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# Powder Technology

# Design and synthesis of novel polyamine collector to recover iron values from iron ore slimes

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## ABSTRACT

The present work relates to the design, synthesis and analysis of amine based reagents for enhanced iron ore-gangue separation using the adsorption (or interaction) energy and flotation experiments of minerals with cationic reagent. The novel Polyamine based cationic collector is modelled and synthesised followed by testing in a lab scale Denver flotation machine for their effectiveness towards reverse flotation of aluminosilicate minerals. The adsorption behaviour is assessed in pH media to understand the affinity of amine cationic collector with the $SiO_4$ tetrahedral and $AlO_6$ octahedral surfaces of kaolinite. Computed interaction energies of cationic reagent shows maximum adsorption with gangue minerals (here alumina, silica and kaolinites) than goethite and hematite. The results of theoretical calculations in terms of the prediction of the order of response of kaolinite and goethite minerals to flotation with amine based cationic reagent were found to match remarkably well with the experimental flotation test results. The flotation analysis reflects the adsorption trend and showed excellent selectivity with respect to alumina/silica removal. The adsorption energy and flotation experiment of amine based cationic collectors paved a whole new chapter in the recovery of iron values from iron ore slimes.

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## 1. Introduction

In India, high grade hematite iron ores have been used for iron and steel making either through blast furnace or electric smelting route. However, the ever increasing demand of steel production and tapering of high grade iron ores has imposed the dependence on low grade ores, fines, slimes and lean grade ores as Banded Hematite Quartz, Banded Magnetite Quartzite or Banded Hematite Jasper etc. The Iron ore slime sample from eastern India generally assays 52%–55% Fe, 7.00–8.5% $Al_2O_3$, and 6.0–7.5% $SiO_2$ [1,2]; Ravishankar et al., 1995; [3–5]. The associated minerals in this iron ore slimes are mostly goethite, kaolinite with some portion of gibbsite. Iron ore slimes have to be processed to a certain level to meet the requirements from the steel producers. However, the commercial exploitation of slimes has not been established so far due to the lack of suitable beneficiation processes which are capable of beneficiating at a very fine size. Flotation is one of the key operations used for the concentration of low-grade iron ores. In reverse flotation of iron ore, amines are the most commonly used collectors for the collection of quartz [45]. Alumina is the major impurity in iron ore slimes which is present mainly as kaolinites. The

alumina to silica ratio is typically greater in these ores and as we all know is detrimental to iron making process through smelting. In general particles of size below 45 μm are difficult to beneficiate. The advantage of higher particle surface area however provides surface active reagents the required scope for its use in beneficiation of ultrafine size [2]. These surface acting forces come into play when we use the flotation techniques for separation i.e. cell flotation and column flotation. In other parts of the world, slime is beneficiated for magnetite and hematite rich iron ore in which silica is the major gangue mineral [6–8]. This technology cannot be implemented in India due to the very different nature of Indian iron ore slime. Compositionally, Goethite is identified as the major iron bearing mineral phase and kaolinite is a common gangue mineral frequently found in iron ore deposits [9]. The detailed characterisation of Indian iron ore slimes have been reported in earlier literature[1,10–12]. Flotation of iron ore slimes from eastern region of India have been reported in literature too [13]. A significant problem with goethite is their high $Al_2O_3$ and $SiO_2$ content. Several researchers have tried various combinations to beneficiate iron ore slimes such as hydro cyclone classification followed by magnetic separation, spiral concentration, selective flocculation and flotation. People have tried flotation with mixed collectors systems too. All these efforts have borne some results and showed the way for the future research [3,5,14–19]. All the efforts have achieved some success with respect to gangue

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reduction in slimes and especially with respect to silica reduction in Indian iron ore slimes. Iron ore minerals like goethite can be floated by a variety of collectors, such as amines, oleates, sulfonates and sulphates [30]. Beneficiation of iron ore slimes containing significant amount of Fe along with $SiO_2$ and $Al_2O_3$ can be concentrated either by reverse cationic flotation of aluminosilicates (kaolin) or direct anionic flotation of Fe [31]. The cationic reverse flotation of aluminosilicates seems to be an attractive route for the concentration of low grade ores [32,33].

## 1.1. Brief review of cationic surfactants for reverse floatation of aluminosilicates

It is an established fact that amines are the most suitable reagents for reverse floatation of iron ores containing aluminosilicates as the main gangue. Primary and secondary amines, ether amines, quaternary ammonium salts, alkyl guanidine collectors constitute the general class of reagents used in the floatation of aluminosilicates [20,21]. Researchers like Jang Liu and Jan miller have worked extensively to investigate the various factors of reverse flotation and reveal the mechanism underlying. They have studied the reverse flotation of bauxite to remove kaolinite by a common amine collector dodecyl amine in one of their recent investigations and analysed the flotation mechanism of kaolinite from bauxite on the kaolinite surface properties, particle interaction, and the interaction between kaolinite surfaces and dodecylamine. This type of understanding can provide important insights for evolving new chemicals for the reverse flotation of kaolinite from low grade bauxite and iron ores [22]. Researchers like Hong Zhong, Guangyi Lui and Yuehua Hu have worked extensively in the area of cationic reverse floatation for the separation of illite, pyrophyllite and kaolinite from diaspore. They have explored the role of cationic collectors like novel amino-trisiloxane Gemini cationic surfactant, butane-1, 4-bis (dimethyl-(3-(3-aminopropyl trisiloxane-3-yl)-propyl)-ammonium bromide) (BBAB) in the reverse floatation of aluminosilicates. People have worked with Gemini surfactants like butane-α,ω-bis(dimethyl dodecyl ammonium bromide) (BDDA) for cationic reverse floatation for the separation of illite, pyrophyllite and kaolinite from diaspora and also studied the effect of cationic groups on 12C-chain collectors in flotation separation of diaspora from kaolinite, pyrophyllite and illite through experimental work and DFT calculations. All these work shows that specific and selective reagents have to be used to separate the wanted mineral from the gangue and DFT is an efficient tool to design these kinds of selective reagents. [23,25–29]. Another important constituent of reverse floatation is the role of depressants. The role and importance of depressants mainly starch and polysaccharides have been a dominant area of research for many years. Recently researchers like Shrimali and Jan Miller have worked extensively on the different aspects of starch adsorption on iron oxide surfaces and proposed the probable interaction mechanisms of starch with iron oxide systems [24].The key to develop a successful
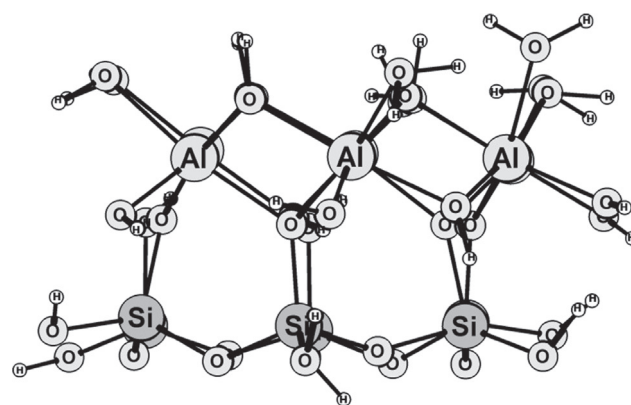


**(a) Kaolinite**
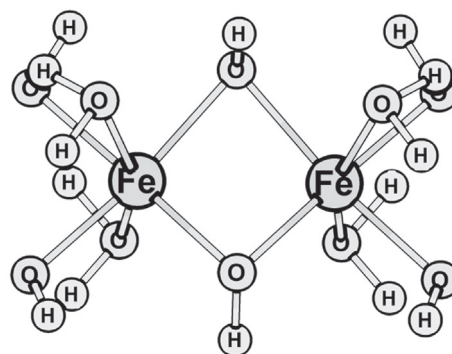


**(b) Goethite**

Fig. 2. The optimized geometries of (a) Kaolinite and (b) Goethite.

flotation separation process for Indian iron ores is thus to find selective reagents for the separation of iron ore minerals (goethite) from alumina containing minerals (kaolinite).

The development of selective reagents which can take out alumina/silica from the ores can be the only practical solution to make the beneficiation of Indian iron ore slimes possible. The selectivity of the collector and mineral interaction is determined by a wide range of chemical, thermodynamic and steric factors. For the beneficiation of multicomponent, highly disseminated and difficult to-treat ore deposits, the conventional approaches of reagent design and selection are inadequate. A quantitative methodology to screen out/identify the appropriate molecular architectures from theoretical computations, is evidently an
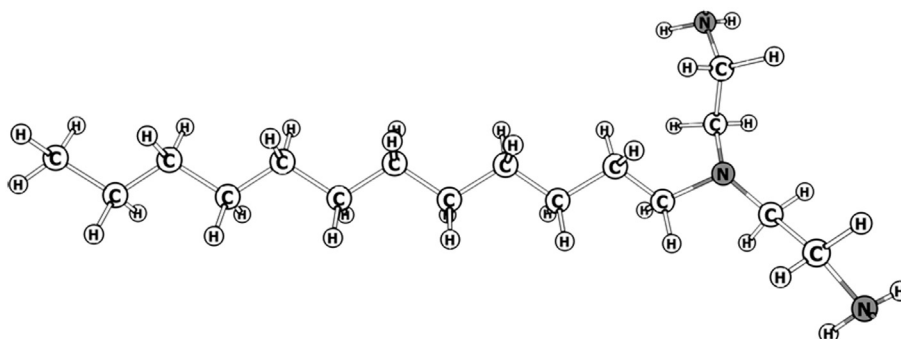


Fig. 1. The optimized geometry of polyamine collector [($C_{12}H_{25}N$ ($C_2H_4NH_2)_2$].