## Original Article

# Apatite flotation using pataua palm tree oil as collector

*Priscila de Oliveira\*, Herman Mansur, Alexandra Mansur, Gilberto da Silva, Antonio Eduardo Clark Peres*

*Department of Metallurgical and Materials Engineering, Universidade Federal de Minas Gerais, Brazil*

### ARTICLE INFO

### ABSTRACT

Most apatite concentrates, essential for the fertilizer industry, are produced using froth flotation, where collectors play a pivotal role. Although vegetable oil-derived surfactants have been used for decades in flotation processes, raw materials which do not compete with human or animal nutrition deserve special attention. This study investigated the pataua palm tree (*Oenocarpus bataua*) oil regarding to its fatty acids chemical profile, saponification and iodine values, acidity index, and evaluated its potential as a source for a collector to be employed in phosphate ore flotation. The results indicated that the pataua oil is primarily composed of oleic acid, with minor contents of linoleic, palmitic, lauric, myristic, palmitoleic, stearic and linolenic acids. Selectivity of apatite from quartz and calcite was achieved at neutral and alkaline pH with dosages as low as $0.3 \, \mathrm{mg.g^{-1}}$. These findings were interpreted based on FTIR and zeta potential measurements, indicating that the collector adsorption onto the apatite surface occurred through mechanisms of chemisorption and calcium salts precipitation. This study suggested pataua oil as a prospective collector in flotation systems aiming at apatite concentration with an environmentally sustainable strategy.

## 1. Introduction

The impact on the environment increasingly raises concerns regarding mining activities. The search for an sustainable industry is encouraging researchers to create innovative solutions for mineral exploitation based on the reduction of pollutants released to the environment and the replacement of reagents by natural renewable resources [1].

Phosphate is an essential non-renewable mineral resource which is neither replaceable nor recyclable in agriculture. The intensive land use for the rising biodiesel and food production stimulates the long-term growth in world demand for phosphate rock [2,3]. However, phosphate ore deposits can present a complex mineralogy, containing impurities that affect phosphorus floatability in the beneficiation plants. As a result, researches have been conducted in an attempt to improve apatite concentration meeting the standards of the fertilizer industry [2,4,5].

Froth flotation is the most important concentration method for beneficiating phosphate ores [2,3]. In this process, most collectors used worldwide are fatty acids obtained

\* Corresponding author.
E-mail: eng.priscila.oliveira@gmail.com (P. Oliveira).

from vegetable species as byproduct of different industries, *e.g.*, tall oil, a byproduct of the paper industry [4,6]. However, the investigations on alternative sources are being encouraged as paper recycling and reagent price are rising [2]. Vegetable oils are currently the most important alternative sources of fatty acids substituting tall oil as they present lower costs and high efficiency at room temperature [2,3]. In order to be used as collectors, these sources must undergo saponification reaction to liberate the fatty acids from triglycerides molecules, by breaking down the ester bonds, and then neutralize them, producing fatty acids soluble salts [4,6]. Vegetable oils from rice bran, grape seed and soybean are well-established inputs for Brazilian phosphate ore processing plants [3,4,6], although facing competition with human or animal nutrition.

Palm trees are one of the main sources of oils and fats amongst vegetable species, although few of them are exploited. In the Amazon region, there are approximately 150 species and some of them are high-potential vegetable oil sources [7]. Palm trees present the advantage of adapting to underused agricultural lands located in tropical areas where climate and other factors impair the most common oleaginous crops. As a result, they can produce oil from both pulp and seed at high yield, even under adverse conditions and minimal care [8,9].

Pataua palm trees (*Oenocarpus bataua*) are present over a large geographical range in the tropical areas of northern South America, including French Guiana, Peru and Brazilian Amazonian region where its fruits can be harvested as a source of vegetable oil from native forests. Traditionally, pataua oil is obtained from the fruit, that is, pulp (exocarp and mesocarp) and seeds, as a yellow-greenish extract that resembles olive oil, with a high content of fatty acids, mostly unsaturated oleic acid (typically >40%) and others, such as palmitic, stearic, lauric, and myristic acids [7,8,10].

Among several collectors derived from vegetable oils [4,11,12], pataua oil collectors represent a potential alternative for sustainable exploitation of natural forests in tropical areas.

Despite the existence in the literature of studies on bio-based surfactants derived from vegetable oils from Amazonian palm trees used in froth flotation [13–15], no reference was found regarding the use of saponified pataua oil as a collector in the flotation of apatite. This study addresses a comprehensive characterization of pataua oil concerning its fatty acids profile and physicochemical properties and investigates its application as collector in apatite flotation.

## 2. Materials and methods

### 2.1. Pataua oil (PO)

The PO sample was extracted from the fruit pulp, by cold pressing in the absence of solvents, and supplied by Amazon Oil Industry (Brazil). The fatty acids profile was obtained using Gas Chromatography (GC) of fatty acid methyl esters, with FAME C14-C22 (Supelco cat n°18917) as standard for the peak identification. The GC analysis was conducted using a HP7820A Gas Chromatographer (Agilent, U.S.A.), with flame ionizing detector, SGE column, and hydrogen as carrier gas.

Iodine value (IV) was measured directly by Wijs Method, according to ASTM D5554-15. The saponification value (SV) and acidity index (AI) analyses were performed in compliance with ASTM procedures D5558-95 and D5555-95, respectively. All values were also obtained indirectly, based on the fatty acids profile.

The analyses were carried out in order to certify that the studied sample is in agreement with the reported characteristics of PO, such as unsaturations and carbon chain length, that influence its performance as collector in plant practice.

### 2.2. Pataua oil collector (POC)

The PO sample was saponified at 75 °C using a 2% (w/v) sodium hydroxide/ anhydrous ethanol solution under reflux for one hour. At the end of the process, the saponified oil was filtered using a filter paper Quanty® (opening 25 μm), generating a solid (POC) and a residual liquid fraction (mostly ethanol and glycerol). POC was dried at 80 °C for 18 h and stored as powder.

The saponification efficiency was verified by Fourier transform infrared spectroscopy (FTIR). The spectra of both PO and saponification products (POC and residual liquid fraction) were obtained and further compared. Moreover, the POC powder was also assessed to verify the liquid phase evaporation and any alteration on the collector's chemical composition which could have occurred. The FTIR spectra were obtained using a Nicolet 6700 apparatus (Thermo Scientific, U.S.A.) in attenuated total reflectance (ATR) mode, with 64 scans within the 4000-675 cm$^{-1}$ range, at 4 cm$^{-1}$ resolution.

The POC Critical Micelle Concentration (CMC) was obtained applying the Du Nouy ring method in a K10ST tensiometer (Krüss, Germany) to measure the surface tension of collector solutions. Prior to all measurements, the platinum ring was flame treated to remove any organic contamination. In each test, a 20 mL sample of solution was used. All measurements were performed at approximately 21 °C and pH 10.0 (±0.1), which was controlled using dilute NaOH and HCl solutions.

### 2.3. Mineral samples

Apatite, calcite, and quartz samples were supplied by Luiz Menezes Comércio e Exportação de Minerais (Brazil). Their purity was found to be approximately 99, 98 and 97%, respectively, using X-ray powder diffraction (PANanalytical X'Pert, Philips, Netherlands) followed by Rietveld refinement and Fourier transform infrared spectroscopy (Nicolet 6700, Thermo Scientific, U.S.A.) analyses.

### 2.4. Fundamental studies

Microflotation tests were conducted using a modified Hallimond tube, operated with nitrogen flow rate of 60 cm$^3$. min$^{-1}$. The procedure consisted of weighing 1 g of the −300 + 106 μm mineral sample, conditioning it in a 50 mL of concentrate collector solution for 5 min, at the adjusted pH, and completing the volume of the tube with deionized water at the pH selected for the test. Flotation was then carried out for 1 min.

Zeta potential measurements were conducted using Electrophoretic Light Scattering in a NanoBrook 90 Zeta-

Plus analyzer (Brookhaven Instruments Corporation, U.S.A.). Apatite, calcite, and quartz were pulverized to the size range below 38 μm. KCl solution (1 mM) was used as the supporting electrolyte and the pH was modulated with NaOH and HCl solutions. The solutions were prepared with deionized water (resistivity 12,8 mΩ.m). The suspensions were prepared by adding 1 g of mineral in 50 mL of supporting electrolyte, agitated for one hour, settled and filtered (particle retention: 6 μm). The Zeta Potential measurements in the presence of collector were performed after 30 min of agitation, followed by pH adjustment and addition of 2 mg of the reagent. The pH was adjusted prior to every measurement.

Fourier transform infrared spectroscopy (FTIR) was performed to investigate the POC adsorption onto the mineral surfaces. For this purpose, 0.15 g of each mineral was conditioned with 100 mL of 50 mg.L$^{-1}$ collector solution for 5 min. The solid fraction was filtered, gently washed with deionized water and dried at 80 °C for 24 h. Diffuse reflectance mode (DRIFTS) was used to characterize the mineral samples, within the 4000–500 cm$^{-1}$ wavenumber range. For each sample, 64 scans were performed, at a 4 cm$^{-1}$ resolution.

## 3. Results and discussion

### 3.1. Oil characterization

The PO fatty acids profile is presented in Table 1. The sample profile resembles that of a typical pataua oil, according to American Oil Chemist's Society (AOCS), and the differences observed in acids composition might be due to natural variations in geographic origin, age of the fruits, and weather conditions, for example, temperature [16]. The total content of species with strong collecting capacity, oleic C18:1 and linoleic C18:2, is comparable to that of collectors obtained from vegetable oils currently used in flotation practice, e.g., tall oil, soybean and rice bran oil [4,6]. Moreover, the predominance of oleic acid over linoleic acid has been shown to improve the collector's performance in phosphate ore flotation [6].

Values of relevant chemical parameters of PO are listed in Table 2. It can be noted a good agreement of iodine and saponification indexes measured by direct and indirect methods and those supplied by Amazon oil and found in the literature. The acidity index is an important standard for the food industry as it should be significantly low for human consumption. In flotation systems, however, high values of acidity index favor the application of PO as a collector. This has been explained by the catalyst role played by the fatty acids salts produced early in the saponification process [17,18].

### 3.2. Saponification reaction

The FTIR spectra obtained for PO and saponification products are presented in Fig. 1, showing the most relevant bands associated with the hydrocarbon chains of fatty acids, carboxylate, and alcohols (ethanol and glycerol). In the spectrum of the solid product (1b), the characteristic bands of triglycerides and free fatty acids (1744 and 1711 cm$^{-1}$, respectively) were substituted by a band at 1558 cm$^{-1}$, characteristic of sodium salts of fatty acids [19], indicating complete, or nearly complete,
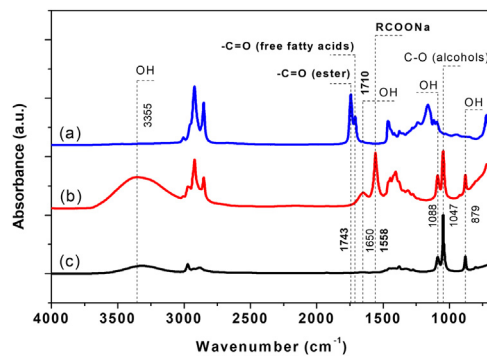


**Fig. 1 – Spectra of PO and saponification products: (a) PO, (b) POC (c) Residual liquid.**

saponification. The bands associated with alcohols are present in both products, as the saponification reaction and the filtration were processed in alcoholic medium (Table 3). The bands observed were compared with previous studies [20–24].

Significant changes were not observed in the solid product after drying, except for the evaporation of volatiles (residual alcohols from saponification and filtration processes).

### 3.3. Critical micelle concentration

The results of surface tension measurements at different POC concentrations, at pH 10, are illustrated in Fig. 2. As indicated, the reagent presents strong tensoactive action, causing significant decrease in the surface tension. The values achieved for pre-micelle and critical micelle concentrations were, respectively, 21 and 53 mg.L$^{-1}$. In this range, the collector species form hemi-micelles [3], which contribute to the relative immobilization of the collector species onto the mineral surface and favors particle collection [25]. The undesirable formation of micelles starts only at 53 mg.L$^{-1}$.

Depending on the tests pH and temperature, a collector might present some variation in the CMC value. CMC values as high 127 and 300 mg.L$^{-1}$ have been reported for pure sodium oleate [3,26]. The significant difference between the reported values for oleate and that found for POC may be attributed to higher packing degree of the former in comparison to the latter. In fact, POC is a mixture of fatty acids sodium salts with different lengths of hydrocarbon chains, which can impair the pairing of two adjacent molecules due to the extra vibrational energy created by the thermal movement of the segment from the largest chain [26].

### 3.4. Microflotation

The apatite, calcite, and quartz microflotation results as a function of POC concentration and pH are presented in Fig. 3. Regarding collector concentration, it was observed high selectivity between apatite and quartz, but the selectivity between apatite and calcite, especially at low and even moderate concentrations, was significantly higher than the previous values reported in the literature [27,28]. Increasing POC concentration slightly enhanced the floatability of calcite, a trend consistent

| Table 1 – Fatty acids profile of pataua oil (%). | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Fatty acid | C12:0 | C14:0 | C16:0 | C16:1 | C18:0 | C18:1 | C18:2 | C18:3 | Other |
| | 2.9 | 0.9 | 12.3 | 3.0 | 3.2 | 60.6 | 12.4 | 0.7 | 4.0 |
| AOCS [16] | | | 9.0 | | 6.0 | 81.0 | 4.0 | | |

| Table 2 – Chemical parameters of pataua oil. | | | | |
|---|---|---|---|---|
| Chemical parameter | Direct method | Indirect method | Amazon Oil | AOCS [16] |
| Iodine Value (g $I_2$. $g^{-1}$) | $76.7 \pm 1.7$ | 78.3 | 75-78 | 75 -80 |
| Saponification Value (mg KOH. $g^{-1}$) | $190.3 \pm 4.3$ | 194.4 | 192 – 209 | 190 – 196 |
| Acidity Index (mg KOH. $g^{-1}$) | $43.2 \pm 0.3$ | | < 20,0 | |

| Table 3 – FTIR bands associated with the spectra of PO and saponification products. | | | |
|---|---|---|---|
| Nominal frequency ($cm^{-1}$) | Functional group | Vibration mode | Range ($cm^{-1}$) |
| 3355 | OH (glycerol) | | 3700–3000 |
| 2971 | —C—H (ethanol) | Stretching | 2971 |
| 1315 | C—O (RCOOH) | Stretching | 1310–1312 |
| | OH (alcohols) | Bending | |
| 1090 | C—O (alcohols) | Stretching | 1080–1086 |
| 1047 | C—O (alcohols) | Stretching | 1045–1048 |
| | C—O (polyalcohols) | | 1050 |
| 879 | OH (ethanol) | Out of planeBending | 877 |
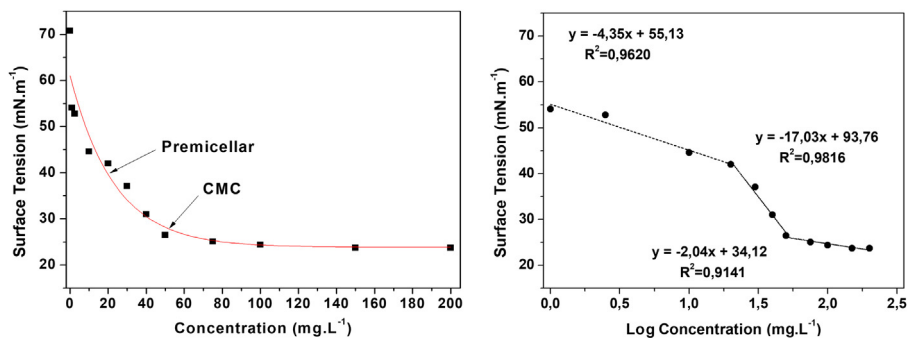


Fig. 2 – Surface tension as a function of POC concentration (A) and log of POC concentration (B), both at pH 10.
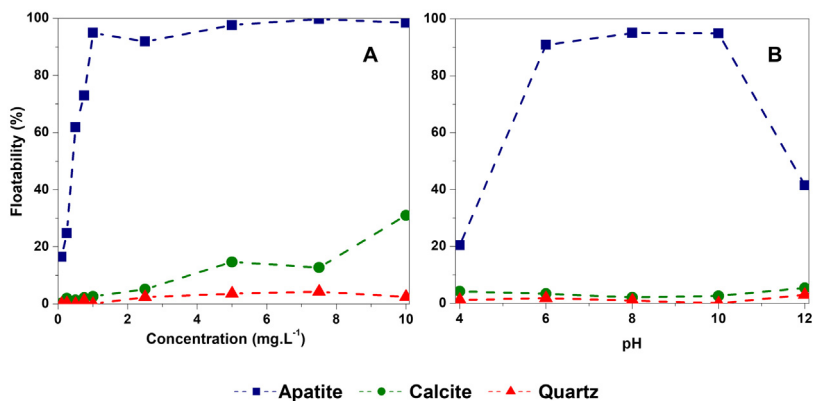


Fig. 3 – Effects of POC concentration at pH 10 (A) and pH (B) on apatite, calcite and quartz floatability.

with the findings of Pugh and Stenius [28] using pure sodium oleate.

The present investigation demanded lower POC concentrations for maximum floatability of apatite than those required with pure sodium oleate from a previous study [28]. This reduction may be also related to the different packing degrees of oleate and POC molecules onto the minerals surfaces.

The maximum floatability of apatite using POC was achieved in the pH range from 6 to 10 for $1 \, mg.L^{-1}$ ($0.3 \, mg.g^{-1}$) collector concentration. Brandão et al. [4] observed similar trends for different fatty acids salts individually, with maximum floatability at pH 7.0. In their work, linoleic acid was the only species which extended its action to pH 10.

Cao et al. [3] studied a mixed collector system for fluorapatite flotation and also observed increased hydrophobicity within the pH range from 3 to 9. Above this range, the contact angle decreased as a response to the collector speciation in the system. According to Vučinić et al. [29] and Cao et al. [3], in the acidic range, the molecular species are predominant, which promotes weak physical adsorption of the collector onto the mineral. In the slightly alkaline medium, the ionic species and the ionomolecular complex are both present, resulting in significantly increased hydrophobicity. Over pH 10, the dimeric specie becomes dominant and its adsorption results in the above mentioned decrease in hydrophobicity as the configuration of the polar groups, oriented towards the solution, exposes hydrophilic groups.

It was not observed significant changes in calcite floatability as a function of pH at $1 \, mg.L^{-1}$ POC concentration. As higher collector concentrations are required to achieve calcite flotation, the pH effect could have been hidden. No influence of pH in quartz floatability was noticed, which is expected as the chemisorption mechanism would require Ca sites at the mineral surface. Physical adsorption would be the main plausible mechanism, but in the pH range investigated both the mineral surface and the ionized collector present negative charge, preventing the collector adsorption.

### 3.5. Zeta potential

Fig. 4 presents zeta potential results for the collector and for apatite, calcite, and quartz, conditioned with collector and in its absence, in the pH range from 4 to 12. POC zeta potential (curve 3 on both sides) suggests that the isoelectric point of the reagent occurs at pH approximately 4, overlapping with the $pK_a$ of fatty acids. The IEP refers to colloidal species which, in the case of fatty acids, occur in acidic solutions, when the solubility limit is exceeded. According to Laskowski [30], these species, in opposition to micelles, present distinct isoelectric points in the acidic pH range. At higher pH values, the ionization of the collector causes the more negative zeta potential values.

The isoelectric point (IEP) of the apatite particles occurs at pH 6.5 (Curve A1). Depending on the composition, apatites may exhibit different electrokinetic behaviors, due to the presence of impurities in the crystalline lattice as a result of ionic substitution, with the IEP varying from 4.0 to 7.4 [3,31–34]. According to Vučinić et al. [29], below the IEP the chemisorption of $H^+$ and $CaH_2PO_4^-$ is accountable for the positive surface

charge of the mineral, while above the IEP, $OH^-$ and $HPO_4^{2-}$ adsorb onto positive surface sites ($Ca^+$ and $HPO_4Ca^+$).

The apatite zeta potential, after conditioning with POC (Curve A2), shows more negative values and a shift of the IEP to a lower pH, in agreement with results from Kou et al. [35]. This behavior was expected, considering that the negatively charged ionized collector adsorbed onto positive calcium sites at the mineral surface.

The zeta potential results for calcite conditioned with collector (Curve B2) in the intermediate pH range were not more negative. This might be due to the fact that the adsorption was too low to cause significant changes, which is corroborated by the negligible floatability of calcite.

The quartz zeta potential results, in the absence of collector (Curve C1), were in agreement with values reported in the literature [36]. The behavior of the mineral conditioned with collector (Curve C2) is similar to that in the absence of collector, which is expected as the mineral presents a significantly negative zeta potential in the pH range investigated and also because no significant floatability was observed, indicating a lower adsorption than that of apatite.

### 3.6. Fourier transform infrared spectroscopy (FTIR)

Figs. 5 and 6 illustrate minerals' FTIR spectra prior to or after conditioning with collector at different pH values. The region between 3100 and $2800 \, cm^{-1}$ is typical of hydrocarbon chain and the region between 1750 and $1400 \, cm^{-1}$ refers to the carboxylate group [35].

The peaks relative to vibrations of the methyl and methylene groups, present in hydrocarbon chains, are located at 2922 and 2852 ($CH_2$), and $2958 \, cm^{-1}$ ($CH_3$) [35,37–39], as observed in Fig. 5. Nevertheless, the significant difference between the intensity of peaks in the apatite spectrum and in the calcite and quartz spectra could reflect the selectivity achieved in the microflotation experiments.

Cis unsaturations are indicated by the peak at $3005 \, cm^{-1}$ [37,38] and can be distinguishable from their geometric isomers trans which have a peak at $3025 \, cm^{-1}$ [37]. The cis bonds are present in the chains of oleate and linoleate molecules from natural oils, which represent a significant fraction of the collector. The disappearance of the peak at $3005 \, cm^{-1}$ in the spectra is associated with the interaction collector-mineral and indicates auto-oxidation of the unsaturations, according to Brandao [40].

Upon the interaction between mineral surface and collector, the spectra show a small shift of the hydrocarbon chains bonds with respect to the spectrum of the mineral at low wavenumbers. According to Lu and Miller [41], this type of shift is typical of increased interaction of hydrocarbon chains, reflecting a poorer degree of collector molecules packing on the mineral surface than as pure dried collector. Adsorbed collector species present extra vibration energy due to different sizes and configurations of hydrocarbon chains, which impairs the interaction among them. In the dried state, on the other hand, the fatty acids salts tend to assume two-layer structures, with the hydrophobic group at one side and the hydrophilic groups at the other, resulting in stronger interaction with decrease in vibration energy.
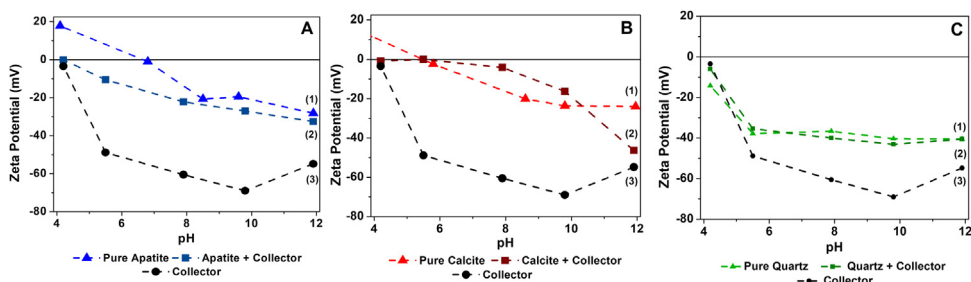
Fig. 4 – Zeta potential of apatite (A), calcite (B) and quartz (C) as a function of pH.
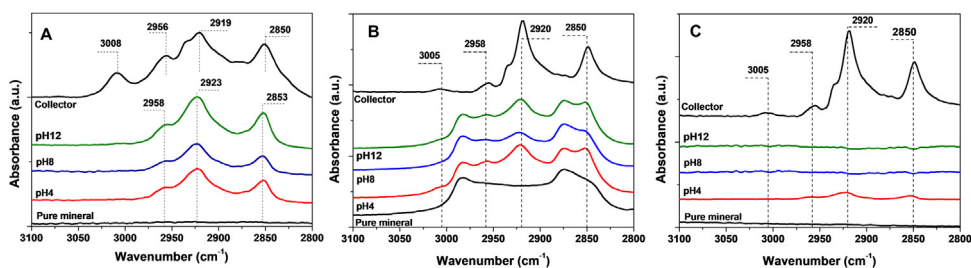


Fig. 5 – Hydrocarbon chain region in the infrared spectra, for the mineral samples prior to and after conditioning with collector at different pH values: A - Apatite; B - Calcite; C - Quartz.
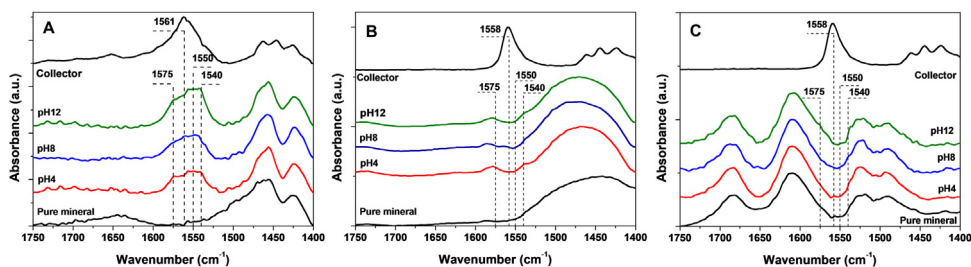


Fig. 6 – Carboxylate group region in the infrared spectra, for the mineral samples prior to and after conditioning with collector at different pH values: A - Apatite; B - Quartz; C - Calcite.

Regarding the effect of pH, the spectrum of apatite indicates that the intensity of the peaks is lower at pH 4 as a result to the low degree of ionization of the collector, which at higher pH values is capable of chemisorption. On the other hand, the peaks of adsorption on quartz are more intense at low pH values, close to the IEP, where the electrostatic repulsion is less significant and physical adsorption is likely to occur.

Fig. 6 shows typical carboxylate absorption peaks at 1575, 1557 and 1540 cm$^{-1}$ which are related to the collector adsorption onto the surface of the calcium-bearing minerals. In this type of system, the carboxylate groups form complex with the metallic ion, mostly by bridging the oxygen atoms from the functional group and the calcium ions at the mineral surface. This may involve one or two calcium atoms and is limited to a monolayer. In the FTIR spectrum, these interactions are identified by an absorption peak centered at approximately 1550 cm$^{-1}$ [35,41–43]. It should be noted that, although the samples analyzed using microflotation and FTIR were conditioned with POC for much shorter time (5 min) than required to achieve equilibrium, the surface speciation of hydroxyapatite and fluorapatite [44] indicates the formation of calcium hydroxy complexes at alkaline pH, reducing the sites available

for collector adsorption, which agrees with the lower floatability of apatite.

Adsorption may also take place when calcium dicarboxylate salt forms in solution and then precipitates onto the mineral surface. This is characterized by peaks at 1575 and 1540 cm$^{-1}$ (Fig. 6). The presence of these two peaks indicates the existence of two coordination types which may occur in the tridimensional carboxylate-calcium complexes: the monodentate mode, with the asymmetric vibration at 1575 cm$^{-1}$; and the bidentate mode, corresponding to the vibration at 1540 cm$^{-1}$ [35,41,43]. The formation of CaOH at alkaline pH [45] can also impair the precipitation of calcium dicarboxylate salt, reducing apatite floatability, as observed in the microflotation results.

## 4.    Conclusions

The composition of POC, with fatty acids of different carbon chains, causes the molecules to present a poorer packing degree than that of reagents composed of one carbon chain type, as pure sodium oleate, resulting in interfacial satura-

tion at lower concentration values. This behavior could be the reason for the lower observed values of CMC and collector concentration demanded for apatite maximum floatability.

Microflotation tests showed that the collector was selective within the neutral and alkaline pH range. Above pH 10.0, the likely adsorption of dimeric species is expected to have caused a decrease in apatite floatability. Yet, quartz and calcite floatabilities were not affected by the pH for the test conditions investigated.

POC adsorption onto apatite was verified for all the analyzed pH values, being chemisorption and insoluble calcium salts precipitation indicated as the main mechanisms. Zeta potential and FTIR showed that the collector adsorption onto calcite and quartz was not significant.

Pataua oil, as many other palm tree species, has no industrial application until now, despite its high availability and good adaptation to underutilized agricultural lands where climate and other factors impair the cultivation of more common oleaginous species. In this sense, pataua oil shows great potential to be used as a source for flotation reagents, especially in substitution for traditional oils.

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