# Studies on Briquetting and Characterization of Coke Oven Sludge

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*Abstract—* **Significant quantities of sludge and slag are generated as waste material or by-product every day from steel industries. They usually contain considerable quantities of valuable metals and materials. It is generally possible to recover some values by physical or chemical mineral processing techniques. Briquetting is such a process to re-cycle this waste coke oven sludge as a useful fuel. In the present study experiments were conducted to determine optimum parameters of briquetting like binder content, water content, pressure and temperature of heat treatment .shatter resistance tests were carried out on briquettes obtained under different conditions. The physical properties of the feed material, the end use of the briquettes and the cost of binder addition have to be taken into account before a selection is made. In the experiments different binders used are starch, bentonite, molasses, iron ore fines and water.**
**The briquettes are obtained at high pressure and they undergo mechanical and treatment according to the characteristics of the processed material, the binder used and the desired product.**

*Keywords—* **Coke oven sludge, Iron ore, molasses, briquettes, compressive strength, DTA/TGA analysis.**

## I. INTRODUCTION

Briquetting is the process of converting low bulk density biomass into high and energy concentrated fuel briquettes. Briquetting units shape fine materials into larger forms of varied shapes, sizes and volumes. In general ,briquetting can be done by using binders, sometimes binder less briquetting also can be made. Binder less Briquetting is always preferred when possible, since it is less complicated and less expensive. Usually binder is used for reasonable briquettes. If the cohesive forces between the particles are low, a binder has to be used before agglomeration. The physical properties of the feed material, the end use of the briquettes and the cost of binder addition have to be taken into account before a selection is made.

The briquettes are obtained at a low or high pressure and undergo mechanical and treatment according to the characteristics of the processed material, the binder used and the desired product, according to the material, the binder that may be necessary and end- product required. The main stages of the process can be as follows:

Product preparation unit (screening-crushing, drying, sieving) Dosing and mixing of product with binder Briquetting unit Post treatment of the briquettes.

The goal of this work is to convert currently unusable industrial coke oven fines into briquettes/pellets that can be useful for industry. They can also be useful for other high-volume markets, including foundry sand mold binders. Previously, the investigators used these in combination with calcium hydroxide as an additive while calcium chloride was added as hardening accelerator. However, the additions of chloride salts have a detrimental effect because chlorine causes corrosion in processing equipment. Therefore, other potential hardening accelerators were investigated during this project.

## II. MATERIALS AND METHODS

### A. Materials Used

Coke oven fines/ sludge, Iron ore fines/ powder for briquetting in this experiment were procured from Visakhapatnam steel plant, vizag, Andhra Pradesh, India.

### B. Equipments Used

- A briquetting machine (pressing type) was used for preparing briquettes with 15000Kg loads applied by hydraulic pressing
- A universal testing machine of capacity 10000KN was used for measuring briquettes compressive strength with constant crosshead speed of 1mm/min.
- A DTG-60H instrument was used for studying DTA/TGA thermal analysis.
- Varnier calipers was used for measuring diameter, thickness of briquettes

## III. EXPERIMENTAL PROCEDURE

In this process, coke oven fines briquettes were made by adding different binders such as starch, bentonite, molasses, Iron ore fines. The binder concentrations used in the experimental studies were 2%, 0.5%, 0.5% and 1% based on coke oven fines weight. (to particulate iron ore of 10gm) and size of the particles below 250μm. The whole powders are mixed homogeneously by using 5 to 15% by weight of water (based on total sample weight) .This mixture was poured into

molds having 25 mm internal diameter and put under pelletizing machine and a load of 15kT was applied. The Briquettes thus formed are removed from the mold and weighed. The green pellets are than air dried in an open atmosphere for 48 hours and then heated in a furnace at $200\pm10^oC$ for 24 hours. The sample is removed from the oven, cooled and weighed. Initially sample no 1, 2, 3 and 4 were prepared. For each sample briquettes were prepared by varying weight of the sample mixture from 6gr, 8gr, 10gr, 12gr, 14gr. For each of the above briquettes compressive strengths have been calculated by using 10,000KN capacity UTM with constant cross head speed of 1mm/min. The same tabulated in table no 2, 3,4 and 5.from the results it can be observed that the briquettes of 10gr weight of sample has a better compressive strength than the all other in each composition with L/D of 4.2.as the coke oven sludge percentage increased from 95.5% to 97.0% compressive strengths are increased, however 95.5% coke oven sludge sample of 10gr wt. has showed the best compressive strength of $14.68N/mm^2$ .this sample L/D ratio 4.2 (for 10gr )kept as constant and compressive strengths were studied by increasing iron ore percentage i.e. from 2% , 4%,6%,8% and 10%. The same was tabulated in table1 (sample no5 to 9). Compressive strengths of these samples were tabulated in table. 6 and observed that 6% iron ore briquette possesses high compressive strength. Differential Thermal Analysis(fig.1) and Thermo Gravimetric analysis (fig.2) were carried out and tabulated in table8. 6% of iron ore sample mixture taken as ideal because of heat evolved is high, and by changing the percentages of starch and molasses studies were carried out same recorded as sample no 10 to 14. Compressive strengths are tabulated in table.7 and DTA (fig.3)/TGA(fig.4) in Table.9.

In preparing the samples the following precautions should be taken.
1. The pulverized ore must have a sufficiently fine particle size distribution.
2. Sufficient moisture is added to make the sample sticky enough to pelletize but not so much moisture that the ore becomes "muddy".
3. Binders concentration is chosen in such a way, to hold the particle grains together after the pellet is dried and hardened

**Characterisation:** Thermal analysis (DTA/TGA) of the samples were carried using A DTG-60H instrument of SHIMADZU.

## IV. RESULTS AND DISCUSSION

TABLE.1 A TYPICAL COMPOSITION OF COKE OVEN FINE PELLETS/BRIQUETTES WITH DIFFERENT BINDERS AND MOLASSES

| S.No | Sample No | Coke oven Sludge (%) | Starch (%) | Mola-sses (%) | Ben-tonite (%) | Iron ore fines (%) |
|---|---|---|---|---|---|---|
| 1 | Sample No 1 | 95.5 | 2 | 1 | 0.5 | 1 |
| 2 | Sample No 2 | 96 | 2 | 0.5 | 0.5 | 1 |
| 3 | Sample No 3 | 96.5 | 1 | 1 | 0.5 | 1 |
| 4 | Sample No 4 | 97.0 | 1 | 0.5 | 0.5 | 1 |
| 5 | Sample No 5 | 95.5 | 1 | 0.5 | 1 | 2 |
| 6 | Sample No 6 | 93.5 | 1 | 0.5 | 1 | 4 |
| 7 | Sample No 7 | 91.5 | 1 | 0.5 | 1 | 6 |
| 8 | Sample No 8 | 89.5 | 1 | 0.5 | 1 | 8 |
| 9 | Sample No 9 | 87.5 | 1 | 0.5 | 1 | 10 |
| 10 | Sample No 10 | 91.5 | 0.25 | 1.25 | 1 | 6 |
| 11 | Sample No 11 | 91.5 | 0.5 | 1.0 | 1 | 6 |
| 12 | Sample No 12 | 91.5 | 0.75 | 0.75 | 1 | 6 |
| 13 | Sample No 13 | 91.5 | 1.0 | 0.5 | 1 | 6 |
| 14 | Sample No 14 | 91.5 | 1.25 | 0.25 | 1 | 6 |

TABLE 2 COMPRESSIVE STRENGTHS OF DIFFERENT BRIQUETTES PREPARED WITH SAMPLE 1

| S.No | Briquette Weight (gms) | Sample Length L (mm) | Sample Dia D (mm) | L/D | Compressive Strength (N/mm²) |
|---|---|---|---|---|---|
| 1 | 6 | 80 | 25 | 3.2 | 13.58 |
| 2 | 8 | 95 | 25 | 3.8 | 12.58 |
| 3 | 10 | 105 | 25 | 4.2 | 14.68 |
| 4 | 12 | 110 | 25 | 4.4 | 12.74 |
| 5 | 14 | 120 | 25 | 4.8 | 11.03 |

TABLE.3 COMPRESSIVE STRENGTH OF THE BRIQUETTES PREPARED WITH SAMPLE - 2

| S.No | Briquette Weight (gms) | Sample Length L (mm) | Sample Dia D (mm) | L/D | Compressive Strength (N/mm²) |
|---|---|---|---|---|---|
| 1 | 6 | 80 | 25 | 3.2 | 12.78 |
| 2 | 8 | 95 | 25 | 3.8 | 12.10 |
| 3 | 10 | 105 | 25 | 4.2 | 13.27 |
| 4 | 12 | 110 | 25 | 4.4 | 12.15 |
| 5 | 14 | 120 | 25 | 4.8 | 9.50 |

TABLE 4 COMPRESSIVE STRENGTHS OF DIFFERENT BRIQUETTES PREPARED WITH SAMPLE 3

| S.No | Briquette Weight (gms) | Sample Length L (mm) | Sample Dia D (mm) | L/D | Compressive Strength (N/mm²) |
|---|---|---|---|---|---|
| 1 | 6 | 80 | 25 | 3.2 | 8.9 |
| 2 | 8 | 95 | 25 | 3.8 | 9.5 |

| 3 | 10 | 105 | 25 | 4.2 | 10.28 |
| 4 | 12 | 110 | 25 | 4.4 | 9.7 |
| 5 | 14 | 120 | 25 | 4.8 | 9.0 |

TABLE 5 COMPRESSIVE STRENGTHS OF DIFFERENT BRIQUETTES PREPARED WITH SAMPLE 4

| S.No | Briquette Weight (gms) | Sample Length L (mm) | Sample Dia D (mm) | L/D | Compressive Strength (N/mm²) |
|---|---|---|---|---|---|
| 1 | 6 | 80 | 25 | 3.2 | 8.7 |
| 2 | 8 | 95 | 25 | 3.8 | 9.2 |
| 3 | 10 | 105 | 25 | 4.2 | 10.12 |
| 4 | 12 | 110 | 25 | 4.4 | 9.4 |
| 5 | 14 | 120 | 25 | 4.8 | 8.5 |

TABLE 6 COMPRESSIVE STRENGTHS OF ABOVE SAMPLES – 5 TO 9

| Sample No | Briquette Weight (gms) | Iron ore% | Sample Length L (mm) | Sample Dia D (mm) | L/D | Compressive Strength (N/mm²) |
|---|---|---|---|---|---|---|
| 5 | 10 | 2 | 105 | 25 | 4.2 | 8.69 |
| 6 | 10 | 4 | 105 | 25 | 4.2 | 9.12 |
| 7 | 10 | 6 | 105 | 25 | 4.2 | 9.76 |
| 8 | 10 | 8 | 105 | 25 | 4.2 | 9.66 |
| 9 | 10 | 10 | 105 | 25 | 4.2 | 8.87 |

TABLE 7 COMPRESSIVE STRENGTHS OF ABOVE SAMPLES – 10 TO 14

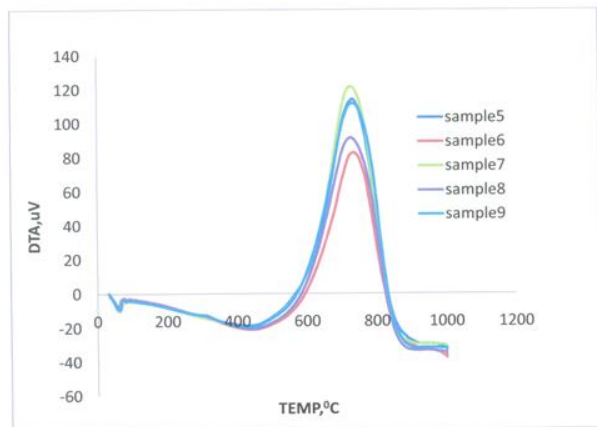| Sample No | Briquette Weight (gms) | Starch % | Sample Length L (mm) | Sample Dia D (mm) | L/D | Compressive Strength (N/mm²) |
|---|---|---|---|---|---|---|
| 10 | 10 | .25 | 105 | 25 | 4.2 | 8.21 |
| 11 | 10 | .5 | 105 | 25 | 4.2 | 8.11 |
| 12 | 10 | 1 | 105 | 25 | 4.2 | 9.86 |
| 13 | 10 | 1.5 | 105 | 25 | 4.2 | 9.66 |
| 14 | 10 | 1.75 | 105 | 25 | 4.2 | 9.56 |



Figure 1: DTA of different compositions of coke oven sludge briquettes samples 5 to 9 with varying iron ore fines composition.
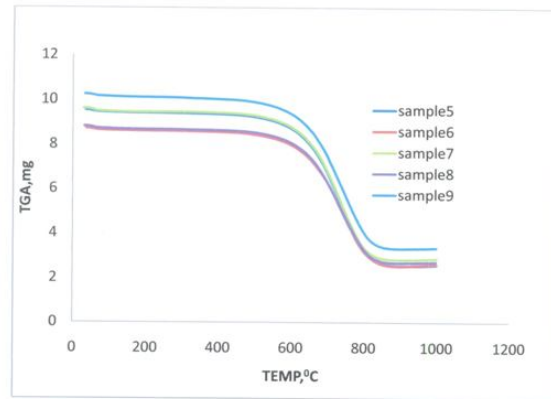


Figure 2: TGA of different compositions of coke oven sludge briquettes samples 5 to 9 with varying iron ore fines composition.

TABLE 8 DTA/TGA OF SAMPLES 5 TO 9

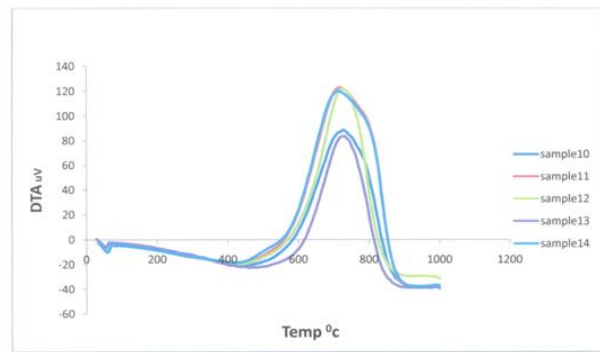| S. No. | Property | Wt.% of Iron ore | | | | |
|---|---|---|---|---|---|---|
| | | 2 | 4 | 6 | 8 | 10 |
| 1 | Heat(KJ/g) | 9.92 | 8.51 | 10.17 | 9.59 | 9.64 |
| 2 | Wt. Loss (%) | 71.32 | 68.87 | 68.49 | 67.38 | 65.65 |



Figure 3: DTA of different compositions of coke oven sludge briquettes samples 10 to 14 with varying starch composition.
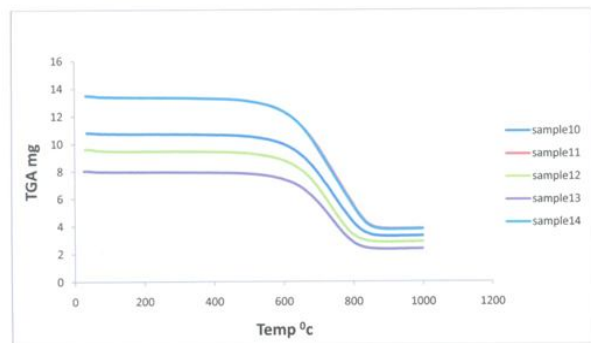


Figure 4: TGA of different compositions of coke oven sludge briquettes samples 10 to 14 with varying starch composition.

TABLE 9  DTA/TGA OF SAMPLES 10 TO 14

| S. No. | Property | Wt.% of Starch | | | | |
|--------|----------|------|------|------|------|------|
| | | 0.25 | 0.5 | 0.75 | 1.0 | 1.25 |
| 1 | Heat(KJ/g) | 8.47 | 9.67 | 8.89 | 9.59 | 10.17 |
| 2 | Wt. Loss (%) | 68.21 | 70.10 | 69.55 | 70.5 | 68.49 |

## V. CONCLUSIONS

Based on the studies carried out briquetting of coke oven sludge with different binders, the following conclusions are drawn

As L/D ratio increased the compressive strength of the Briquettes increased, but with further increase in L/D ratio it decreased. The compressive strength at L/D =4.2 was found to be maximum.

Maximum compressive strength of the Briquettes 14.68 N/mm 2  was observed for L/D ratio of 4.2 for sample 2

In case of sample 3 & sample 4 as the sample L/D ratio increases from3.2 to 4.2(L/D) the compressive strength of the briquettes increased but with further increase in L/D ratio of the samples collapsed.

As the coke oven sludge percentage increased from 95.5% to 97.5% (Table 1) good compressive strength was obtained, however 95.5% coke oven sludge(sample2) showed the best compressive strength.

There was no much change in compressive strength of sample compositions sample 5 to 9 and sample 10 to 14 with change in L/D ratio as shown in Table 4.6. The average compressive strength was 7.80 and 8.92 for 5to 9 and 10 to 14 sample compositions respectively.

DTA curves (fig.1) of Briquettes with sample compositions 5 to 9, show that all are exothermic in nature. At 700°c the peaks were maximum and showed an average latent heat of 9.56 kJ/g. Maximum latent heat was observed for sample 7.

TGA curves(fig.2) shows an average weight loss percentage of 68.34 and weight loss was observed between 6000c to 8000c.

DTA curves (fig.3) of Briquettes with sample compositions 10-14, show that all are exothermic in nature. At 700°c the peaks were maximum and showed an average latent heat of 9.35 kJ/g. The TGA curves(fig.4) shows an average wt. loss percentage of 69.37 and wt. loss was observed between 600°c to 800°c

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