1. INTRODUCTION

About 6.3 million tonnes of bauxite is being processed at National Aluminium Company Limited (NALCO), Damanjodi, Orissa, India, to produce around 1.57 million tonnes of alumina per year. Owing to mechanized mining, selective mining of high-grade bauxite is not possible. Hence, to mine 6.3 million tonnes of bauxite per year, an equal amount (6.3 million tonnes) of mining waste is being generated and dumped at the mine site. The mining activities and waste dumping at mine site is shown in Fig. 1. During production of 1 ton of alumina, 10 tons mining / metallurgical waste is generated which is shown in Fig. 2. At times, these are also used for backfilling of abandoned mines. Thus any attempt to utilize the huge waste rock for industrial application is a challenging problem.

It is an important to note that most of these mining waste materials are PLK rock containing bauxite with kaolinite in varying proportions of colouring impurity minerals such as hematite, goethite and rutile. The PLK rock contains about 45–56% Al₂O₃, 0.3–30% Fe₂O₃, 0.97–3%TiO₂, 20–30% SiO₂ and 20–30% LOI. In spite, these PLK rock contain metallurgical grade Al₂O₃

Abstract: Partially Lateritised Khondalite (PLK) rocks are the waste materials generated as a result of mining of bauxite. The major discolouring elemental impurity in the PLK is iron oxides, which render it unsuitable for its use as a refractory material. The iron can be removed by suitable beneficiation methods. The main aim of this present investigation is to prepare a value added material from the mining waste by preparation of PLK rock containing less than 2% Fe₂O₃ which finds application in the refractory industries and then preparation of brick suitable for industrial applications. The feed sample containing 4.31 % Fe₂O₃ subjected to hydrocyclone for refractory of low iron content product in the underflow. The results of these studies reveal that the hydrocyclone underflow sample contains 1.9% Fe₂O₃ is suitable for making bricks due to presence of low iron content. However, bricks are made from a feed sample as well as from hydrocyclone underflow and overflow products. Physical, chemical and thermo-mechanical properties of these bricks are evaluated. Mineralogical properties of these bricks are also correlated with the thermo-mechanical properties. The developed bricks are compared with the standards for their suitability in industrial applications. Hence the bauxite mining waste can be a value added material but not a waste material which at present creates environmental pollution at the mining site.

Key words: PLK Rocks; Underflow; Hydrocyclone; Beneficiation; Brick; Refractories
content, are not suitable to use in metallurgical industry, due to presence of high amount of reactive silica (kaolinite – Al₂SiO₄). There are two main form of silica which occurs in bauxite (i) reactive silica mainly as kaolinite (Al₂O₃·2SiO₂·2H₂O) and (ii) quartz (SiO₂) (Cormick et al., 2002). Thus, this material can be used as an alternative materials for refractory, ceramic and filler industries. After suitable beneficiation techniques of this material, it can be a potential resource to recover refractory, ceramic and filler grade materials for industrial applications (Rao et al., 2007). Some of the literature is collected on refractory purpose of bauxite, but not exactly about the PLK rocks.

Bhatacharya and Mookherjee (Bhatacharya and Mookherjee, 1987) discussed about the beneficiation of bauxite. They also discussed about the different grades of bauxite of India. Refractory grade bauxite contain Al₂O₃% > 56-58%, SiO₂% < 5% and Fe₂O₃% < 3%. Scheider et al. (Scheider et al., 1987) studied about the refractory grade bauxites and their firing at different conditions. They took two different samples CR1 and CR2. Here, CR1 contain Fe₂O₃% is 1.47% and TiO₂ 3.48%, whereas CR₂ contain Fe₂O₃% is 1.75% and TiO₂ 3.36%. After firing in air and gases, CR1 shows the microstructure like corundum, mullite, tiellite and glass. Their analyses also reported in their paper. Indian bauxite ores can be used in refractory industries reported by Rao & Ansari (Rao & Ansari, 1992). Sadler and Venkataraman (Sadler and Venkataraman, 1991) described the process to remove iron from bauxite containing 2.7% Fe₂O₃. By using calcinations followed by magnetic separation, a product obtained from Alabama bauxite containing 1.5% Fe₂O₃. Nivedita et al (Nivedita et al., 1996) discussed about the removal of iron from bauxite containing 5.6% Fe₂O₃ by leaching study to make the product suitable for the refractory industry. Besra etal. (Besra et al., 1996) studied about the calcareous bauxite for refractory use after flotation studied. They studied about the low grade bauxite ore containing 4.5% iron and 2.7% calcium which renders the refractory use. They suggested two stage process for removing both iron and calcium by calcinations followed by magnetic separation and flotation. It was possible to obtain a product containing about 2.7% iron and 1.5% calcium, which was suitable for refractory use. Rao et al. (Rao et al., 1997) used the magnetic separator to reduce the iron minerals so that it is suitable for refractory application. Banerjee et al. (Banerjee et al., 2000) used the gaseous reduction process followed by magnetic separation to reduce the iron content from the bauxite ore from Jamanagar, which can be used for the refractory purpose. Central Pollution Control Board (CPCB, 2007) studied that the desirable Indian bauxite range for Fe₂O₃% and TiO₂% are varies from 2-4% and 2-4% respectively for refractory purpose.
They also wrote that Fe$_2$O$_3$% and TiO$_2$% are the detrimental composition present in bauxite which can be utilized for the refractory purposes. In this paper an attempt has made for making bricks from different samples of PLK rocks. This developed brick properties are compared with the standard brick properties for its suitability in industrial applications.

2. MATERIALS AND METHODS

2.1. Raw Material

PLK rock sample was collected from Panchpatmali plateau of NALCO, Damanjodi, Orissa. Two representative sample (feed as Sample 1) was prepared by stage crushing to below 1mm size. One of the representative samples was subjected to hydrocyclone to separate superfine from the PLK rock sample. The bricks were prepared from the samples such as feed sample (Sample 1), hydrocyclone underflow sample (Sample 2) and hydrocyclone overflow sample (Sample 3). The experimental plan for the preparation of the materials for the refractory purpose is shown in Fig. 3.

2.3. Hydrocyclone studies

Hydrocyclone studies were carried out using Mozley hydrocyclone unit at different vortex finder and spigot diameter at a constant pressure (0.5 bar). The particle size analysis of overflow and underflow were determined by using standard sieves and as well as by using particle size analyzer (Cilas 1064). The iron content of overflow and underflow was also determined by wet chemical method.

Conditions are given below:

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyclone size</td>
<td>mm</td>
<td>50</td>
</tr>
<tr>
<td>Vortex finder diameter</td>
<td>mm</td>
<td>14.3, 11.1, 8.1</td>
</tr>
<tr>
<td>Spigot diameter</td>
<td>mm</td>
<td>8.0, 6.3, 4.7</td>
</tr>
</tbody>
</table>

These are the d$_{75}$ and d$_{25}$ sizes, respectively. The efficiency of separation, or the so-called imperfection $I$, is then given by

$$I = \frac{d_{75} - d_{25}}{2}$$

2.3. Development of Bricks

Total six numbers of clot compositions were made, each of two clots compositions were prepared from feed sample (Sample 1), hydrocyclone underflow sample (Sample 2) and hydrocyclone overflow sample (Sample 3). One of the clot compositions was made by taking 100% raw material. Two percentages of dextrin and one percentage of molasses were added to the raw material. The moisture content was 2.7%, whereas another clot was made from 70% raw material and 30% SRM 30. Two percentages of dextrin and one percentage of molasses were added to the clot composition. Similarly another four clot compositions were made from hydrocyclone overflow and underflow samples, two numbers of compositions from each materials. The detail study is shown in Fig. 4 and also shown in Table 1 for raw materials to prepare the clots.

All these compositions mixed in a laboratory counter current mixer for homogeneity with the addition of some water for about 20 minutes. The mixers were formulated into clot shape in high capacity hydraulic press with a specific pressure.
of 400kg/cm² pressure at FSP400T/12 pressing center. Care was taken during pressing to avoid cracks and lamination. Six numbers of clots were made from feed sample (Sample 1), hydrocyclone underflow sample (Sample 2) and hydrocyclone overflow sample (Sample 3).

All the clots were dried in an industrial drier at a temperature of 100-120°C for 24 hours to drive out moisture which may lead to explosion during firing. After drying all the clots were checked for physical defects and loaded in top bench of the kiln car giving equidistance gap for ensuring uniform firing. The size analyses of the clots are shown in Table 2. All the clots were fired at temperatures of 1580°C and 1630°C for 6 hours in industrial tunnel kilns with a predetermined scheduled firing rate.

The clots were taken for the preparation of brick composition as shown in Table 3. Sillimanite fine, ball clay, molasses and dextrin were added to the clots mixed in a laboratory counter current mixer for homogeneity with water for about 20 minutes. The mixtures were formulated into brick shape in high capacity hydraulic press with a specific pressure of 400kg/cm² pressure at FSP 400T pressing center. Sufficient care was taken during pressing to avoid cracks and lamination. Total six numbers of bricks were made. All the bricks were dried in an industrial drier at a temperature of 100-120°C for 24 hours to drive out moisture which may lead to explosion during firing. After drying all the
bricks were checked for physical defects and loaded in top bench of the kiln car giving equidistance gap for ensuring uniform firing. All the bricks of different compositions were fired at a peak temperature of 1580°C for 6 hours in industrial tunnel kilns with a predetermined scheduled firing rate. No special gas used for specific reaction. Experiments were carried out in the ambient atmosphere.

Sieve analysis and moisture content were determined according to Bureau of Indian Standard. Green size and bulk density were also determined according to of Indian Standard (BIS). Physical properties such as apparent porosity, bulk density, cold crushing strength and modulus of rupture were determined according to BIS standard. Thermo-mechanical properties such as refractories under load was determined according to BIS. Spalling test was carried out following DIN 51068 standard. The mineralogical phase analysis of the feed, overflow and underflow samples was carried out using PANalytical X-Pert X-ray powder diffractometer with Mo-K\(\alpha\) radiation (\(\lambda =0.709\text{\ Å}\) ) from 6° to 40° scanning angle at a scanning rate of 0.02°/sec. Phase analysis was done in X-ray diffractometer supplied by Philips using copper target and nickel filter for the developed bricks. Micro structural analysis was done under reflected light in a Leitz universal microscope with image analyzer.

### 3. RESULTS AND DISCUSSION

Chemical analysis of jaw crushed and roll crushed product of Partially Laterised Khondalite rock (ROM) sample is shown in Table 4. The data indicate that the coarse size fractions contain less iron content whereas the fine fractions contain more of iron. Chemical analysis of feed sample (Sample 1), hydrocyclone underflow sample (Sample 2) and hydrocyclone overflow sample (Sample 3) are given in Table 5. The analysis of
the samples given in Table 5, indicate that the feed material contains 4.31 % of Fe$_2$O$_3$, whereas overflow sample contain Fe$_2$O$_3$ 5.39% and underflow sample contain 1.87% Fe$_2$O$_3$. Underflow sample is suitable for making of bricks as iron content is low.

XRD of the sample1, sample 2 and sample 3 is shown in Fig. 5. Kaolinite, gibbsite, goethite and quartz phases are found in XRD of feed. Kaolinite, gibbsite and goethite show major phase and minor phases are magnetite, hematite, quartz and orthoclase in overflow sample. Kaolinite and quartz are the major phases and hematite and goethite are minor phases present in underflow sample.

PLK sample is stage crushed and ground to below one mm. size. This product has been subjected to hydrocyclone at different operating
conditions to recover fines (below 45 µm). The results of these studies are shown in Table 6. The data indicate that with increase in the spigot diameter, the production of the fines is increasing from 4.7 to 8.0 mm vortex finder. This observation is same for other vortex finders. The production of fines is also increasing with increase in the vortex finder. Hence, in order to increase in the feed capacity of the hydrocyclone, the vortex finder 11.1 mm diameter and the spigot diameter 8.0 mm has been considered as the best optimum condition for recovery of fines from PLK sample. The overflow sample contains 5.39% $\text{Fe}_2\text{O}_3$ and underflow sample contains 1.87% $\text{Fe}_2\text{O}_3$. The commonest method of representing hydrocyclone efficiency is by a performance or partition curve is given in Fig. 6, which relates the weight fraction, or percentage, of each particle size in the feed which reports to the apex, or underflow, to the particle size. The data indicates that the cut point or separation size is 25 µm. The cut point, or separation size, of the cyclone is defined as the size for which 50% of the particles in the feed report to the underflow, i.e. particles of this size have an equal chance of going either with the overflow or underflow. The efficiency of separation, or the so-called imperfection I, is then given by 6 micron.

$$\text{Efficiency of separation} = I = \frac{31-19}{2} = 6$$

4. EVALUATION OF CLOT

The clot which is made from feed has $\text{Al}_2\text{O}_3$ content 37.08%, $\text{TiO}_2$ is 1.35% where as $\text{Fe}_2\text{O}_3$ percentage is 4.31%. $\text{SiO}_2$ content is 40.81% and the other constituents are very less. Mineralogical analysis by XRD shows major phases like Mullite and Cristobalite. Corundum phase is identified in the clot which made from raw material with conditions to recover fines (below 45 µm). The results of these studies are shown in Table 6. The data indicate that with increase in the spigot diameter, the production of the fines is increasing from 4.7 to 8.0 mm vortex finder. This observation is same for other vortex finders. The production of fines is also increasing with increase in the vortex finder. Hence, in order to increase in the feed capacity of the hydrocyclone, the vortex finder 11.1 mm diameter and the spigot diameter 8.0 mm has been considered as the best optimum condition for recovery of fines from PLK sample. The overflow sample contains 5.39% $\text{Fe}_2\text{O}_3$ and underflow sample contains 1.87% $\text{Fe}_2\text{O}_3$. The commonest method of representing hydrocyclone efficiency is by a performance or partition curve is given in Fig. 6, which relates the weight fraction, or percentage, of each particle size in the feed which reports to the apex, or underflow, to the particle size. The data indicates that the cut point or separation size is 25 µm. The cut point, or separation size, of the cyclone is defined as the size for which 50% of the particles in the feed report to the underflow, i.e. particles of this size have an equal chance of going either with the overflow or underflow. The efficiency of separation, or the so-called imperfection I, is then given by 6 micron.

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<table>
<thead>
<tr>
<th>Details</th>
<th>Physical properties of the samples</th>
<th>XRD phases</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Bulk density, g/cc</td>
<td>True porosity, %</td>
</tr>
<tr>
<td>Clot 1 (a)</td>
<td>2.57</td>
<td>11.38</td>
</tr>
<tr>
<td>Clot 1 (b)</td>
<td>2.47</td>
<td>14.83</td>
</tr>
<tr>
<td>Clot 2 (a)</td>
<td>2.23</td>
<td>28.53</td>
</tr>
<tr>
<td>Clot 2 (b)</td>
<td>2.38</td>
<td>23.47</td>
</tr>
<tr>
<td>Clot 3 (a)</td>
<td>2.52</td>
<td>13.99</td>
</tr>
<tr>
<td>Clot 3 (b)</td>
<td>2.51</td>
<td>12.85</td>
</tr>
<tr>
<td>Clot 4 (a)</td>
<td>2.35</td>
<td>24.84</td>
</tr>
<tr>
<td>Clot 4 (b)</td>
<td>2.35</td>
<td>23.45</td>
</tr>
<tr>
<td>Clot 5 (a)</td>
<td>2.68</td>
<td>8.53</td>
</tr>
<tr>
<td>Clot 5 (b)</td>
<td>2.48</td>
<td>14.48</td>
</tr>
<tr>
<td>Clot 6 (a)</td>
<td>2.4</td>
<td>22.3</td>
</tr>
<tr>
<td>Clot 6 (b)</td>
<td>2.52</td>
<td>18.7</td>
</tr>
</tbody>
</table>
addition of SRM 30. The data are shown in Table 7.

Similarly the clot which is made from hydrocyclone under flow has Al₂O₃ content 37.08% and the SiO₂ is less i.e. 41.89%. TiO₂ is 2.39% where as Fe₂O₃ percentage is 1.91%. XRD analysis shows complete conversion to Mullite (3Al₂O₃.2SiO₂) and Cristobalite (SiO₂), which are the general crystalline compounds present in this type of materials. In this material also corundum phase is found in the clot which made from raw material with addition of SRM 30.

The clot which is made from hydrocyclone over flow has Al₂O₃ content 37.09% and the SiO₂ is less i.e. 40.32%. TiO₂ is 0.97% where as Fe₂O₃ percentage is 5.39%. XRD analysis shows complete conversion to Mullite and Cristobalite. Here corundum is found as minor phase in both the clots.

5. EVALUATION OF BRICK

Bricks were made by taking these clots. The brick which was marked 1 has been made from clot 1 fired at different temperatures like 1580°C and 1630°C. The feed can be used as such for brick making. However, high value added materials such as filler materials can also be prepared from the feed (PLK rocks) sample. In view of this, recovery of fine materials using hydrocyclone has been attempted for using filler industries. The underflow sample has prepared for bricks which is more suitable for refractory application. The hydrocyclone overflow sample has also been taken for the preparation of brick. But this brick shows the surface coated with iron spots and cracks on the surface of the brick due to

<table>
<thead>
<tr>
<th>Properties</th>
<th>Feed sample</th>
<th>Underflow sample</th>
<th>Overflow sample</th>
<th>35% Al₂O₃ Standard Brick</th>
</tr>
</thead>
<tbody>
<tr>
<td>Green size, mm</td>
<td>251x128x5</td>
<td>231.5x117x</td>
<td>251X128X</td>
<td>231.5x117x</td>
</tr>
<tr>
<td></td>
<td>1.5</td>
<td>74</td>
<td>51</td>
<td>51</td>
</tr>
<tr>
<td>Green B.D., gm/cc</td>
<td>2.29</td>
<td>2.17</td>
<td>2.43</td>
<td>2.35</td>
</tr>
<tr>
<td>Apparent porosity, %</td>
<td>23.3</td>
<td>30.9</td>
<td>10.1</td>
<td>20.7</td>
</tr>
<tr>
<td>Bulk density, gm/cc</td>
<td>2.19</td>
<td>2.07</td>
<td>2.4</td>
<td>2.35</td>
</tr>
<tr>
<td>Apparent specific gravity</td>
<td>2.77</td>
<td>2.998</td>
<td>2.608</td>
<td>2.961</td>
</tr>
<tr>
<td>Cold crushing strength, kg/cm²</td>
<td>456</td>
<td>394</td>
<td>858</td>
<td>645</td>
</tr>
<tr>
<td>Spalling (Air quenching), Cycles</td>
<td>15+</td>
<td>15+</td>
<td>15+</td>
<td>15+</td>
</tr>
<tr>
<td>Refractoriness under load Ta, °C</td>
<td>1430</td>
<td>1450</td>
<td>1420</td>
<td>1450</td>
</tr>
</tbody>
</table>

Table 8. Physical, chemical and thermo-mechanical properties of fired bricks
high iron content. The photograph of bricks are shown in Fig. 7, where it is found the good structured bricks are made from underflow sample rather than from overflow sample.

The properties of these bricks are compared with standard 35% $\text{Al}_2\text{O}_3$ refractory standard brick available in the market which is given in Table 8. This table shows the different properties related to physical, pyro-physical, thermo mechanical properties. The apparent porosity is within range for the bricks made from underflow

![Brick from Feed Material](image1)
![Bricks from underflow sample](image2)
![Brick from overflow sample](image3)

Fig. 7. Brick prepared from (a) feed sample (b) underflow sample and (c) overflow sample of PLK rocks

![XRD of bricks made from feed samples](image4)

Fig. 8. (a) XRD of the bricks made from feed samples

![XRD of bricks made from underflow samples](image5)

Fig. 8. (b) XRD of the bricks made from underflow samples

![XRD of bricks made from overflow samples](image6)

Fig. 8. (c) XRD of the bricks made from overflow samples
sample, whereas for brick made from feed sample and overflow sample show more slightly than standard value. The bulk density is almost matching for all the bricks. Cold Crushing Strength (CCS) value is more or less compared to the standard brick. Refractoriness under load (RUL) value is more than the standard value for underflow sample, whereas for other two samples it is slightly less than the standard value. Table 9 shows chemical and mineralogical properties of

![Fig. 9 (a) Microscopic studies of the bricks made from feed samples](image1)

![Fig. 9 (b) Microscopic studies of the bricks made from underflow samples](image2)

![Fig. 9 (c) Microscopic studies of the bricks made from overflow samples](image3)
developed products and the standard marketed product.

The Fe₂O₃% for brick from overflow sample is 4.64% which is much more than the standard value and also Fe₂O₃% for brick from feed sample is also slightly more than the standard value. But the brick from underflow sample is less than the standard value given in Table 9.

XRD graphs of all three bricks made from feed, overflow and underflow samples are given in Fig. 8. The XRD of brick 1 and brick 2 made from feed sample show mullite as major-phase and cristobalite as minor-phase. Mullite and Cristobalite are the major phases found in the bricks. XRD of brick 3 and brick 4 made from underflow sample show mullite as major phase and cristoballite as minor phase. The same phases are also seen in bricks made from overflow samples.

Fig. 9 shows the micro-photograph of the refractory brick made from feed, hydrocyclone overflow and underflow materials. The photomicrograph shows three different phases, which are found in all the thin section specimens.

Mullite - Needle shaped incipient mullite crystals are found in all fractions and in the matrix part of the sample. This needle shaped is shown in Fig. 9 (a) with an arrow mark. Those regions in which feldspar particles are well mixed with kaolinitic clay or where feldspar has gone through clay agglomerates form elongated needle-shaped crystals termed secondary mullite (since they form later in the firing process) (Martin Marquez, 2010). In the matrix part the mullite crystals are little bigger but less in quantity as compare to that of the coarse and middling fractions.

Glass - The alumino-silicate glass is present both in matrix as well as in the coarse and middling fractions in a huge quantity with other impure silicate phase. In the matrix part the glassy phase bridges the coarse and middling fractions throughout the sample.

Cristobalite- Cristobalite is present in circular patches, mostly in the coarse fraction of the specimen and with glassy phases also.

The specimen shows a compact microstructure
where both interconnected and isolated pores are present randomly throughout the sections. However, the glassy phase predominant matrix shows a continuous bridging between the coarse and middling fraction that impart the compactness to the specimen.

From the above analysis report of bricks, it is observed that the chemical composition, RUL and cold crushing strength (CCS) are good. However, the physical properties such as apparent porosity (AP) and bulk density (BD) are almost matching for the bricks developed from underflow sample. But the spalling resistance properties of the bricks are very low. This is due to improper manufacturing process. The bricks made from the overflow sample may not be suitable as it contains more iron and due to this iron content the spotting are found on the brick surface and also shape of the brick shows the unsuitability which can be clearly seen in Fig.5. But this very fine overflow sample can be utilized in filler industries after suitable beneficiation and chemical treatment. But as per the brick standard the brick from hydrocyclone underflow sample is suitable for the refractory industry. This mining waste (PLK rock) can be utilized as chimney flue, boiler, cupola and steel industry as per the marketable standard of 35% Al₂O₃ grade.

6. CONCLUSIONS

1. Owing to mechanized mining, selective mining of high-grade bauxite is not possible and hence which causes 6.3 million tons of mining waste at the mine site per annum.
2. The size analyses of the jaw and roll crushed products show that the fine materials contain more iron content.
3. The vortex finder 11.1 mm diameter and the spigot diameter 8.0 mm of the hydrocyclone has been considered as the best optimum condition for recovery of fines from PLK sample. The overflow sample contains 5.39% Fe₂O₃ and underflow sample contains 1.87% Fe₂O₃.
4. The properties of the bricks are compared with standard 35% Al₂O₃ refractory brick available in the market.
5. XRD analysis shows complete conversion to Mullite (3Al₂O₃·2SiO₂) and Cristobalite (SiO₂), which are the general crystalline compounds present in this type of materials. The photomicrograph shows three different phases are found all across the section of the specimen such as mullite, glass and cristobalite.

7. ACKNOWLEDGEMENTS

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