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Utilization and value addition of copper tailing as an extender for development of paints

Mohini Saxena*, Lokesh Kumar Dhimole

Regional Research Laboratory (CSIR), Hoshangabad Road, Near Habibganj Naka, Bhopal 462026, Madhya Pradesh, India

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Abstract

The present study deals with characterization of copper tailing waste, test for possibilities of hazards and its potential reuse as an extender in paints. The waste is a siliceous material containing aluminum oxide, iron oxide and sulphate in significant concentrations. In the primary stage waste is acidic in nature, which makes it unsuitable for paint. This acidity is removed from the waste by simple sieving and grinding. The prepared mass was characterized for basic properties of an extender like oil absorption, specific gravity, pH, etc. Toxicity studies were also conducted in term of leaching of heavy metals by standard techniques (USEPA using TCLP). Properties of the prepared paint's film in terms of hardness, adhesion, resistance to abrasion, resistance to impact, resistance to corrosion (under humidity and salt fog), etc. were evaluated and compared with a similar formulation of conventional extender and found satisfactory. Results from the experiments indicated that developed extender is environmentally clean and cost-effective.

Keywords: Copper tailing; Heavy metals; Leaching; Extender; Paints

1. Introduction

Metal industry is the backbone of any modern and civilized country. The use of metal is growing day by day in view of industrial development and advancement in the technology. Metals are extracted from their ores. The ores usually may contain very small concentrations of the targeted metal, i.e. 1–2% for copper and 0.05% for uranium [1]. As a result, large amount of impurities are separated during metal extraction. These impurities are waste and need safe disposal. Mining and metal extraction procedures produce waste at different stages. Waste rocks are produced during mining operations [2]. Tailings are generated during preliminary stages of ore beneficiation, and hydrometallurgical wastes are generated during metal extraction [3]. Tailings come as a by-product of ore beneficiation processes like crushing, grinding, concentration, etc. [4]. Tailings are siliceous materials containing silica, as the main constituent. Some quantity of iron oxide and alumina are also present in tailings [5,6]. However, the composition may vary widely from site to site depending on type of rock mined. In general ores of most of the heavy metals are sulphur-based minerals, almost always accompanied by pyrite and/or pyrrhotite [7,8]. Tailings also contain heavy metals like Cu, Mn, Cr, Se, Ni, etc. [7]. The waste covers large area of land for their disposal as tailing impoundments and piles. The presence of sulphur causes big problem when this waste comes in contact with air, water and moisture, which transforms sulphur in to sulphate in the series of reaction with due course of time, thereby making the waste acidic with the production of sulphuric acid [9]. This problem is known as acid mine drainage [10]. The chemical process involves incorporation of heavy metals in surface water reservoirs, ground water storage and fertile soil [3]. Therefore, the disposals of these wastes have become a great challenge for scientific community. The best way to overcome the challenge is its utilization leading to valuable outcomes.

Those tailings, which are siliceous materials, can be used where silica is required as ingredients such as in building material and road construction industry. A lot of attempts have been made in this respect; bricks of comparatively higher strength were prepared by tailing wastes [11]. Productions of glass wool and glass fibre have been exploited using tailings as source of

* Corresponding author. Tel.: +91 755 2459827; fax: +91 755 2459827.
E-mail addresses: saxenamohini@hotmail.com (M. Saxena), lokeshdhimole@rediffmail.com (L.K. Dhimole).

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silica by the researchers [12,13]. Copper tailing had been used in highway construction [14].

Moreover, an attempt has been made in the present study to utilize copper tailing in paints as an extender. Previously fly ash waste from thermal power plant had been used as an extender and found better as compared to similar types of conventional extenders [15]. Blue dust waste from iron industry, red mud waste from aluminum industry was utilized in preparation of priming coats replacing conventional commercial iron oxide [16–18]. Improvement in surface properties of red mud was also reported in literature [19].

Organic coatings or paints are generally applied for the protection of substrate against the environment and for aesthetical needs. This is a much economical way to protect metals from corrosion as compared to electroplating and other means of surface protection. Polymer is used as a binder in this case. It plays a major role in terms of barrier properties like impermeability for vapours of corrosive substances. A smooth surface is responsible for glossy appearance of the coating. However, solid part of the paint system includes pigments and extender. The pigment contributes to colour, hiding and tinting, while the extender imparts mechanical properties to the coatings. Some extenders are also used as corrosion inhibitor like iron oxide while silica is the best choice for abrasion resistance [15]. In addition extenders are economically viable. Therefore, their addition makes the formulation cost-effective as well. Due to great demand, paint industry is growing with a rapid rate of 2.5% per year. All the components of paint system can be synthesized on industry level except the extenders, which are, natural minerals and need mining and extraction. A very high amount of these natural minerals is wasted during the process of grinding, crushing and levigation [20]. The percentage of recovery is very low for these extenders, i.e. 30% for china clay and 70% for calcite [21].

2. Materials and methods

2.1. Characterization of copper tailing waste (as received)

Copper tailing waste was collected from Khetri, Rajasthan, India. It was characterized for elemental composition, mineral phases, content of heavy metals, pH. Elemental composition was determined by Energy Dispersive X-Ray Analyser, Oxford, attached with Scanning Electron Microscope, JEOL5600, Japan. Identification of mineral phases was done by X-ray diffraction technique using Philips diffractometer (pw 1710). Heavy metal content was determined by acid digestion method using an atomic absorption spectrophotometer [22].

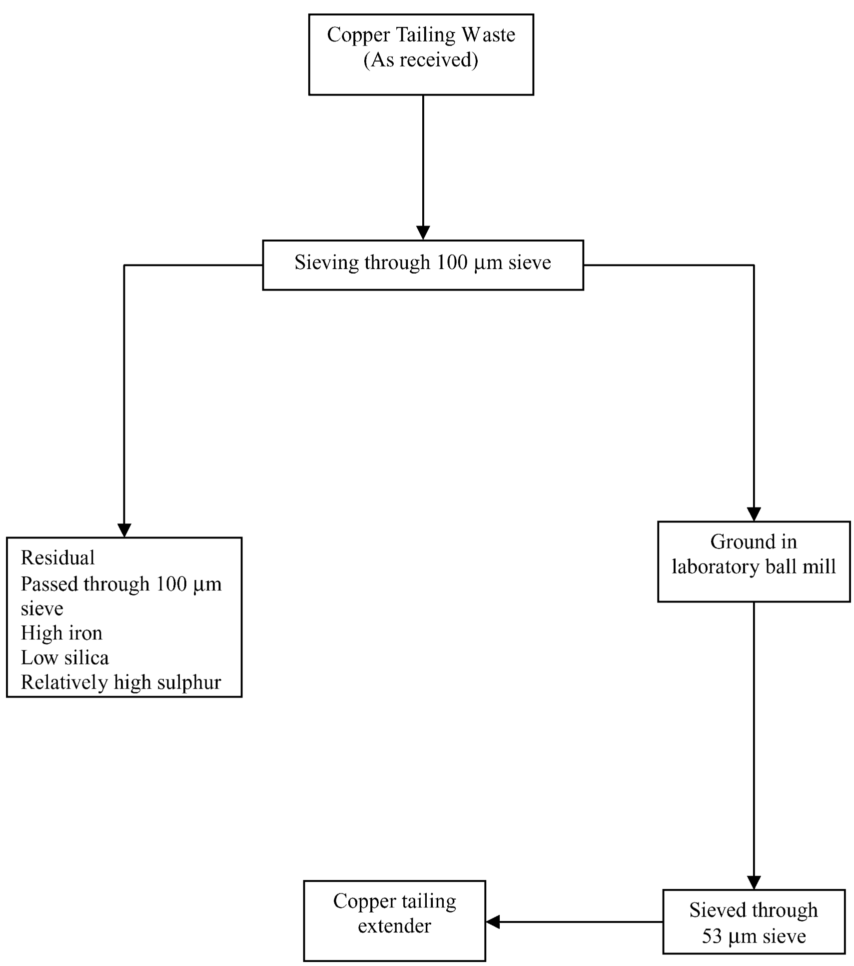

Fig. 1. The process, adopted to separate coarse and fine particles of as received copper tailing waste.

2.2. Processing of extender

The coarse particles of as recieved copper tailing waste were separated using a 100-μm sieve. This coarse fraction was ground into fine powder by ball mill and sieved again through a sieve of 53 μm. The process is given in Fig. 1.

2.3. Characterization of copper tailing after processing

After the processing, the prepared extender was again characterized for the properties as described earlier. Extender properties of copper tailing extender such as pH, specific gravity and oil absorption were determined by standard procedures (ASTM D-1208-89, ASTM D-281-89 and ASTM D-153-89, respectively) [23–25] and compared with those of conventional extenders. The extender was also characterized for reactivity with acids and alkaline solutions according to method prescribed by Oil Technologist Association of India [26]. Leaching behavior of heavy metals from the extender was also studied as per the standard procedure of Environmental Protection Agency, USA, using a toxicity characteristics leaching procedure (TCLP) equipment, supplied by Millipore Water Purification System Ltd., Billerica, MA, USA.

2.4. Raw materials for paint formulations

Commercial grade epoxy ester resin (Synpol 5545) was used as binder in prepared paint formulations. Titanium dioxide was used as white pigment and other raw materials like solvent (mineral spirit), driers, antiskining agents and antisettling agents were also used in preparing paint formulation.

2.5. Preparation of paint formulations

Six formulations of paint having different percentages of copper tailing extender and conventional calcite extender were prepared to carry out the comparative study. The process involves mixing all ingredients and adding appropriate quantity of solvent to the mill base to obtain desired consistency prior to form a smooth paint paste. The mill base was then ground in a ball mill until the desired fineness of grind gauge was achieved (ASTM D 1210-1988) [27]. Curing agents were added in to paints just before the application on test specimens. Measurement of viscosity of all the six formulations was done using ASTM D 1200-1993 [28].

2.6. Preparation of specimens for characterization of paint film

Mild steel panels having dimensions of 150 mm × 100 mm were used for measuring the hardness, impact resistance, adhesion, humidity resistance and salt fog resistance while panels of dimensions 60 mm × 50 mm were used for testing of Abrasion resistance. Surfaces of the panels were cleaned with emery paper to remove rust and then degreased with suitable solvent like xylene. The coating applied on the surface of all the test specimens, was through hand handed spray gun except for hardness where thin film was applied by mechanical bar applicator. Drying time of paint formulation was measured as per standard method (ASTM D 3732-1993) [29].

2.7. Characterization of paint film

Hardness of the film was measured on the 7th day of application in accordance with ASTM D-2134-93 [30], using Sward type hardness rocker. Impact test was carried out using a Dupont impact tester. This consists of a vertical stand graduated in inches through which certain weights of 100, 200, 300 g, were allowed to fall on the pointers of different diameters. Pointer was kept vertical on the paint film. The observation of any damage on paint film was analysed in units of gram/inch. Abrasion behavior of paint film was tested using a high stress Suga (Two Body) abrasion tester for 200 cycles. The abrasive medium used was an emery paper bonded with SiC particles of desired size.

Cross hatch cutter following standard method of ASTM D-3359-93 [31] enabled to determine the adhesive property of the paint film on the metal surface.

Testing of the painted panels in humidity chamber and in salt fog chamber was carried out for durability studies. Specimens coated with all six formulations were kept in the humidity chamber for 7 days where the samples are exposed to temperature cycles of 42–48 °C at an interval of 70 min. This provides extreme conditions of condensation of water vapour on the test specimens. Visual observations were made every day throughout the test to see any blistering and sign of corrosion on the test panels. Salt fog chamber provides conditions for permeation of salt saturated water vapour through the paint film. This test was continued for 4 days. Surfaces of the exposed panel after the test were observed after 24 h for blistering and corrosion.

3. Results and discussions

3.1. Properties of processed extender

Copper tailing waste in its as received stage is not appropriate to use in paints as an extender. The waste is acidic in nature having pH 3.6 which may lead to corrosion when the prepared paint is applied on metal surface. To get less acidic nature of the waste, coarse particles were separated by a simple physical process as explained in Fig. 1. After processing the waste, there was a marked improvement in the pH from 3.6 to 6.5. This is because of low surface area of the separated coarse particles, which leads to less accumulation of acid. The difference in the ratio of surface area to mass for a particle of 1 cm in diameter and a particle of 1 μm diameter is 1–10,000 [32]. The observed pH of 6.5 suits the requirement of an extender in paints. Also chemical (Table 1; Figs. 2–4) and mineralogical characteristics (Fig. 5) of the copper tailing extender reveal its suitability towards extender application. XRD analysis gives maximum peak of 100% relative intensity of SiO_2 as quartz. Al_2O_3 and Fe_2O_3 are also present as significant mineral phases in different combinations. The mineral phases are given in correspondence to the observed

Table 1
Chemical composition of different fractions of copper tailing

Compounds	Percentage in raw waste	Percentage in extender	Percentage in residual
SiO_2	49.91	63.18	38.70
Fe_2O_3	31.85	17.74	41.93
Al_2O_3	7.63	10.89	8.05
SO_3	3.00	2.31	4.21
K_2O	1.24	1.04	1.59
MgO	2.69	2.02	2.26
CaO	1.88	1.33	1.40
CuO	1.79	1.49	1.84

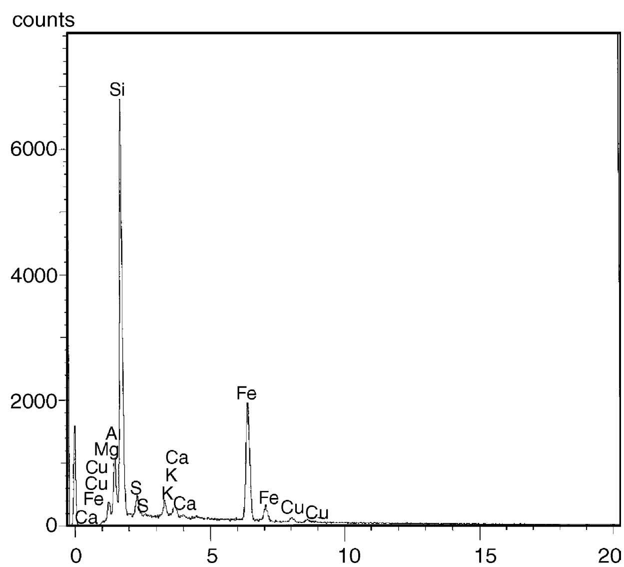

Fig. 2. Analysis of as received copper tailing waste by energy dispersive X-ray analyser (EDXA).

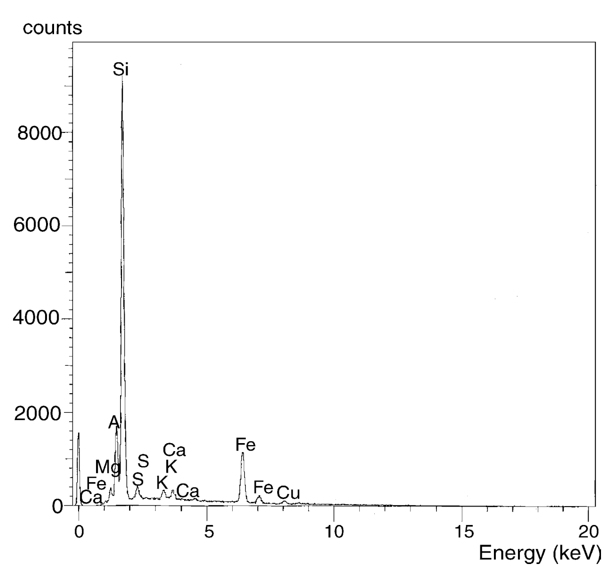

Fig. 3. Analysis of copper tailing extender by energy dispersive X-ray analyser (EDXA).

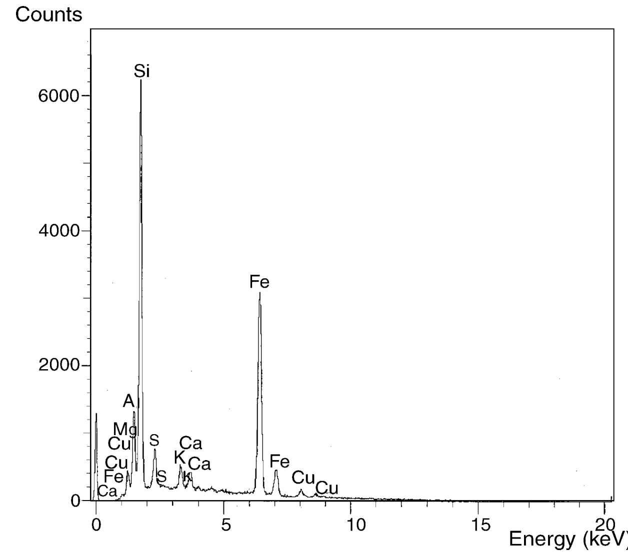

Fig. 4. EDXA of residual copper tailing remained after separation of coarse particles.

peaks intensity in Fig. 5. Mineral phases like Silica and aluminum silicates are reported as good abrasive materials [15]. EDX analysis of the raw copper tailing, processed copper tailing and finer residual part remaining after separation of coarse particles is shown in Figs. 2–4, respectively, and Table 1. The percentage of silica and alumina increased, however iron oxide content reduced in the extender (Table 1; Figs. 2 and 3). It is found that, iron content passed through sieve and found high in finer residual part (Fig. 4). Oxides of calcium and magnesium are also present. This composition is some how similar to diatomaceous silica, an extender for paint that contains SiO_2 86%, Al_2O_3 5% and some amount of Fe_2O_3.

3.2. Environmental studies

Heavy metal concentrations in the processed waste are in the trace amount except iron and copper. The waste is characterized for heavy metals like Cu, Mn, Zn, Pb, Ni, Cr, Co and iron. The concentration is given in Table 2. The presence of these heavy metals requires proper study of their leaching properties. The heavy metal concentration in Table 2 shows that less amount of heavy metals is present in the extender as compared to metallic compounds that are used in paint like red lead (Pb_3O_4), Chrome Orange ($PbCrO_4$. PbO), lemon chrome ($PbCrO_4$), etc. which

Table 2
Heavy metal concentration in processed waste

S. no.	Heavy metals	Concentration (ppm)
1	Copper	71
2	Manganese	16.50
3	Zinc	1.50
4	Lead	0.43
5	Nickel	1.65
6	Chromium	2.56
7	Cobalt	0.50
8	Iron	2290

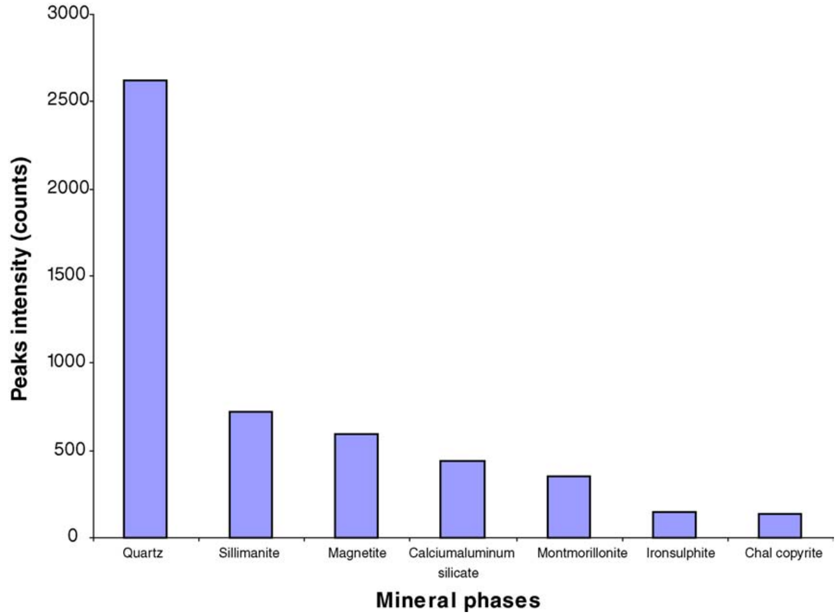

Fig. 5. X-ray analysis of copper tailing extender.

Table 3
Leaching of heavy metals from copper tailing extender

S. no.	Heavy metals	Concentration (ppm)	TCLP limits (ppm)
1	Arsenic	0.001	5
2	Barium	BDL	100
3	Cadmium	BDL	1
4	Chromium	0.017	5
5	Lead	BDL	5
6	Copper	0.30	–
7	Zinc	0.40	–
8	Selenium	0.001	1
9	Nickel	0.86	70

contain very high amount of these toxic substances, usually in percentage. The possible environmental hazards in term of leaching characteristics of heavy metals was also examined and found below the range of limits prescribed by the USEPA under toxicity characteristics leaching procedure. Leaching behavior of heavy metals is given in Table 3, which shows that insignificant concentration of heavy metals is leached out from the waste. This leaching study indicates it to be safe and non-hazardous. Moreover, leaching is expected to reduce further because extender is used in paint system where polymer binds it under water impermeable solid layer and the same behavior is noticed practically also.

3.3. Extender properties of processed waste

Table 4 shows a comparison of the copper tailing extender with other conventional extenders. The oil absorption value of copper tailing extender is 22–24%. Low oil absorption indicates economical viability of the extender because of low consumption of binder without compromising aesthetic properties like gloss, dispersion and interfacial bonding of the paint. Oil absorption of an extender also gives an idea of theoretical critical pigment volume concentration (CPVC), which shows maximum percentage of extender or pigment in paint over which air starts to occupy the place between pigment particles. CPVC for the waste is 58%. Specific gravity is 3.01, which is within the range of other commercial extenders and does not cause any hard settling during the storage of the paint. The processed extender is also found to be alkali resistant while very slight colour fading occurs in acidic solution of hydrochloric acid. The ability of the processed extender to be dispersed in paint system was found to be satisfactory on Hegman's gauge. The particle size has reduced to below

Table 4
Comparison of copper tailing extender with other conventional extenders

Extenders	Colour	pH	Oil absorption (%)	Specific gravity	Reactivity
Copper tailing	Dark grey	6.5	22–24	3.01	Inert to alkaline, insoluble in water, slight colour fading in HCl
Calcite	Off-white	8.74	29	2.80	Soluble in dilute mineral and acetic acid
China clay	White	–	30–60	2.60	Chemically inert
Barytes	White	–	20–24	4.50	Chemically inert
Talc	White	–	–	2.65–2.85	Partly soluble in dilute HCl
Silica	White	–	–	2.80	Chemically inert

Table 5
Description of prepared formulations

S. no.	Formulation	Description
1	1(a)	Formulation with 50% pigment concentration of copper tailing
2	1(b)	Formulation with 50% pigment concentration of calcium carbonate
3	2(a)	Formulation with 40% pigment concentration of copper tailing
4	2(b)	Formulation with 40% pigment concentration of calcium carbonate
5	3(a)	Formulation with 30% pigment concentration of copper tailing
6	3(b)	Formulation with 30% pigment concentration of calcium carbonate

15 μm while this is near to 10 μm for conventional calcite based paint.

3.4. Properties of paint

The scheme of paint formulation is given in Table 5. The viscosity of the paint developed with copper tailing extender is slightly lower as compares to the paints with conventional extenders (Fig. 6). Drying time was similar to calcite based paint formulations. The performance of paint film in terms of mechanical strength and durability in adverse environmental conditions is sufficiently good (Table 6). Formulation nos. 1(a) and 1(b) with 50% had maximum hardness. It gives good resistance to hammer, i.e. impact resistance (direct), which was found identical to the formulation using conventional calcite extender. Silica imparts good abrasion property in copper tailing extender. Formulation no. 2(a) with 40% is found to have lowest weight loss, which shows that at 40% pigment-extender gives good resistance to wear. Increasing the pigment percentage to 50%, however, gives less wear resistance as compared to the formulation by conventional calcite extenders. In general higher hardness of the material is indication of good wear and impact resistance but for systems like paint, wetting of extender particles; interfacial bonding between paint and substrate and between extender and resin is also essential to consider. Thus, resin percentage also affects mechanical behavior. This is supported by maximum wear rate of formulation nos. 3(a) and 3(b) with 30% pigment-extender concentration. This may be because of low surface hardness of paint film in these two formulations. Hardness increases with increasing percentage of extender. Despite these good mechanical features, the adhesion of the all formulation was found very good as per ASTM standards, i.e. less than 5% of the area is affected when cross cut area is pulled by an adhesive tape.

All the six formulations were exposed to 100% humidity and high temperature conditions in humidity chamber. Formulation nos. 3(a), 3(b), 2(a) and 2(b) were completely free from corrosion and blistering while 1(b) had shown very slight blistering. More blistering has taken place in the formulation no. 1(a). However, the metal surface remained free from corrosion. The blistering in the 1(a) started on the 6th day of test.

The performance of paint in the salt fog chamber was found satisfactory except 1(a), where slight blistering was observed.

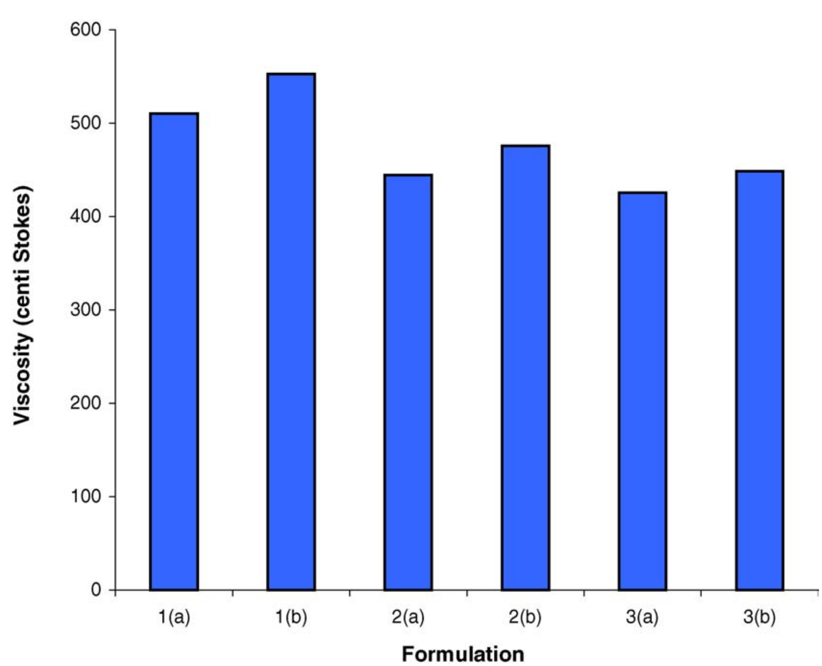

Fig. 6. Viscosity of prepared paint formulations.

Table 6
Mechanical and durability properties of paint coating

Properties/formulation	Hardness (%)	Abrasion resistance (wear rate, $\times 10^{-9}$ m^3/m)	Impact resistance direct (passing level) (g/in.)	Adhesion (level of adhesion)	Corrosion resistance (humidity chamber)	Corrosion resistance (salt fog chamber)
1(a)	11.0	0.167	500/20	4B	Slight blistering	Slight blistering
1(b)	10.0	0.164	500/20	4B	Satisfactory	Satisfactory
2(a)	8.57	0.163	500/20	4B	Good	Good
2(b)	7.14	0.166	500/20	4B	Good	Good
3(a)	7.14	0.168	500/20	4B	Good	Good
3(b)	7.14	0.169	500/20	4B	Good	Good

4. Conclusions

Outcome of the present study, indicates that copper tailing waste after proper processing has good potential to be used as extender in paints with respect to oil absorption, pH, specific gravity, etc. Physico-mechanical properties of the paint film like hardness, impact resistance and abrasion resistance and adhesion were found better compared to the conventional extender. The performance in corrosive atmosphere was good except the paint with 50% pigment. This was due to blistering at the end of the tests in the humidity and salt fog chamber. The processed extender satisfies EPA norms and falls in non-hazardous category. The aim of this study was to provide an idea for researchers of paint and other related fields to go for alternatives, especially for waste materials to save non-renewable natural resources.

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