Characterization of sludge from industries for possible recovery in Arts

Caracterización de lodos de industrias para un posible aprovechamiento en artes

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Abstract

Sludge from oxidation ponds from different industries was evaluated to study its composition through a chemical characterization and to determine a possible route for its recovery. For the characterization of the sludge, gravimetry for SiO2 content, infrared spectroscopy, X-ray diffraction, thermogravimetric analysis, X-ray energy dispersion spectrometry (EDS), and X-ray fluorescence (XRF) were used. It was determined that the paint industry sludges have a higher SiO2 content with 17.2 ± 0.2 % by mass and different phases present in the sludges such as albite, quartz, and rutile were identified, as well as metals of interest in pigments such as iron, chrome, and zinc. The previous demonstrates that even though the matrix is highly complex; there are routes for the use of this residue.

Keywords: Art, materials, pigments, sludge, wastewater.

Resumen

Se evaluaron lodos de lagunas de oxidación, provenientes de diferentes industrias con la finalidad de estudiar su composición mediante una caracterización química, así como determinar una posible ruta para su valorización. Para la caracterización de los lodos se utilizó gravimetría para el contenido de SiO2, espectroscopía infrarroja, difracción de rayos X, análisis termogravimétrico, espectrometría de dispersión de energía de rayos X (EDS) y fluorescencia de rayos X (XRF). Se determinó que los lodos provenientes de la industria de pinturas poseen un mayor contenido de SiO2 con un 17,2±0,2 % en masa y se identificaron diferentes fases presentes en el lodo como albita, cuarzo y rutilo, además de metales de interés en pigmentos como hierro, cromo y zinc. Mostrando que a pesar de que la matriz es altamente compleja existen rutas de aprovechamiento de este residuo.

Palabras clave: Arte, agua residual, lodos, materiales, pigmentos.

Introduction

The Facultad de Bellas Artes de la Universidad de Costa Rica (Faculty of Fine Arts of the University of Costa Rica) seeks to promote art, that is, the ability to express beauty through painting, sculpture, and music (Real Academia Española, 2021). This entity together with the Escuela de Bellas Artes (School of Fine Arts) has a genuine interest in contributing to professional training, and the generation and dissemination of knowledge in the area of the arts since 1897 to benefit the development of social protection and recognition of the diversity of cultural heritage from their undergraduate and postgraduate courses (Facultad de Artes, 2023). All this while raising awareness about environmental responsibility and promoting a policy for the use and reuse of waste.

The waste that will be used and reused as raw material in art is the sludge that is generated in the wastewater treatment plants (PTAR) as a product of the conditioning of the water for its discharge (Zhu et al., 2020). The oxidation ponds in WWTPs are the most used option for water treatment (Metcalf et al., 1991). At the bottom of these lagoons, the large amount of sludge that is generated must be extracted regularly so it can be used in various applications.

Sludge is divided into primary sludge and secondary sludge. Primary sludges usually contain a lot of organic matter and high activity of microorganisms (Kwarciak-Kozłowska, 2019), while secondary sludges are found in oxidation ponds with little organic load and high contents of water, silicates, clays, carbonates, and metals (Canziani & Spinosa, 2019). The latter is of great interest because of their chemical composition of some pigments, glass, ceramics, and enamels. (Esteves et al., 2010).

In recent years, different initiatives for the use of sludge have been launched for energy use (G. Yang et al., 2015; Yi et al., 2014); as an addition to soils to improve their quality (Kwarciak-Kozłowska, 2019; Passuello et al., 2012); as an incorporation into construction materials as reinforcement, (Camareno et al., 2006) and even to produce higher value materials such as zeolites (Carranza & Montero-Villalobos, 2016).

However, it is established that the applicability of the sludge from the industries in Fine Arts, from now is only called Arts; can be in painting and sculpture. The possibility of its use in music is excluded. Secondary sludge is the most viable for this purpose. In this sense, there is the use of mud in the work of Guy Riefler when extracting pigments from the mud of the rivers of the coal mines in southeastern Ohio, United States. With it, John Sabrow gave color to his artistic works (Balmer, 2015). Likewise, obtaining ceramic pigments spinel of $Ni(Cr^{3+}, Fe^{3+})_2O_4$ by Carneiro et al., (2018) using red mud, the use of yellow waste sludge with high iron oxide contents to prepare ceramic glaze pigments by Ovčačíková et al., (2021). Obtaining glass-ceramics from urban and industrial wastewater sludge in Egypt by Garcia-Valles et al., (2011) and the use of sludge from alumina extraction to manufacture glass and glass-ceramics (J. Yang et al., 2008) are some examples of the use of sludge in arts.

However, the applicability of the sludge obtained from the WWTPs depends on their composition and physicochemical characteristics, which in turn, depend on the origin of the wastewater from the processes. Since Costa Rica, which has an economy mainly dual, combines the export area with the non-export sector (OECD, 2020), both sides of the Costa Rican economy were chosen; being paint and metallurgical industry that correspond to the first group and agricultural industry of the SME sector, to identify which of these muds could be applied in artistic expression either in sculpture or painting.

This article proposes to study the physicochemical characteristics of an environmental residue, such as the sludge from some Costa Rican industries to propose it for non-conventional artistic applications and mark a first step so that in the future the School of Arts will be self-sufficient in producing basic supplies for its courses in a sustainable way over time with environmental awareness.

Methology

Sample collection: Secondary sludge samples from lagoons or oxidation tanks of three Costa Rican industries were collected to determine their applicability in the artistic area. Companies that could potentially have a high percentage of SiO_2 were selected, this compound being of great added value in the vitrification or enameling process, for this reason, paint, agricultural and metallurgical industries were selected.

Humidity determination: The humidity of all the collected sludge was determined through gravimetry. For this, the wet sludge was placed in an oven at 100 °C for a period of 12 h.

Silicon Dioxide Percentage: The quantification of silicon dioxide was carried out on a dry basis by gravimetry in the Laboratory of Analytical Services (LASA), of the Escuela de Química (School of Chemistry) of the University of Costa Rica, following their own established the methodology.

Infrared spectroscopy: To obtain the bands of the functional groups associated with the silicates, carbonates, and oxides present in the sludge sample, a PerkinElmer Frontier FT-IR-ATR model was used. Ten (10) scans per sample were made with a large background to obtain the final spectrum. The scan was from 4000 cm^{-1} to 450 cm^{-1}.

X-ray diffraction: A Brucker D8 Advance brand diffractometer with a LynxEye detector and a scan from 5° to 60° was used to identify the crystalline phases of the components present in the sludge and thus determine the ease of their separation/recovery.

Thermogravimetric analysis: A TGA equipment, TA Instruments brand, model Q5000, with an inert atmosphere was used using a heating ramp of 5 °C/min from 25 °C to 600 °C, to study the behavior of the sludge concerning temperature and identify volatile compounds and degradations associated with silicates, oxides, and carbonates of the mud.

X-ray energy dispersion spectrometry (EDS) and X-ray fluorescence (XRF): To determine the elemental composition, a HITACHI model S3700-N scanning electron microscope was used, where the X-ray fluorescence technique was used (XRF) to detect the heavier metals and X-ray dispersion spectrometry (EDS) to quantify the lighter metals. The dried mud powders were placed on a double carbon tape attached to a sample holder that was inserted into the equipment and then a vacuum was applied to carry out the measurements.

Results and discussion

The collection of sludge from lagoons and oxidation tanks from different industries was carried out according to Table 1 to determine the percentage of SiO_2; thus, to know the potential that they would have in the process of vitrification, ceramics, or sculpture at an artistic level.

The presence of SiO_2 is important within the structure of vitrified ones because as it increases, the vitrified phase increases, and in heavy metal-problem muds, polymerization of the silicate contributes to the chemical stability of the metals in the matrix (Kuo et al., 2003, 2008).

In contrast, there are three categories of modern ceramics: glass, glass ceramics, and engineering ceramics. Traditional ceramics fall under this last group and includes everyday ceramics, sanitary and construction ceramics, ceramics for arts and crafts, chemical ceramics, electrical ceramics, and more. All of the aforementioned are primarily made of natural silicates such as clay, quartz, feldspar, or kaolin, where the SiO2 content is crucial to the characteristics and final appearance of the ceramic (Ion, 2005; Zeng et al., 2021).

Sludge can be used as an addition to regular concrete cement for sculpture or it can be used to change the forge or porosity of concrete mosaics (Armbruster, 2019; Camareno et al., 2006). The humidity in the sludge was also measured, which was crucial because the processes of transport and dehydration or drying require a lot of energy (Zhen et al., 2012). In this regard, the more moisture there is in the sludge, the more challenging it will be to adapt it for reuse and use in other applications. According to Table 1, the agrochemical business, followed by the paint industry, and the metal industry, which has the least amount of water in the sample, are the ones with the highest water content. This information helps characterize the sludge and determine its potential for reuse because the high moisture content in any material complicates its transportation. After all, the right packaging and container must be used. It is also important to consider that a drying process requires a lot of energy, which increases costs and makes the process more expensive.

The difference in moisture percentages is primarily attributed to the sample collection method because the shape of the lagoon or oxidation tank influences the ease of sample collection and, as a result, the amount of water that is collected with it. Although these amounts are significant, secondary sludges with moisture contents of up to 99% have been reported (Canziani & Spinosa, 2019).

As it can be seen within the previous table, the paint industry is the one with the most elevated rate of silicon dioxide on a dry basis, making the paint company the one with the most noteworthy conceivable outcomes to

Table 1. Moisture percentage and silicon dioxide percentage of three sludge samples from different industrial processes

Industry	Moisture Percentage (%)	SiO2 Percentage (%)
Paints	26.5 ± 0.5	17.2 ± 0.2
Agrochemicals	30.5 ± 0.5	2.7 ± 0.2
Metals	14.3 ± 0.5	0.5 ± 0.2

Source: Prepared by the authors

be utilized in applications such as design or vitrification. For this reason, it is that the slime from the paint industry was chosen for the taking after characterization, in such a way that its composition can be known and proposed for pigments, paints or the like after appropriate treatments.

Two amassments of sludge from the paint industry were then carried out, one in the low engendering season and the other in the high engendering season, both were dried, and the percentage of inorganic matter in them after drying was resolute.

In this case, it is carried out in the same way on a dry basis and it is denoted that both batches have a similar amount of inorganic matter, the one obtained in the low season being greater according to Table 2.

Oxidation lagoons work to improve the microbial quality of waters and decrease the oxygen requirement (BOD) by the time they reach receiving waters; therefore, lagoon design has traditionally focused on BOD removal, although oxidation ponds important secondary functions of sedimentation of organic and inorganic solids that carry the process waters (Davies-Colley et al., 1995; Verbyla, 2016). When using the secondary sludge obtained from the oxidation lagoons, the use of inorganic material is present. That is the reason why when the season is low, the water flow is lower and the industries, respecting the water retention time to reduce the BOD, require more time collecting wastewater. As a consequence, they also store a greater amount of inorganic solids such as clays, silicates, carbonates, etc. (Metcalf et al., 1991).

However, it is important to note that the difference between the data reported in Table 2 is very small, so it could be considered that the inorganic matter in the paint sludge samples is independent of the production season. It should be noted that, since these are stable products over time, the intention is to recover the inorganic compounds since the organic compounds are mostly degraded due to coming from an oxidation lagoon.

Table 2. Percentage of inorganic matter in the selected sample.

Painting Company	Percentage of inorganic matter (%)
Lot 1 (Low season)	50.5 ± 0.1
Lot 2 (High season)	49.5 ± 0.1

Source: Prepared by the authors.

Next, the SiO_2 quantification process is perferomed because the inorganic matter is not only silicon but, it also contains other oxides such as TiO_2, CaO, K_2O, and Al_2O_3, among others, in their different phases. To do this, % SiO_2 is obtained separately for the batches and the entire homogenized sample. The results are shown in Table 3.

Table 3. Percentage of silicon dioxide of the selected sample.

Painting Company	SiO_2 (%)
Lot 1 (Low season)	43 ± 1
Lot 2 (High season)	50 ± 3
Homogenized sample 1	44 ± 3
Homogenized sample 2	37 ±1

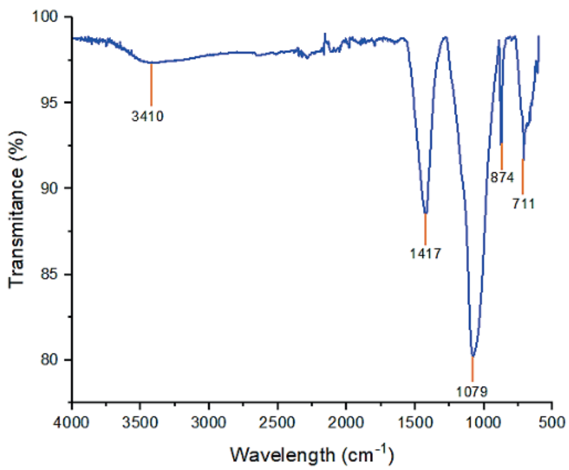
Source: Prepared by the authors.

The percentage of SiO_2 is an important characteristic in calcined sludge (without organic material) since a considerable increase is shown for the sludge that contains organic matter, thus going from 17.2 ± 0.2 to the data reported in Table 3.

That $SiO_{2,}$ in the paint industry specifically, is used as fillers in the formulation which are added in powder and work to generate body and volume in paint, change properties such as viscosity, and its final shine when it is dry. (Carbonell, 2011, 2014) Therefore, it is natural that in peak production season there is a higher amount of this material. Also, as mentioned above, the SiO_2 content is not only used in paints as it is also important when used in vitrified or ceramics and that generates a wide range of possibilities for using this residue by eventually recovering it.

Regarding the characterization of the sludge, the Infrared spectrum of the sample without organic matter is shown in Figure 1, in which bands associated with the crystalline phases shown in Table 4 can be observed.

Figure 1. The infrared spectrum of the inorganic matter of the sludge.



Source: Prepared by the authors.

The band, 3410 cm^{-1} would correspond to water occluded within the sample, and the band at 1079 cm^{-1} to the stretching of the Si-O bonds. While the bands 1417 cm^{-1}, 874 cm^{-1}, and 711 cm^{-1} correspond to the presence of carbonates (Morales Acevedo & Pérez Sánchez, 2003).

Although these bands are the most notorious, it is important to highlight that there are other functional groups in the matrix whose bands are superimposed. The band at 796 cm^{-1} corresponding to Si-O in the $CaCO_3/SiO_2$ (Reig et al., 2002) or TiO/SiO_2 mixtures, in addition to the band at 960 cm^{-1} corresponding to Ti^{4+} that can be substituted with Si^{4+} in the compound (Ren et al., 2008) it is not shown, the same happens with the bands at 960 cm^{-1} and 1060 cm^{-1} corresponding to the Al-O bonds of the albite (Feng et al., 2012).

Likewise, X-ray diffraction was used to determine the phases of the main components obtained in it the values of Table 4.

Table 4. Crystalline phases detected by X-ray diffraction

Crystalline phase	Compound
Calcite	$CaCO_3$
Albite	$NaAlSiO_8$
Quartz	SiO_2
Rutile	TiO_2
Hematite	Fe_2O_3
Titanium and silicon oxide	O_4SiTi

Source: Prepared by the authors.

It is observed that the sludges are composed of various crystalline phases, this makes sense considering that they were extracted from oxidation ponds where mostly clays are found that are chemically a combination of aluminum and silicon with the general formula $Al_2O_3{\cdot}2SiO_2{\cdot}2H_2O$ and sometimes found as feldspar ($K_2O{\cdot}Al_2O_3{\cdot}6SiO_2$ or $Na_2O{\cdot}Al_2O_3{\cdot}6SiO_2$), silicates in the form of quartz with the formula SiO_2, carbonates CO_3^{2-} and various metals (Evans & Langdon, 1976; Zeng et al., 2021).

However, according to the thermogravimetric analysis shown in Table 5, there is a first degradation corresponding to the water occluded within the crystalline network, which was visualized in the infrared spectrum of Figure 1 in the 3410 cm^{-1} band. On the other hand, it has been reported that the degradations between 600-750°C correspond to the dehydroxylation of Ca $(OH)_2$, while at 750-850°C they are due to the decomposition of carbonates into CaO and CO_2, this due to the amount of calcium contained in the sludge in the form of calcite (Rodríguez et al., 2012; Segura Sierpes et al., 2016).

As previously mentioned, in Costa Rica the final disposal of Sludge is regulated by current legislation; therefore, it is essential to ensure that the chemical composition of the sludge complies with parameters established by law. For this, the complementary techniques of X-ray energy dispersion spectrometry (EDS) and X-ray fluorescence

Table 5. Study of degradation with temperature, through a thermogravimetric analysis.

Temperature	Percentage of loss (%)	Identity
0-200 °C	5.8	Occluded wáter
600-750 °C	10.8	Dehydroxylation $(CaOH)_2$
750-850 °C	0.1	CO_3 decomposition

Source: Prepared by the authors.

Table 6. Percentage of the concentration of each component present in the sludge

Element	Technique	
	EDS (%)	XRF (%)
C	4.70±0.20	9.9±1.7
O	43±4	32.0±2.0
Na	0.70±0.20	0.0±0.0
Mg	1.05±0.13	0.18±0.05
Al	8.51±1.4	3.49±0.06
Si	11.5±1.1	6.08±0.08
S	0.33±0.01	0.24±0.02
K	0.408±0.001	0.33±0.03
Ca	NA	15.86±0.32
Ti	13.2±2.7	23.73±0.59
Cr	0.74±0.08	0.60±0.06
Fe	1.88±0.54	4.58±0.26
Cu	NA	0.05±0.01
Zn	NA	2.85±0.12

Source: Prepared by the authors

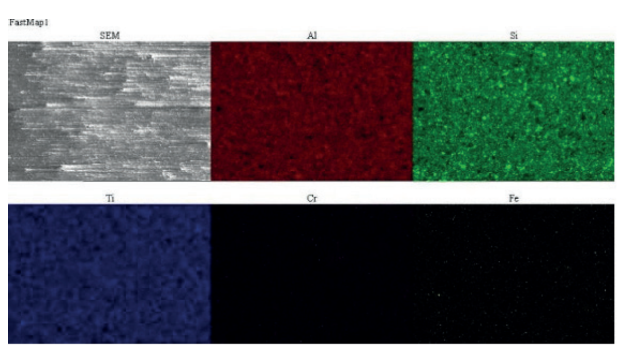
(XRF) are used. The first is used to determine the number of light metals in the sample while the second is used to quantify the heavier metals.

Results are shown in Table 6, it is important to emphasize that in the XRF technique, when visualizing only the heaviest fraction, the percentage calculations are made according to the quantity and sensitivity of elements that can be visualized, hence the difference in the data for each frame.

In this instance, Table 6 demonstrates that only Chromium (Cr) might be detrimental if oxidized among the components found by EDS; nevertheless, the concentration is not as high as that demonstrated in the majority of elements, among which titanium, aluminum, and calcium are also present in addition to silicon. Copper and zinc are present in trace amounts when the elemental composition is examined using the XRF technique, particularly copper, which has a value of 0.05 to 0.01%.

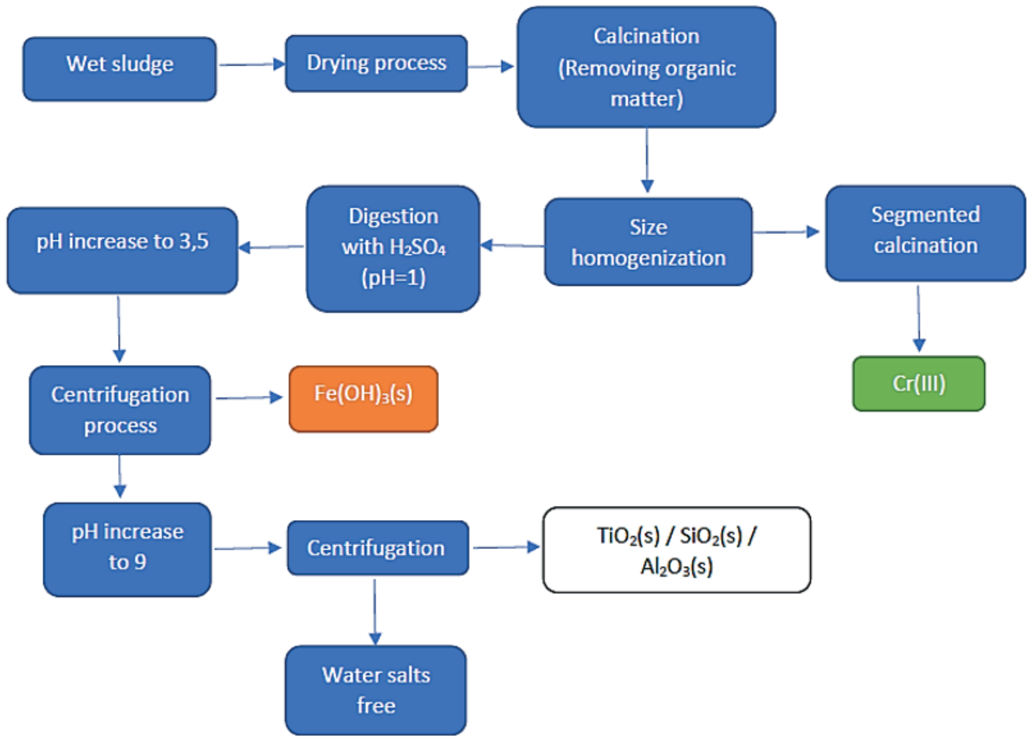
It is also known from mapping the sample that the distribution is homogeneous for each of the metals with the largest percentage of importance, which is crucial when carrying out recovery methods and purification of the sludge components to enable their reuse. A homogenization process had to be performed on each sample collected to have a sample as a whole and verify whether the sample had a homogeneous content or if it was segmented, depending on the level of homogenization performed. This homogeneity is shown in Figure 2. It is important to note that several collections were made at different times to obtain the final sample.

Figure 2. Mapping of metals by EDS



Source: Prepared by the authors

Figure 3. Block diagram of the sludge recovery and separation process.



Source: Prepared by the authors

Since the calcined sludge does not contain enough SiO_2 to adequately vitrify and melt, it is not advised to use it neat for vitrification applications based on the results of the aforementioned characterizations, which reveal a very complicated composition inside the mud matrix. As it contains significant levels of titanium and silicon oxide as well as albite, it might be utilized in items like ceramics or enamels as an alternative (Carter & Norton, 2013). Or even the potential utility of each component if the right acquisition strategy is used. Following the primary goal of developing a long-term sustainable path for the School of Arts where there is a raw material that may be used in numerous procedures.

For the sludge used and characterized, the scheme of the possible recovery process is shown in Figure 3

When compared to the phases indicated in Table 4, the data shown in tables and Figure 2 make sense. Due to its low cost, non-toxic nature, and high refractive index, titanium oxide has been a well-known pigment since the 19th century. Its use expanded after the prohibition on lead pigment since it functions well as a white pigment and is added to other colors as a filler in paint formulation (Chen et al., 2022). Additionally, as Ti is more expensive than other metals and since it makes up a bigger fraction of the sludge than other metals, extraction and purifying techniques can be suggested to reuse it as a pigment or a burden. For this, digestion with sulfuric acid and subsequent centrifugation can be used (See Figure 3) (Khezri et al., 2013), which will produce a concentration of TiO2 in the mixing matrix.

However, digestion with sulfuric acid not only increases the concentration of Ti but also that of Al, which is another of the elements that sludge contains the most as albite. Since. according to literature, the best conditions for extraction of Al from sludge is using a pH of 1 using sulfuric acid or a pH of 13 using NaOH (Truong et al., 2021; Van Truong & Kim, 2022), it is important to mention that the extraction with sulfuric acid will be functional to concentrate both Ti and Al, although an increase in the concentration of metals such as Mg will also be shown.

Regarding the Ca present in the sludge, it is found as calcite according to Table 4. This compound does not need to be purified or extracted since $CaCO_3$ is used by itself as a pigment (Learner et al., 2007) or within the matrix as a catalyst to carry out reactions at high temperatures to obtain other pigments, for example, derivatives of Cr (Kwon et al., 2018; Long et al., 2021). Iron, chromium, and zinc, which are used to make pigments, are among the elements of aesthetic relevance that EDS and XRF also reveal in addition to the aforementioned metals (Correia et al., 2007).

Iron can be recovered by different methods using digestions with either hydrochloric acid, ammonium oxalate, or nitric acid (Rasmussen et al., 1996; B. Yang et al., 2021), whereupon it dissolves and is later precipitated as an oxide or forms part of phosphate, in terms of use in arts it is best to use it as oxide due to its reddish coloration (Correia et al., 2007).

Chromium on the other hand can be recovered by similar digestion methods; however, highly toxic soluble chromates or dichromates would be produced. Therefore, an alternative is a solid phase recovery at high temperature (≈1000 °C) using a carbonate as catalyst (sodium or calcium carbonate) and an oxygen-rich atmosphere, in such a way that sodium or calcium chromate is obtained (Long et al., 2021) this alternative is useful in sludges such as those treated in this article that they contain calcite in their matrix since the catalyst must not be added externally.

On the other hand, zinc (in the form of ZnO) is found in the sludge because it was once used as a white pigment instead of TiO_2, for example in the works of Pier Paolo Pasolini, where no traces of TiO_2 were found (Learner et al., 2007). In the sludges analyzed, the amount of Zn is found to a lesser extent than Fe, this element can be extracted in the same way as Fe with HCl, however, a $ZnFe_2O_4$ compound will be formed, for which it is recommended to use sulfuric acid as it is more selective between Fe and Zn (Vereš et al., 2012). Following these extraction processes, when using sulfuric acid, Ti, Al, and Zn can be recovered with traces of Mg, all of these elements used in the formation of white pigments or as fillers in the formulation (Correia et al., 2007).

Despite the matrix's extreme complexity it may be seen from this diagram that each material that is broken down in the figure can be used as a product, avoiding the need to collect many products that would increase the purifying expenses for the final product. However, since the major goal is to lead the way in the utilization of materials made from trash, you don't want to sell each product separately.

Conclusions

- The amount of SiO_2 in the sludge from the oxidation lagoons of Costa Rican industries was calculated, and the results showed 17.2 ± 0.2% for the paint industry, 2.7 ± 0.2% for the agrochemical industry, and 0.5 ± 0.2% for the metal industry. This indicates that the paint industries have a significant amount of potential for use in artistic endeavors due to the high SiO_2 content.
- The chemical characterization of the sludges reveals that they are secure because their matrix lacks potentially harmful elements. However, it presents metals like iron and copper that, if the right use path is taken, can be employed as pigments.
- A usage plan that involves the recovery of titanium, aluminum, and silicon oxides that can be utilized in enameling or ceramics can be established due to the sludge's composition.

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