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Rheological characterization of concentrated jarosite waste suspensions using Couette & tube rheometry techniques



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ABSTRACT

Hydraulic disposal of jarosite wastes at high solid concentrations may be considered as a safe mode of disposal from environmental and economic points of view. The rheological and pipe flow characteristics of jarosite waste slurry samples in mass concentration ranging from 40 to 60% have been investigated. The rheological experiments performed on the samples using both Couette and tube rheometry techniques indicated non-Newtonian pseudo plastic behaviour and were well described by a power law model in the studied range of concentrations. The pressure drop for the suspension was predicted from the estimated true flow curve data in a novel type tube viscometer after incorporating necessary wall slip and entrance-effect corrections. The predictive capabilities of the pressure drop model in pipe flow velocity range of 0.8–1.8 m/s dramatically improved when compared with the pipeline experimental data in a 100 mm nominal bore pipe. Therefore, it is quite imperative to adopt suitable rheometry techniques to avoid discrepancy in predicting the true flow behaviour of the concentrated jarosite waste suspensions for economic hydraulic disposal.

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1. Introduction

The process slurries within mining, mineral and metal extraction operations with varying physicochemical characteristics exhibit complex rheological behaviours. The estimation of true flow characteristics of these suspensions with non-Newtonian behaviour poses a real concern in several engineering fields. These suspensions may exhibit shear thinning or shear thickening, yield stress, normal stress differences or shear banding depending upon their interactions, particle properties and concentration [1–6]. The theoretical and numerical investigations of these suspensions under laminar flow regime have been reported with more complex rheological models [7–9]. The characterization of microstructure affecting the flow behaviour of a suspension has been investigated by various researchers employing numerical techniques [10–12]. The flow curve of concentrated non-colloidal suspensions containing significant amount of fine (flocculating) particles can be estimated by using various geometries such as Couette (rotational) parallel plate, cone and plate or tube rheometry. The accurate rheological measurements of these suspensions are quite challenging because perturbative effects such as wall slip, shear localization, particle migration, high tendency towards sedimentation and slip layer thickness can arise during the experiments [13-22].

The corrections for wall slip and entrance effects with Couette rheometry techniques are a tedious task as it requires a series of bobs and cups of different shapes, sizes and roughnesses [23–25]. On the other hand, the tube rheometry technique offers a simple and robust method of measuring steady shear stress–shear rate data for time-independent and visco-elastic fluids over wide ranges of shear rates (~50 to 1000 s^{-1}). Due to its low cost, simplicity in construction and quick on-site measurements in industrial settings to obtain the true rheological response of a dense mineral suspension, this technique is preferred over other rheometry systems [26,27].

Presently, ~75% of world's zinc metal is produced through hydrometallurgical route and generates a large quantity of jarosite residues during its processing. In India, Hindustan Zinc Limited (HZL) with its smelter and mining units located in Rajasthan, Andhra Pradesh, Bihar and Odisha generates approximately 2.5 million tons of jarosite per annum [28]. In literature, the rheological and pipe flow behaviour of the fine jarosite waste slurry is scanty. The present improper disposal method and landfill cost management for various mineral and other industrial hazardous wastes including jarosite residues urge for a safe, environmentally sound and effective disposal system [29,30]. To maximize the transport and disposal system, the rheological and pipe flow characteristics of these huge wastes should be well understood which may lead to accurate prediction of pressure loss and other operational parameters such as deposition flow rates and slope of the underlying base from rheological parameters [31–33].

In the present study, efforts are made to characterize the rheological and pipe flow characteristics of jarosite–water suspensions at high solid concentrations (40–60% by weight) for its safe and economical disposal. The study methodology includes physical–chemical characterization,

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Nomenclature

ID	Internal Diameter
NB	Nominal Bore
PSD	Particle Size Distribution
SEM	Scanning Electron Microscope
Symbols	
Cw	Mass concentration
d_{50}	Median diameters of particle (µm)
dwm	Weighed mean diameter of particle (µm)
D	Diameter of tube (m)
К	Consistency index
n	Flow behaviour index
n`	Slope from the log-log plot of τ_{w} vs 8V/D in Fig. 7(a)
Δp_{t}	Total pressure drop measured on a specified length L of
r.	the tube (Pa)
Δn _u	Pressure drop necessary to maintain a viscous flow in
-PV	the tube (Pa)
Δn _a	Pressure drop associated with the entrance effects (Pa)
0	Suspension flow rate (m^3/s)
V	Suspension velocity (m/s)
V	Slip velocity (m/s)
ν _s τ	Shor stress (Pa)
• -	Wall shear stress (Da)
.'w	$\frac{1}{2} \sum_{n=1}^{\infty} \frac{1}{2} \sum_{n=1}^{\infty} \frac{1}$
Ŷ	Shedi Idle (S)

rheological characterization and true flow curve estimation for the jarosite samples by using both Couette (rotational) and tube rheometry techniques. Based on the true flow curve data, a non-Newtonian power law head loss model was employed to predict the head loss of jarosite slurry at high solid concentrations. The predicted head loss was compared with the experimental pipe head loss data in a 100 mm NB pipe to evaluate the applicability of the head loss model for designing & installation of commercial jarosite slurry pipelines.

2. Materials and methods

2.1. Physico-chemical characterization of jarosite samples

In the present study, the solid fine jarosite samples were obtained from the Vedanta group at Hindustan Zinc Limited (HZL), Chanderiya, Chittorgarh, Rajasthan, India. The samples were air-dried and cleaned from foreign materials and stored in a glass container. For characterization, sampling was drawn from the air-dried sample adopting the coning and quartering method. The particle size distribution analysis was done using a Malvern make laser particle size analyzer (MASTERSIZER 2000). For the purpose the samples were first dispersed in distilled water to indicate the obscuration value of 12%. Then ultra-sonication of the samples was carried out for about 1 min to avoid formation of any agglomerates of jarosite particles. The PSD of jarosite sample is plotted in Fig. 1. The median (d_{50}) and weighed mean (d_{wm}) diameters of the sample were found to be 3.31 µm and 4.25 µm, respectively. It also indicated that jarosite is silty clay loom in texture having 68.6% silt sized and 31% clay sized particles. The particle density of the samples was measured using a standard specific gravity bottle and was determined to be 2950 kg/m³. The pH was measured directly in the flasks with a Denver instrument company (Denver, CO) Model 215 Bench top pH meter, equipped with a 3 M KCl highperformance pH/ATC glass-body electrode. Calibration was done prior to each series of measurements using pH 1.0, 2.0, 4.0, 7.0 and 10.0 buffers to capture the range of possible pH values with slurry mixing time. The pH of jarosite waste slurry was found to be 2.36 indicating high acidity. The



Fig. 1. Particle size distribution of jarosite samples.

chemical composition of the jarosite samples was carried out by Philips PW22440X-Ray Spectrometer (XRF).

The major elements (calculated in oxide form) were as follows: SO_3 (42.21%), ZnO (22.45%), Fe₂O₃ (15.21%), SiO₂ (9.18%), Na₂O (8.07%), PbO (1.69%), Al₂O₃ (1.13%), CaO (0.76%) and K₂O (0.31%).

The mineralogical studies of the samples were carried out by an X-Ray Diffractometer (Make: PAN ANALYTICAL, NETHERLAND, Model: X-Pert PRO PAN ANALYTICAL) with X-PERT HIGHSCORE PLUS software packages. The microstructure characteristics were analyzed by a Scanning Electron Microscope—Model JEOL, JSM-5600, Japan with Energy Depressives X-ray Spectroscopy (EDS) analysis facilities. The typical X-ray diffractogram of jarosite is presented in Fig. 2. It is observed from the figure that the major mineral phase in jarosite is iron sulphate hydrate (2Fe₂O₃SO₃5H₂O) and potassium chromium sulphate hydroxide.

The microstructure analysis of jarosite sample is presented in Fig. 3 and it reveals that most of the particle surface is smooth, irregular in shape with multiple humps.

2.2. Rheological characterization

The rheological studies with the jarosite samples were carried out using both Couette and tube rheometry techniques. Initially, the rheological experiments were conducted using a HAAKE Rotational Rheometer (Model: RheoStress 1, Thermo Fisher Scientific). A cylindrical sensor system having bob and cup radii of 20.71 mm and 21.7 mm respectively was



Fig. 2. X-ray diffraction of jarosite showing phase constituents.

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