In present practice, iron ore fines are utilized in the blast furnace through sintering and pelletization. While a larger fraction is used in sintering to maintain permeability, very fine fractions of it are used in pelletizing. But, only very high-grade fines are used in these processes. For using low-grade fines, beneficiation is necessary before agglomeration for a blast furnace. Many fines contain very high alumina and silica with low iron like slime, overburden, etc. are treated as a waste till date. Similarly, some coals are non-cocking and have very high ash content are not used. Investigators have tried to use these non-coking coal and low-grade iron ore from the last few decades. CSIR-NML has used low shaft furnace in the sixties wherein pig iron was produced by low shaft furnace using non-coking and semi-coking coal. Several low shaft furnaces worldwide is producing iron using OXICUP [1] wherein iron ore briquettes are used.

Iron ore carbon composite briquettes have been made in different techniques of without binder and with the binder. The thermal plasticity of coal has been used to bind the particle, where, carbonaceous material and iron ore are closely adjoined at the micro-level [2-6]. Here no binder has been used. It is prepared by pressing a mixture of the fine coal particles and iron ore fines in a cylindrical die followed by heat treatment. When the mixture of coal (~ 60%) and iron ore fines (~40%) are heated at 300-400 °C temperature, the terry portion of coal becomes viscus which on pressing, form a good bonding. Investigators have used binder also in the composite briquettes when there is no binding property in the used carbonaceous materials. Michael et al. [7] used Australian hematite iron ore, low volatile charcoal, and 2 % starch as a binder. The green briquettes were heated at 1300 - 1350 °C to achieve the cold compressive strength beyond 3800 N. Mousa et al. [8] has used 1.8% of torrefied pelletized sawdust (TPS), Briquettes with 86.2% of steel mill residues and 12% cement have been produced on an industrial scale with sufficient mechanical strength.

In most of the composite briquetting techniques, the primary aim was to use it in the blast furnace to reduce the energy consumption and control the temperature of the thermal reserve zone [2-7]. Oita Works No. 2 Blast Furnace [6] has used carbon composite briquettes up to 54 kg/tHM the carbon consumption can be decreased significantly. To implement this in a large blast furnace, a rapid curing process of briquettes using steam is developed. In an experimental study [5], a 95 % reduction degree has been achieved in about 10 min descent time to the 1100°C heat reserve zone to bottom. They have also reported that these new agglomerates decreases the fuel rate and CO₂ emission and shorten the thermal reserve zone.

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Cold bonded carbon composite briquettes have been used in a low shaft furnace or mini blast furnaces [9, 10] to utilize the steel plant's waste oxides and carbonaceous materials. Self-reducing agglomerates called C-bricks have been made using sludges, mill scale, blast furnace dust, sponge iron fines, and ore fines [1]. These are melted together with scrap in an OXYCUP® shaft furnace and produced blast furnace quality pig iron. They successfully utilized the above waste materials for making blast furnace quality pig iron.

Cold bonded carbon composite self-reducing briquettes have been used in rotary hearth furnace [11,12]. Blast furnace dust was used as a carbon source in briquettes containing magnetite concentrate (30-40%) and mill scale (30-40%) [11] and found a good reduction. Combined binder as cornstarch for low-temperature bonding and sodium silicate for high-temperature bonding was used by Han et al. [12] to achieve good strength in briquettes.

From the above studies, it is obvious that carbon composite briquettes have been prepared by several investigators for the utilization of both iron oxide fines and C containing fines. They used several techniques of preparing the briquettes and utilized these briquettes in the blast furnace, mini blast furnace, low shaft furnace or rotary hearth furnace. Many of them have found advantages in blast furnace operation and successfully produced pig iron form this. The present study aims at utilizing two waste materials viz, iron ore slime and Jhama coal. Iron ore slime is generated during ore washing. It is a micro-fine material with high gangue materials. It is not suitable for sintering or pelletization due to its high fineness and presently it has no use in metallurgical purposes. Jhama coal has very poor caking property and poor strength at high temperatures. Therefore, it is neither used in iron making nor other metallurgical purposes. If the slime is agglomerated with Jhama coal for its use in iron making, Jhama coal will be acting as both reducing and heating agent. Thus, the agglomerate of slime with Jhama coal can reduce the coke rate in the blast furnace. Many investigators have tried to make iron ore briquettes with cement as a binder but, the briquettes with slime and Jhama coal have not been tried out so far. Therefore, in this study, the composite briquettes were made and subsequently characterized for using it in a low shaft furnace or mini blast furnace. Since, iron ore slime contains a high amount of alumina and silica inorganic binders such as bentonite, cement, etc. in high proportion for this will not be suitable to make the briquettes, usable in the subsequent iron making process. Therefore, it was thought to use such a material that will act as a binder as well as a fluxing agent in iron making stage. Calcined lime is such a suitable candidate that can give binding as well as increase the basicity of briquettes to improve its chemical and physical properties. Therefore calcined lime was used as a binder to make these composite briquettes.

2. EXPERIMENTAL

2.1. Making of briquettes

Iron ore slime for this study was taken from Tata steel, Noamundi mines and lime was used as a bonding material and prefluxing whose chemical composition is shown in Table 1. Chemical analysis of Jhama coal is also presented in Table 1.

Jhama coal was sized to different size fractions like 0.21 mm, -1 mm, -2 mm, etc. Slime was charged in a ball mill for 1-2 min and then screened to -72 mesh. The size fraction of slime after milling is presented in Table 2.

Iron ore slime, Jhama coal, and calcined lime fines were mixed in varying ratios in a rotary mixer as shown in Table 4 in a dry condition for 15 minutes. Then adding around 5-7% water with the mixture, briquetting of the moist mixture was done into the rectangular or cylindrical briquettes of 8 cm³ and 50 cm³ volume, respectively with pressure ranging between 50 kg/cm² and 100 kg/cm². Natural curing of the briquettes was done under the ambient condition for a period of 6-12 hours. Then it was kept in a closed humidified chamber under the humidity of 80-95% for 5- 10 days. Drying of cured briquettes was done under the ambient condition for a period of 2 - 6 days and sized it for testing. Characterization of the sized briquettes was done through measurement of strength properties at high temperature, reducibility index (JIS: M8713), reduction degradation index (JIS: M8720) to evaluate its suitability in a low shaft furnace or mini blast furnace.

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	Fe _{total}	CaO	SiO ₂	Al ₂ O ₃	Na ₂ O	K ₂ O	S	Р	Ash	VM	FC
Slime	59.88	0.98	4.4	5.09	0.053	0.003	0.011	0.18			
Lime		91	2.5	1.7							
Jhama Coal	-	-	7.44	0.42	4.41	0.27	0.46	0.12	16.37	10.6	73.03

 Table 1 Chemical composition of slime and lime (wt%)

Table 2 Size fraction (BIS mesh) of slime(wt%)

+72	+72 -100	+100 -150	+150 -200	-350
21.1	7.5	4.0	6.7	60.61

2.2. Characterization

To measure the cold crushing strength (CCS) of the briquettes the briquettes were sized to 20-25 mm blocks by cutting with a hacksaw and grinding in emery paper to give it a regular cube shape. Then the briquettes cubes were subjected to CCS measurement. The material testing machine (Hounsfield) was used for this measurement. The maximum load per unit area for breaking off a briquettes cube is termed as CCS.

The briquettes cubes were heated at varying temperatures to assess its thermal stability. After cooling the briquettes cube, the CCS was measured as above. Further, the briquettes cubes were kept in a tubular furnace fitted with an Instron and after heating at elevated temperature, the compressive strength was measured online.

The large briquettes were broken into small lumps of size between +10 and -30 mm for the testing reduction degradation index (RDI). RDI of several briquettes was measured as per standard: JIS: M 8720-2001. The percentage of fines generation (-3.15 mm) after reduction and subsequent tumbling in a drum (200 mm length and 130 mm dia) is termed as RDI.

The percentage of reduction of ore in reducing the gas atmosphere at elevated temperature is called the Reducibility index (RI). Good RI is important in BF operation, especially, to achieve good productivity and low coke rate. Broken lumps of briquettes in the size range between +10 and -30 were subjected for reducibility Index (RI) testing as per standard: JIS: M 8713-2000 at 900 °C. With small lumps, some 25x25x25 mm cube shapes briquettes were also reduced to check the CCS after reduction. The CCS after reduction was checked by the above method.

Apart from the standard RI, the reduction of briquette was measured at further high temperature viz. 950, 1000, and 1050 °C also to assess its suitability at high temperature in reducing atmosphere.

The softening melting test was done as per the conventional process to assess its behavior of softening and melting in the furnace.

3. RESULTS AND DISCUSSION

3.1. Properties of briquettes with the very fine size of Jhama coal

Slime briquettes with varying compositions have been made as given in Table 3. The size of Jhama coal particle was-0.21 mm and the size fraction of slime is shown in Table 2. All the prepared briquettes were subjected to CCS measurement at room temperature as well as after heating at 600 and 800°C. The results are shown in Table 3.

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Sample	C	omposition Briq	uettes made		CCS value of different slime briquettes after heating at different temperature(kg/cm ²)			
No	Iron Ore slime, %	C % (as Jhama coal)	OPC Cement, %	CaO	Roon temperature	After heating at 600 °C	After heating at 800 °C	
1	90	0	10	0	52	-	26	
2	90	0	0	10	53	-	53	
3	83	7	0	10	48	73	60	
4	59.5	30.5	10	0	28	28	13	
7	96	0	0	4	45	74	58	
8	96	0	4	0	51	55	63	

Table 3 (Composition	of different	briquettes	made and	their CO	CS values aft	er heating a	t different ten	perature

The above results in Table 3 show that at high C level the (Briquette 4) briquettes show very poor strength. This is due to the smooth surface property of coal. Further, the table indicates that lime and cement shows similar strength properties in briquettes. Cement contains significant alumina and silica. Due to high alumina and silica, it will consume more lime during smelting in a low shaft furnace and produce high slag volume resulting increase in the coke rate. Here slime itself contains very high alumina and silica. To make a suitable slag in a low shaft furnace or mini blast furnace, a significant quantity of lime addition is necessary with the charge. Accordingly, the calcined lime was preferred as a binder in briquettes for the next sets of experiments.

In view of the above, the next set the briquettes were made with calcined lime and a lower amount of Jhama coal as shown in Table 4. The table shows that at high C percentage strength is lower. After heating at 800°C, the strength decreases drastically. Initially at 400 °C increase in strength was observed that may be due to the drying of briquettes. During heating at the rate of 6 °C/min in the chamber furnace took time to remove moisture, but, prolong heating or heating at much higher temperature should deteriorate CCS. Figure 1 shows that strength decreases with an increase in coal percentage at high temperature and it goes up to a very low level for the 30% coal in briquettes.

Sample No	Iron ore slime,			ating at different temp, kg/cm ²		
	%	coal)		At room temp	500 °C	800 °C
1A	90	0	10	70	127	144
2A	83	7	10	81	137	96
3A	75	15	10	59	93	56
4A	70	20	10	70	-	26

Table 4 Strength of briquettes with varying percentage of Jhama coal

Reducibility (RI) and reduction degradation index (RDI) versus C percentage of the above briquettes are plotted and shown in Figure 2. RI decreases to some extent for a high percentage of C. However, the value was found to be very encouraging. Internal C has no role in improving RI. Only CO gas acts as a reducing agent. It is envisaged from the figure that up to 70% indirect reduction is possible in the blast furnace (BF) condition for 15% carboncontaining briquettes. This result indicates that in a blast furnace or low shaft furnace mainly CO gas is there in the stack region. Therefore, the reduction of briquettes will be happened by CO gas only. But, internal carbon in the briquettes will remain unchanged throughout the stack region at 900 °C. RDI of 20% coal containing briquettes was found to be around 27%, which may be acceptable for low shaft furnace and mini blast furnace.



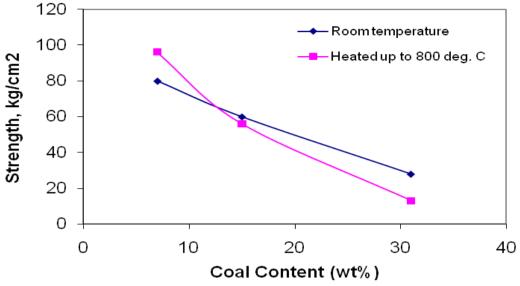
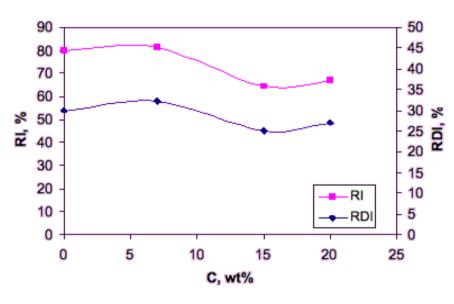


Figure 1 Deterioration of compressive strength with increase in coal percentage



RI and RDI vs C in briquettes

Figure 2 RI and RDI property of briquettes at varying C percentage

The compressive strength of the reduced briquettes (20-25 mm cube) in 28-30% CO atmosphere (using RI setup) was measured and found to be lower than the non-reduced briquettes. The effect of C in briquettes on CCS of reduced briquettes after the RI test is shown in Figure 3. The strength gradually reduces as C content increases. This is because of two reasons one is the initial strength of higher C containing briquettes is less than the lower one. Another reason is that C remains unreacted during the reduction in CO-reducing atmosphere resulting in an increase in C content in the reduced sample. For higher C containing briquettes, this increase in residual C is much higher than the lower C containing briquettes. The presence of this high amount of C affects more on the strength properties of high C briquettes.

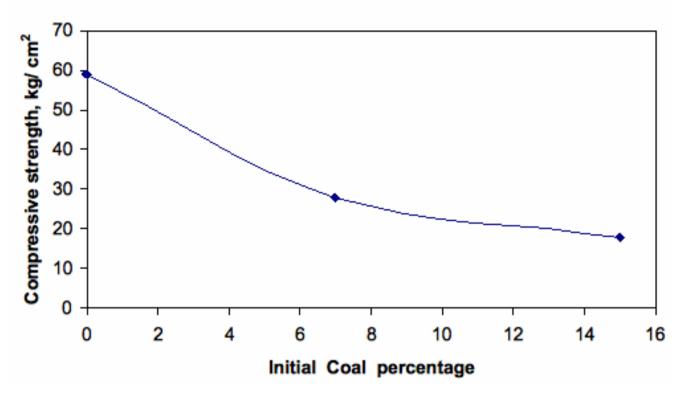


Figure 3 Strength of briquettes after RI test

3.2. Properties of briquettes with the coarser size of Jhama coal

Jhama coal is very difficult to grind to a very fine level in the muller mixer due to its hardness. Earlier experiments show that C in Jhama coal does not participate in the reduction of iron oxide in the CO-gas atmosphere. As low shaft furnace gas will contain some CO_2 (5-6%), some reaction with C would be there as per the following reaction;

 $CO_{2} + C = 2CO...(1)$

Since CO content is very low, the requirement of C also will be very low. Therefore, considering this minor requirement of fine coal and difficulties in grinding the coal size was reduced to -2 mm. The size fraction of -2 mm coal is given in Table 5.

+14	+30 -14	+60 -30	+72 -60	+100 -72	+150 -100	+200 -150	-200
6.2	35.8	25.6	2.0	8.5	4.3	5.0	12.6

Table 5 Size fraction of -2 mm coal (wt%)

The strength of the briquettes decreases with an increase in heating temperature (Figure 4). 20% of coal briquettes show much higher strength than 30 % coal briquettes. 20% coal of -2 mm size and -0.21mm size shows similar CCS (70 kg). Further, from the earlier experiment, it is clear that C has a very minor role in the reduction of iron oxide in a gaseous reducing atmosphere. Therefore, 20% coal of -2 mm size was taken for the rest of the experiments for subsequent study.

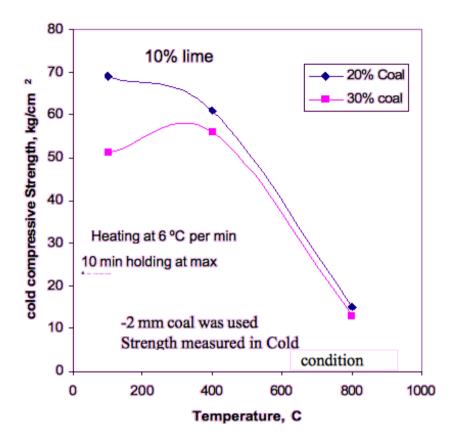


Figure 4 Effect of heating on strength properties of coal composite briquettes made with -2 mm coal fines

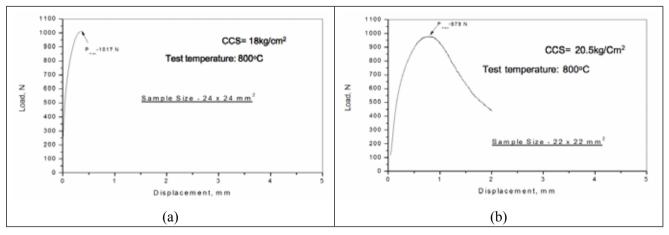


Figure 5 CCS of briquettes (with 20 % Coal of -2 mm size) measured at elevated temperature

The briquettes were kept in a tubular furnace fitted with Instron and heated to 800 °C. The strength of briquettes was measured at elevated temperature to examine the behavior of it in low shaft furnace bed during movement at high temperature. The data are shown in Figures 5a and 5b. It shows 18-20 kg strength at 800°C which looks to be very good.

3.2.1. Reduction test at reducing atmosphere for 30 % C containing briquettes

The reduction property of C composite briquettes has been tested in two conditions such as (i) in an oxidizing atmosphere and (ii) in the CO atmosphere as discussed below.

Reduction of Briquettes in an oxidizing atmosphere

When samples were heated in a chamber furnace in closed lid condition, the samples got to reduce and the weight losses of respective samples are shown in Table 6. Within 30 min samples were sufficiently reduced. The percentage of metallization is shown in Table 7. It is evident from the table that at 1000 °C, the percentage of metallization is not so high, but it increases significantly up to 66% at 1150 °C for 20% C containing briquettes. i.e this is the temperature zone where maximum reduction happens. The possible reaction for this reduction are;

 $C + O_2 = 2CO (g)$...(2) $Fe_2O_3 + CO = FeO + CO_2$...(3) $FeO + CO = Fe + CO_2$...(4)

Although low shaft gas hardly contains any oxygen, some (5%) CO_2 is there which may react with C in briquettes to produce CO gas for reduction of iron oxide as per Reaction 1.

Table 7 shows the degree of metallization of several C composite briquettes heated at different temperatures with varying time. It is depicted from the figure that when briquettes were heated for a longer time keeping a constant temperature at 1000 °C, metallization decreases. This may be because initially briquettes reduce but after a certain time, reoxidation happens in the briquettes in the air atmosphere of the furnace. Further, with an increase in heating temperature, the degree of metallization increases because of faster reaction at high temperature. The amount of C in briquettes has also been found as a major factor for the reduction because of the briquettes with 30% coal show better metallization (84%) than that with 20% coal.

San	nple	Temp, °C	Time, min	Initial weight, g	Final weight, g	% wt loss
20% Coal	10% lime	1150	30	31.088	22.605	27.28
			45	28.885	21.065	27.07
			60	28.472	20.362	28.48
30%Coal	10% lime	1150	30	27.584	19.201	30.39
			45	34.790	24.025	30.94
			60	27.806	19.438	30.09
20% Coal	10% lime	1000	30	29.031	25.221	13.12
			45	30.146	22.692	24.72
30%Coal	10% lime	1000	30	27.819	23.020	17.25
			45	29.081	25.335	12.88

Table 6, Amount of weight losses in different condition of heating for different briquettes

 Table 7 Degree of metallization in reduction in air atmosphere

Sample type	Curing temp, °C	Curing time, min	Total iron in briquettes, %	Metallization, %
20% coal	1000	30	40.98	26.1
30% coal	1000	30	36.87	14.23
30% coal	1000	45	35.032	11.7
20% Coal	1100	45	50.32	65.97
20% coal	1150	60	49.78	66.47
30% coal	1150	45	44.19	84.39

Reduction of briquettes in CO atmosphere

In order to examine the metallization in CO atmosphere 500 g samples were heated in the RI set up, then reduced the sample by flowing 30% CO + 70 % nitrogen-containing gas. The reduction results are shown in Table 8.

Although, Sl No 2 and 3 are done in identical temperature and time duration, weight loss in Sl. No. 3 is lower than that in No 2. This is because in No 3 CO gas was started at very low temperature (500 °C) during the adiabatic heating condition, however, in No 2 it is started after reaching 1100 °C in isothermal condition. During heating to reach 1100 °C in the air atmosphere, C burns out in Sl. No 2, but in No 3, C did not burn due to the absence of oxygen (CO atmosphere). During carbon burning, the above Reactions 1 through 4 happen which reduces the iron oxides present in the briquettes. Therefore, the percentage of metallization is much higher in Sl No 2 than Sl No 3. For the same reason, the percentage of metallization is high in Sl No 1 also because co gas was started after reaching the sample temperature to 1000 °C. It is also evident from the table that the temperature increase from 1000 °C to 1050 °C does not have any significant effect on improving the percentage of metallization. In BF or mini BF stack, there will be CO atmosphere, therefore the percentage of metallization is expected to remain as low as 57%, which is also encouraging.

Sl No	Temp ^o C	CO gas started at ^o C	Initial wt, g	Final wt, g	% loss	Metallization, %
1	1000	1000	500	355	29	76
2	1050	1050	500	344	31.2	77
3	1050	500	500	360	28	57

Table 8	Weight loss	on reduction	in CO	atmosphere
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It may be noted that after the reduction of briquettes in CO atmosphere, almost all C in the briquettes has been found unreactive. Therefore, if these briquettes are charged in the BF or mini BF, or the low shaft furnace, after the reduction in the CO atmosphere at the shaft, most of the C will remain unreacted. This C will reach the cohesive zone and may increase the viscosity of bosh slag and deteriorate the properties of the softening melting zone. Therefore, a softening melting property study is imperative and explained below.

Softening melting Properties of 20% C containing briquettes were done at Tata Steel. The obtained curve is shown in Figure 6. It shows 1150 °C as the softening point. However, the completion of softening i.e melting point could not be identified because of high C content in briquettes restricted the flow of liquid or due to high alumina and lime content of briquettes. Almost whole C remains unreacted because of the CO atmosphere in the reaction capsule. Since, melting and droplet formation do not happen in the test, it may be presumed that these briquettes will form a viscous cohesive zone in a blast furnace or mini blast furnace which will affect the permeability of the furnace and reduce the production. Therefore, 100% briquettes charging with the furnace but not 100%.

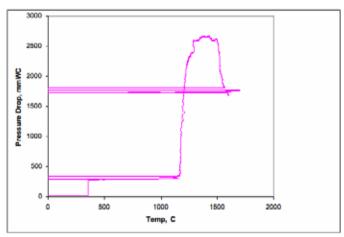


Figure 6 Softening and melting behavior of briquettes (20% Jhama coal)

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3.2.2. Briquettes with cement and lime as combined binder

Only lime bonded briquettes show up to 70 kg/cm² CCS. To make stronger briquettes, cement and lime combined binders have been used. The properties of the briquettes are presented in Table 9. It appears stronger briquettes than only lime or only cement.

Briquettes composition, %			CCS after cu	uring and dryin	ng kg/cm ²		CCS after reduction at	
Coal	Slime	lime	cement	3 days	5 days	7 days		900°C
20	72	4.8	3.2	50	76	104	28.7	13 kg/cm ²

Table 9 Properties of briquettes with cement and lime combination

Even after the reduction in CO atmosphere, it shows 13 kg/cm² CCS. Therefore, the cement lime combined bonded slime-Jhama coal briquettes can be used in a mini blast furnace or low shaft furnace more suitably than only lime or only cement-bonded briquettes.

From the above study, it is envisaged that briquettes have good strength at normal temperature. However, when it is heated to ambient temperature its CCS deteriorates to a limited extent but it does not crumble. It can be charged in the mini blast furnace or low shaft furnace suitably. The reduction property of the briquettes shows that carbon remains unreactive in the CO atmosphere up to around 1100 °C but the briquettes soften at 1150 °C. Therefore, this carbon may reach the cohesive zone and reduce the slag viscosity and permeability of the furnace. Due to this 100% charging is not recommended. However, this C present in slag at and below the cohesive zone may react directly with iron oxides and increase the rate of direct reduction and productivity. Some unburnt carbon may reach the raceway of the furnace and may burn to produce CO gas which will provide the thermal energy to the furnace. Thus, C in the composite briquettes may increase productivity and provide thermal input. Therefore, the developed composite briquettes may be charged in a mini blast furnace and low shaft furnace to utilize the slime and Jhama coal

4. CONCLUSIONS

- (i) Burnt lime has very good potential to provide strength to the briquettes. The slime-Jhama coal composite briquettes with 10% lime show good strength after 7-10 days of curing in the normal atmosphere under the shed in a moist condition.
- (ii) Strength at high temperature was also measured using Instron fitted with tubular furnace and found appreciable CCS (11-15 kg/cm² at 800 °C) The briquette has very good RDI (28%) and RI (>70%). Briquettes get reduce in the oxidizing atmosphere above 1000°C and it is significant at and above 1150°C.
- (iii) Binder in the combination of 3.2% cement and 4% lime showed better strength properties than10% lime.
- (iv) The carbon in the briquettes remains almost unreactive after a reduction in CO atmosphere. Due to this when these briquettes will be charged in the furnace C will come to and below the cohesive zone which will help direct reduction of iron oxide. Highly viscous slag has been found in the softening melting test because of the presence of C in slag. Therefore, to maintain the permeability of the furnace 100% briquettes charging may not be possible, however, a partial replacement of iron ore with these briquettes are recommended. A plant trial to assess the performance of the developed briquettes in a mini blast furnace is required for this.

5. ACKNOWLEDGMENT

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