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A novel process for making radiopaque materials using bauxite—Red mud

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Abstract

Red mud, which is an aluminum industry waste, has been utilized[1] for making X-ray radiation-shielding materials. A novel method for making radiation shielding materials utilizing red mud and barium compound has been developed by ceramic processing route using phosphate bonding. The red mud based shielding materials (RMSM) are characterized for their X-ray attenuation characteristics. The shielding, i.e. half value thickness (HVT) for different energies of X-ray photons for RMSM have been computed and compared with conventionally used shielding materials namely concrete and lead, it is found that the (HVT) of the red mud based shielding materials, in comparison to concrete, is significantly very less for the various energies of X-ray photons.

The X-ray powder diffraction studies confirmed the presence of celsian, bafertiste and iron titanium oxide as the major shielding phases in the RMSM. Scanning electron microphotographs have revealed the compacted and continued integrated morphological characteristics of the various shielding phases in the matrix of shielding materials. The mechanical properties namely compressive strength and impact strength evaluation test showed that RMSM meets the standard specifications laid down for radiation shielding concrete and ceramic tiles. Based on the above studies, it is found that RMSM, can preferably be used for the construction of X-ray diagnostic and CT scanner room to provide adequate shielding against X-ray photons.

Keyword: Red mud

1. Introduction

Red mud is the waste generated during aluminum production from bauxite.[1–3] It is reported that production of 1 tonne of metallic aluminum generates about 2 tonnes of red mud.[3] At all the world's 85 alumina plants, 1.0–1.6 tonnes of red mud is generated per tonne of alumina and it is estimated that over 66 million tonnes of this waste is impounded annually in the world. The disposal of such a large quantity of this alkaline waste sludge is expensive (up to 1–2% of the alumina price), as it requires a lot of land (approximately 1 km^2 per 5 years for a 1 Mtpy alumina plant) and causes a number of environmental problems.[4] The disposal of red mud tailing costs the industry US$ 3 per tonnes of alumina production.[5]

The enormous quantity of red mud generated every year posses a very serious and alarming environmental problem.[6] To solve the disposal problem, voluminous research and development work for the utilization of red mud has been carried out all over the world, but to date very few techno-economical solutions have been found out.[1–3,6] Studies on the use of red mud for making inexpensive and efficient adsorbent for removal of cadmium, zinc and arsenic,[7,8] fluoride,[9] lead, chromium[10] from aqueous solutions has also been reported recently. So far, the various uses of red mud developed includes, as acidic amender,[3] in making building materials namely bricks,[11] ceramics, tiles,[12] glazes[13] and red mud–polymer composites panels as wood substitute,[14] iron rich cement,[15] etc. Fundamental studies carried out for the extraction of iron oxide or titanium oxide are reported to be economically unsustainable[16] and therefore red mud as such has been used for various applications. Red mud has also been used for catalytic hydro-de chlorination of tetrachloroethylene[17] for the treatment of gold ores,[18] in making silicate bonded unsintered ceramics,[19] heavy clay products,[20] sintered ceramics,[21] etc. In view of above, there is a great scope to evolve innovative strategy and to develop novel functional applica-

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[1] Patent applications for the developed process have already been flied in USA as well as in India vide Application no .US—11/026115/05 and 1888/Del/04, respectively.

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tions of red mud based materials, for effective utilization of red mud.

The application of radiation technology in medicine, agriculture, nuclear reactor and other industries is increasing day by day all over the world. However, the use of radiation is invariably associated with very well established harmful effects of X-ray radiation, such as destroying the tissue of animates bodies and white blood cells, i.e. W.B.C. Conventionally, the shielding materials based on lead and lead containing compositions are well known and widely used but such shields are characterized by high toxicity in production and recycling and therefore poses environmental pollution problem.[22,23] Lead is ranked number one on the U.S. EPA's Top 20 Hazardous Substance Priority list.[24] The increased awareness has recently led to extend the application of titanium for use as future non-toxic shielding material, instead of lead because titanium is not a hazardous material.[25] The non-toxic metals which could play a role in shielding X-ray radiation are among[26] the titanium, iron and aluminum. Apart from lead based compounds, concrete is often used as a structural and shielding material,[27–29] but its use has two major drawbacks: (i) the requirement of very large thickness of concrete blocks, for obtaining effective shielding and (ii) very poor resistance of concrete to radiation, resulting in losses in mechanical strength due to radiolysis of water of hydration in the concrete.[28] However, these, shortcoming of concrete has led to the development of various shielding materials[30] and concrete.[31] Shielding concrete is made from special aggregates for use as radiation shielding and ordinary Portland cement. These special aggregates are among the barite (barium sulphate), peridotite (iron magnesium silicate), pyrite (iron sulphide), magnetite (ferrous and ferric iron oxide),[32] hollandite (barium aluminate titanate)[33,34] and barium aluminate.[34] It is reported[35] that the barium enriched cement shows adequate shielding to gamma radiation in comparison with lead in addition to possessing very good compressive strength and abrasion resistant. Similarly, the barium based materials, such as barium silicate and barium aluminum silicates have been used as, radiopaque fillers for making dental composite resin, because they shield X-ray radiation.[36,37] The use of ceramic, glass–ceramics and synroc based radiation shielding materials is gaining increased research interest for shielding and encapsulation of surplus radioactive waste from nuclear power plant and defense sources. These materials are based on one or more of the compositions based on barium aluminosilicate, phosphates of iron, titanium, calcium, magnesium, etc.[38,39] Further a critical insight of chemical compositions of all these phases of special aggregates, responsible for imparting shielding property to these shielding concrete, mainly consist of phases based on four major element, namely iron, titanium, aluminum and barium. Further, the red mud generated in million tonnes, also contains a fairly high quantity[27] of titanium oxide (5–25%), in addition to very high percentage of iron oxide (20–65%) and aluminum oxide (10–27%) and therefore it can be used as useful resource material for making X-ray shielding materials, it was therefore thought worthwhile to explore the possibility of developing red mud based shielding materials, by in situ crystallization of various effective shielding phases, such as barium iron titanium silicate (bafertisite), barium aluminum oxide, iron titanium oxide, barium silicate, barium alumino silicate, etc., utilizing red mud and barium containing waste/compounds.

Based on the above facts and discussion, investigations, on exploring the suitability of red mud for making functional materials, which have X-ray radiation shielding properties, were carried out and the results of the same are presented in this current paper.

2. Materials and methods

2.1. Raw materials and chemicals

The red mud obtained from Hindustan Aluminum Company (HINDALCO) Renukoot, India was used as received, after making a representative sample by the method of coining and quartering. The barium hydroxide and sodium hexameta phosphate, chemicals of GR grade of Merck make India, were used as such without any further purification.

2.2. Preparation of green tile samples and their sintering

The samples of shielding materials in the form of ceramic tiles and cubes were prepared based on our earlier research investigation on sintering characteristics of red mud, fly ash and pyrophyllite.[40–43] The raw material mixtures were prepared by homogenizing red mud with barium hydroxide additions up to 50 wt.% at 10% increments. The mixtures obtained were homogenized with 10 wt.% alkaline phosphatic binder aqueous solution. After homogenization, the raw mix was then compressed in a steel mould at a pressure of 300 kg/cm^2 (using, Digital compression testing machine, model no. AIM 308E-DG of AIMIL Ltd. India make), to obtain samples of tiles of dimension 10 cm × 10 cm × 2.0 cm and samples of cube of dimension 50 cm × 50 cm. The green samples were then dried in an air oven at 110 °C for 1-h duration and then sintered in an electrical muffle furnace. The firing cycle was programmed as follows: heating from ambient temperature to 400 °C at a heating arte of 20 °C per minute, holding for 30 min at 400 °C; heating to 950 °C at rate of 10 °C; holding for 60 min at 950 °C, heating to 1300 °C at a heating rate of 20 °C per minute, holding for 1 h at 1300 °C; and finally cooling of samples in the furnace itself down to ambient temperature.

2.3. Determination of X-ray attenuation characteristics

The determination of X-ray attenuation characteristics of shielding material was carried out in the Standard Safety System Division of Bhabha Atomic Research Center (BARC), Mumbai, India, under following measurement conditions:

(i) Measurements were done with (I) and without filters (I_0) to determine the tenth value thickness (TVT) of the beams.
(ii) Measurements were done at a distance of 60 cm from the surface of the cone to the center of the chamber (i.e. at a distance of 100 cm from the X-ray focal spot).

(iii) Dose rate meter UNFORS Instrument (Sweden) type 9001, sr. no. 12394.
(iv) Measurements were done in charge mode and leakages were noted and corrected for.
(v) Temperature and pressure were measured using calibrated thermometer and barometer and the readings were corrected for the same. The measurement uncertainty may be ± within 5%. For evaluating X-ray attenuation characteristics the shielding materials samples were made in the form of circular disc of 25 mm diameter and 9, 10, 12 and 17 mm thickness.

2.3.1. Determination of the mechanical properties of the shielding materials

The bulk density determination has been performed as per the standard procedure prescribed for ceramics.[44] The sintered tiles samples of size 10 cm × 10 cm × 2.0 cm were evaluated for their impact strength following the procedure laid down in the specifications drawn for ceramic tiles.[45]

The procedure for measuring impact strength involved use of failing weight type instrument. The impact strength was carried out by placing the bottom surface of the tile on a 60 mm equilateral triangular support. A steel ball of 30 g weight was allowed to drop on the top surface of the tile sample from an initial height of 25 cm. The height of the free fall of the steel ball was increased in small increments till failure. Impact strength was calculated as per the formula.

$$\text{impact strength} = \frac{W \times h}{t},$$

where W is the weight of the steel ball in (kg), h the height of free fall of steel ball in (m) and t is the thickness of the tile in (cm).

The compressive strength measurements of the cube of shielding material was performed as per the standard procedure prescribed for the testing of concrete.[45] The measurement were carried out using Digital compressive strength testing machine, model no. AIM 308E-DG of AIMIL Ltd. India make. The dimension of the shielding samples for compressive strength evaluation were of 50 cm × 50 cm size and kept constant for all the composition studied.

2.3.2. Investigation of phases formed in the sintered shielding materials

The investigation of various phases formed in the sintered shielding materials made using optimized processing parameters was carried out using a Philips model 1710 X-ray diffractometer, using Ni filtered Cu Kα radiation.

2.3.3. Morphology of powdered sintered shielding material sample

The morphology of the various phases formed in the shielding tile samples made using optimized processing parameters was studied using a Jeol model JEM-35-CF scanning electron microscope.

3. Results and discussion

3.1. Characterization of red mud

3.1.1. Chemical analysis

The chemical composition of red mud was determined by standard wet chemical analysis method of chemical analysis.[46] The chemical analysis showed (Fig. 1) that the various oxide percent content as follows: Fe_2O_3, 31.88; TiO_2, 21.20; Al_2O_3, 20.10; SiO_2, 8.50; CaO, 2.99; Na_2O, 6.0%. The loss on ignition was found to be 10.0%. The wet sieve analysis of red mud shows that the 78% of the particles are below 48 μm size.

3.2. Phases identification in sintered shielding materials

Identification of the various phases present in the red mud as such and formed during sintering in the shielding material was carried out by comparing the experimental inter planar spacing (*d*-values) with those of the respective likely substances listed in the JCPDS standard X-ray diffraction data files.[47] The XRD patterns obtained are given in Figs. 2 and 3, respectively, and the respective phases identified are given in Tables 1 and 2.

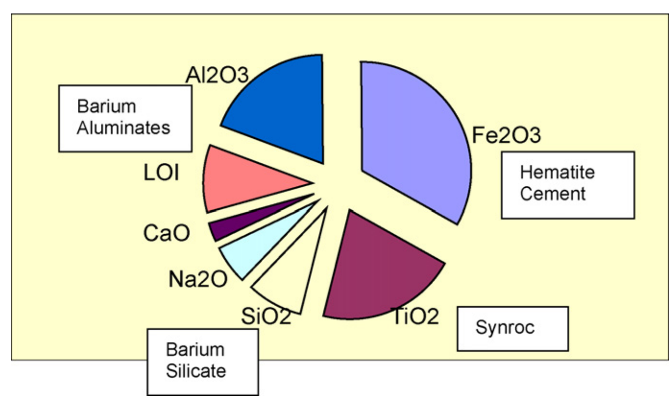


Fig. 1. Chemical analysis of red mud and major prospective shielding constituents.

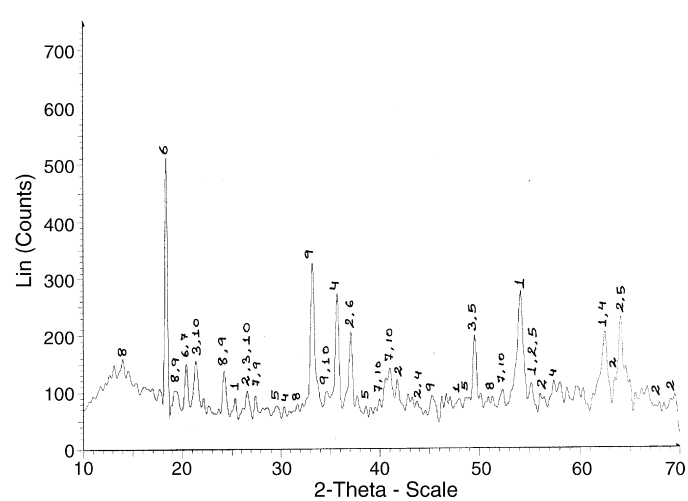


Fig. 2. XRD of red mud.

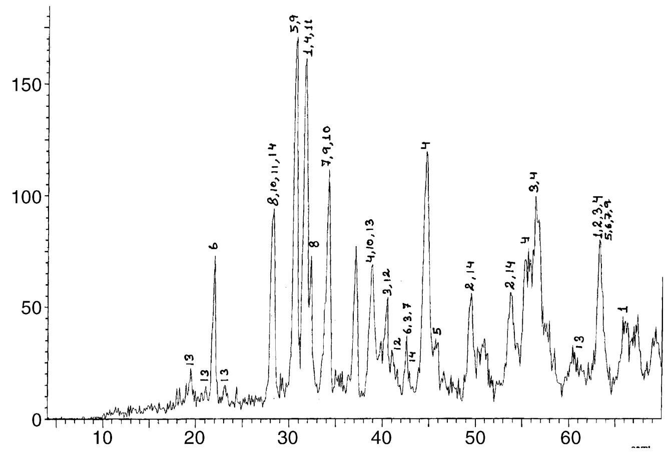


Fig. 3. XRD of red mud based shielding material (RMSM).

Table 1
Phases present in red mud

S.no.	Red mud	PDF	Crystal structure
1	Anatase	21–1272	Tetragonal
2	Rutile	21–1276	Tetragonal
3	Quartz	33–1161	
4	Hematite	33–664	Cubic
5	Bohmite	21–1307	Orthorhombic
6	Gibbsite	29–41	
7	Bayerite	20–11	Monoclinic
8	$Na_5Al_3CSi_3O_{15}$	15–469	Hexagonal
9	Cancrinite	25–776	Hexagonal
10	Chantalite	29–1410	Tetragonal

The results of chemical analysis and X-ray powder diffraction analysis of red mud exhibited the presence of a diverse mineralogical phases and compounds which act as a source materials for obtaining varieties of shielding phases (cf. Fig. 1). The presence of iron oxide helps in obtaining hematite concrete phase, anatase and rutile helps in acquiring shielding property of synroc, i.e. titante ceramics phases, aluminium and silica in getting barium aluminates and barium silicate phases, respectively.

Table 2
Phases present in RMSM

S.no.	RMSM	PDF	Crystal structure
1	Barium iron titanium oxide	26–1032	Hexagonal
2	Iron titanium oxide	13–270	Tetragonal
3	Barium titanium oxide	17–661	–
4	Barium meta titanate	8–372	Hexagonal
5	Barium iron titanium oxide	21–101	Orthorhombic
6	Barium silicate	10–45	–
7	Bafertisite	14–541	Orthorhombic
8	Barium titanium oxide	26–762	Orthorhombic
9	Barium aluminium silicate	28–125	Hexagonal
10	Barium aluminium oxide	28–121	–
11	Iron titanium oxide	9–320	–
12	Magnetite	19–629	FC cubic
13	Aluminium phosphate	20–44	
14	Iron phosphate		

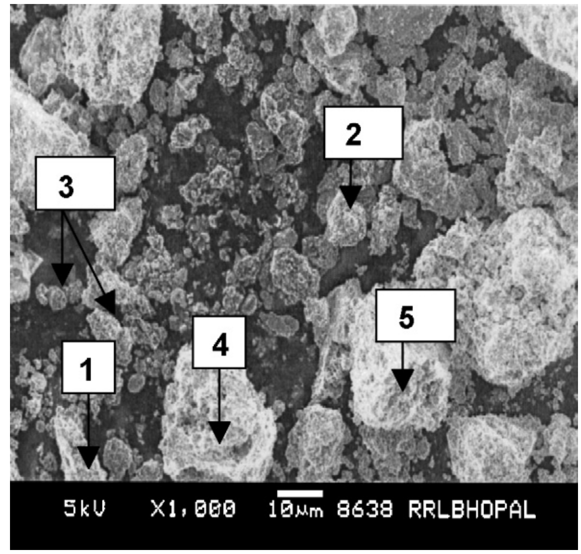


Fig. 4. SEM of red mud (scattered morphology): (1) tetragonal-anatase, (2) tetragonal-rutile, (3) hematite spherical, (4) hexagonal cancrinite and (5) orthorhombic-boehmite.

3.3. Morphology of powdered sintered shielding material samples

The scanning electron microphotographs exhibiting microstructure of red mud as such and of shielding tile samples are given in Figs. 4 and 5, respectively. The presence of oxide of sodium, silica, calcium[48] and barium[49] promotes liquid phase sintering in the red mud based ceramics leading to the formation of very dense matrix which is responsible for providing high mechanical strength and effective shielding. The microphotographs of shielding sample clearly show the compacted and continued integrated morphological characteristics. The microphotograph clearly reveal the surface texture of very dense hexagonal—barium iron titanium oxide, tetragonal—iron titanium oxide, cubic—magnetite and orthorhombic bafertisite phases (cf. Fig. 5), where as the scattered morphology texture

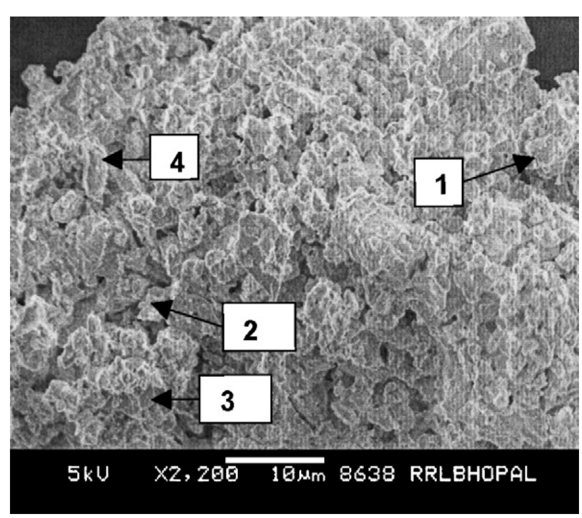
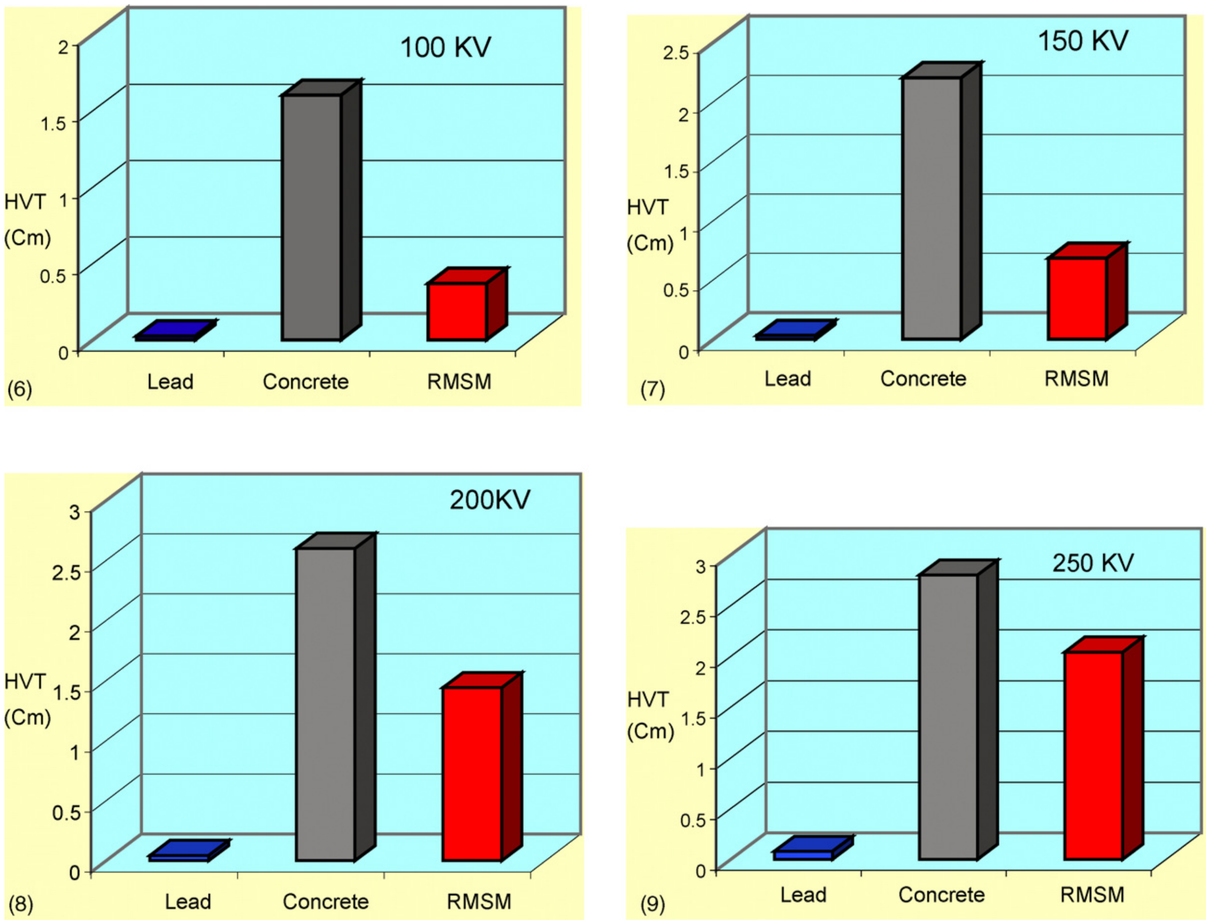


Fig. 5. SEM of RMSM: (1) hexagonal-barium iron titanium oxide, (2) cubic-magnetite and (3) orthorhombic-bafertisite.



Figs. 6–9. Comparison of shielding thickness in terms of half value thickness (HVT) at different energies of X-ray: (6) X-ray 100 kV, (7) X-ray 150 kV, (8) X-ray 200 kV and (9) X-ray 250 kV.

like aluminum silicates distributed with heavy constituents phases, such as tetragonal—anatase and rutile, spherical hematite, hexagonal—cancrinite and orthorhombic—boehmite are seen in the microphotograph of red mud alone (cf. Fig. 4).

3.4. Determination of the attenuation characteristics of the shielding materials

The results of narrow beam X-ray attenuation characteristics of shielding materials at various effective energy of X-ray in terms of the half value thickness (HVT) for different energies of X-ray photons are given in Figs. 6–9. On the basis of these data the half value thickness (HVT) has been computed and compared[50] with conventional concrete and lead materials and shown in Table 3. From the HVT values of shielding materials at various energies of X-ray photons, it is found that shielding materials if converted to thick slab of about 6 in. thickness can preferably be used for the construction of X-ray diagnostic and CT- scanner room to provide adequate shielding against X-ray photons.

Table 3
Comparison of shielding thickness in terms of half value thickness (HVT) of red mud based shielding materials lead and concrete at different energies of X-ray photons

KVp of X-ray beam	Lead	Concrete	Red mud based shielding materials
100	0.025	1.6	0.37
150	0.029	2.2	0.68
200	0.042	2.6	1.44
250	0.080	2.8	2.04

3.5. Mechanical properties of shielding materials

The result of bulk density determination of various composition of shielding material sample is given in Fig. 10. The result shows that shielding materials possess significant high bulk density, i.e. up to 3.4 g/cm^3 in comparison to 2.2 g/cm^3 of red mud. This increased bulk density is responsible for imparting effective shielding to the developed RMSM.

For evaluating the compressive and impact strength of the shielding materials, samples were made in the form of cube and ceramic tiles, respectively. The results obtained for compressive strength and impact strength determination are given in Figs. 11 and 12, and it is found that shielding materials samples meets the requirement of compressive and impact strength prescribed in the Indian standard specification nos. 9103–1999 and nos. 777–1970 for ceramic tiles and concrete, respectively.

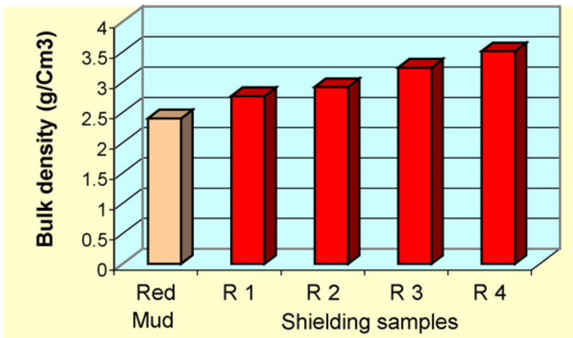


Fig. 10. Density of red mud and shielding samples.

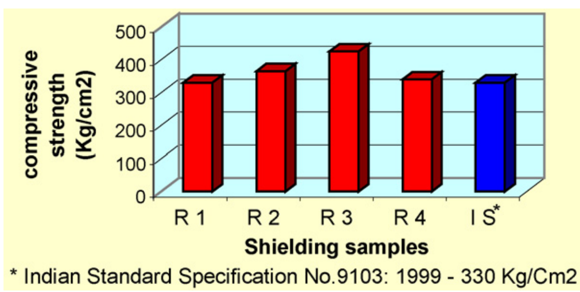


Fig. 11. Compressive strength of shielding samples.

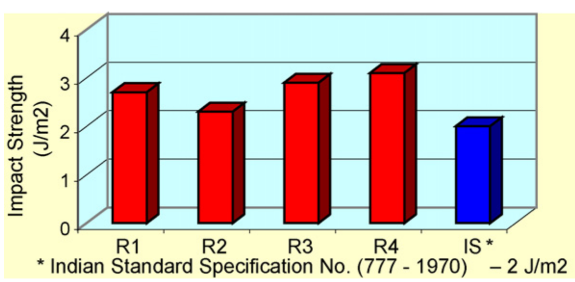


Fig. 12. Impact strength of shielding samples.

Therefore, shielding materials can be used as structural materials for the X-ray diagnostic and CT-scanner room installation.

4. Conclusions

Based on the results of present studies, carried out on the development of X-ray radiation shielding materials using red mud and barium containing compounds, following conclusions can be drawn:

1. The results obtained represent a fundamental starting point for utilization of red mud in mixture with barium compounds, as resource material for making X-ray shielding materials.
2. The shielding thickness (HVT) of the red mud based shielding materials, in comparison to concrete, is significantly very less for the various energies of X-ray photons, i.e. 100, 150, 200 and 250 kV and therefore red mud based Shielding materials can provide effective shielding at very less thickness itself.
3. The red mud based shielding materials (RMSM) confirm to the requirement of compressive strength and impact strength as specified by Indian Standard for cementious shielding materials and ceramic tiles.
4. The RMSM have exhibited the most effective shielding for 100 kV X-ray and adequate strength requirement of structural materials and therefore can preferably be used for the construction of X-ray diagnostic and CT-scanner room.
5. The X-ray powder diffraction studies of the RMSM sample have confirmed the presence of different shielding phases namely barium iron titanium oxide, iron titanium oxide, magnetite, barium aluminum silicate (celsian), bafertisite, etc.
6. The scanning electron microphotographs of the shielding tile sample have clearly revealed very dense morphologies of hexagonal—barium iron titanium oxide, tetragonal—iron titanium oxide, cubic—magnetite and orthorhombic bafertisite phases.
7. The formation of the dense and continued integrated morphological characteristics is responsible for imparting observed effective shielding and structural characteristics to the RMSM.

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