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PERSPECTIVES OF USING LIQUID WASTE FROM THE PROCESSING OF VANADIUM CATALYTIC RESIDUES IN CONSTRUCTION MATERIALS

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Abstract: Adequate industrial waste management requires not only treating the primary waste, but also the by–products generated during its processing, in order to avoid secondary environmental impacts. This principle applies to the management of spent catalysts from sulfuric acid production, which contain vanadium. Their processing by leaching with sulfuric acid and precipitation generates a liquid waste composed mainly of sodium sulphate. In this work, this new liquid residue (5.5 % sulphate, <0.30 mg/L vanadium and lead) was characterized and its use in abrasive wheels formulated with Portland 35 cement and abrasives was evaluated, resulting in an improvement in their mechanical properties, which means it can be considered for possible use. The results allow for the comprehensive recovery of these residues, both technically and environmentally, which would close the production cycle.
Keywords: industrial waste management, liquid waste, vanadium, construction materials

INTRODUCTION

The generation of industrial waste represents a significant environmental challenge, as when it is not managed properly, it can cause risks to health and the environment.

For the production of sulfuric acid, catalysts containing more than 3% vanadium pentoxide are used, which become spent over time. Once these catalysts are spent, they constitute industrial waste and must be properly disposed of as waste. In Cuba, there are currently two plants that produce sulfuric acid where these catalytic residues are generated, and it has been established that the residues will be confined indefinitely in the National Hazardous Waste Confinement Facility [7].

Spent catalysts are one of the preferred secondary raw materials for vanadium extraction, as their vanadium content is higher than the values found in many of the ores that contain this element. In addition, the average useful life of these catalysts is approximately 2 to 5 years, and can reach up to 10 years [9, 22]. Currently, many countries widely use secondary resources containing vanadium to recover the element.

Therefore, in order to avoid the confinement of catalytic waste, efforts are being made to find alternative processing methods that allow for the recovery of vanadium, an element for which there are no deposits in Cuba.

At the Universidad Central "Marta Abreu" de Las Villas, the processing of spent catalysts from sulfuric acid production is being studied, which basically consists of acid leaching and subsequent precipitation, allowing the recovery of a product composed mainly of vanadium, which can be used in the manufacture of welding consumables [19]. However, the hydrometallurgical processing of catalysts generates new residues, a solid residue that is generated during leaching and consists of 98 % silica, and a liquid residue that is produced during precipitation, consisting mainly of sodium sulphate, but may be contaminated with metallic elements such as V, Zn, Ni, Mg, Fe, and Cr [20, 21].

The diagram shown in Figure 1 illustrates the different stages of the research, which includes the study of new waste generated from catalyst processing and should conclude with possible alternative uses for each type of waste generated.

The generation of liquid waste must be managed under technical and environmental criteria to minimize its impact, being necessary the characterization of the same for which physical, chemical and biological parameters such as COD, BOD_5, suspended solids, heavy metals, pH, temperature, etc. are determined. Taking into account the characteristics of the catalytic waste, it is essential to determine the content of heavy metals in the liquid waste

generated, being possible the presence of the elements V and Pb, given their presence in the spent catalyst.

Figure 1. Stages of the investigation

Based on the results of the characterization of the liquid waste, the strategy for its discharge is drawn up, and there are norms that establish the maximum permissible values. In Cuba, one of these standards is NC 27/2012: Discharge of wastewater to land water and sewage, however, this standard does not include vanadium, being necessary to be governed by international standards [17].

Globally, various industrial activities generate metal sulphates, which are subject to strict limitations on sulphate concentrations in wastewater. Although sulphate ions are not considered hazardous, sulphate limitations are established to reduce the environmental pressure caused by increased salt concentrations in natural waters, especially in freshwater. Methods for recovering sulphates that reduce the concentration of sulphates in wastewater have recently become a topic of interest for several studies, including the precipitation of sulphate in the form of poorly soluble compounds, such as gypsum, ettringite, or barite, through chemical or electrochemical coagulation, or separating sulphate using membranes or ion exchange methods. However, these methods are not very efficient, as the removal of sulphate ions is not easy due to their high solubility and stability in aqueous solutions [11, 24].

An additional possibility is to reuse part of the sulphate solution in other processes, rather than considering it waste, which would contribute to the circular economy [24].

Therefore, to avoid the disposal of these new waste materials, the possibility of using them in the manufacture of construction materials is being evaluated.

In Cuba, environmental protection has always been of paramount importance. An example of this is the approval of Law 150 of the Natural Resources and Environmental System, which states in Article 4 that the State protects the environment and the country's natural resources. The objective of this law is to regulate the actions of the State, citizens and society in general to guarantee the implementation and operation of the Natural Resources and Environment System in Cuba [8].

In addition, Article 75 of the Constitution of the Republic of Cuba states that "all persons have the right to enjoy a healthy and balanced environment. The State protects the environment and the country's natural resources. It recognizes their close linkage with the sustainable development of the economy and society to make human life more rational and ensure the survival, well–being and security of present and future generations" [4].

The possibility of using these new residuals in the construction industry leads to the application of the principles of circular economy. The concept of circular economy is based on the fundamentals of the environmentalist school, and proposes a change to the "reduce, reuse and recycle" paradigm for a deeper and more lasting transformation, which will reduce the impact caused by human activities on the environment [10]. Under this approach, the waste becomes the "food" raw material of natural cycles or is transformed to become part of new technological products, with a minimum energy cost [10].

In general, there are no studies in the literature that provide a comprehensive assessment of the processing of catalytic waste containing vanadium. Much of the research focuses on methods for extracting and recovering vanadium in order to achieve more efficient processes. However, chemical processes commonly generate waste that, depending on the process involved, can be contaminating.

The following aspects summarize the importance of the research topic:

— Reduction of environmental impact, which may be associated with the disposal of waste that may contain metallic elements and sodium sulphate.

— Use of waste, in this case sodium sulphate contained in liquid waste generated from other chemical processes
— Circular economy approach to chemical processes developed for industrial waste
— The proposal could be applied to other waste that is processed using the methodology developed

Therefore, the objective of this work is to evaluate the possibilities of using the liquid residue generated from the chemical processing of spent catalysts based on the results of their characterization.

PROCEDURE, METHODS AND EXPERIMENTAL PART

Raw material

The analysis sample studied consists of the liquid residue generated during the chemical processing of spent catalysts from the manufacture of sulfuric acid, precipitating with Na_2CO_3 (RL). The spent catalysts that were processed come from the Patricio Lumumba Plant, in Pinar del Río, which is currently not working.

Catalyst processing. Generation of liquid waste

The general scheme describing the chemical processing of spent catalysts is shown in Figure 2.

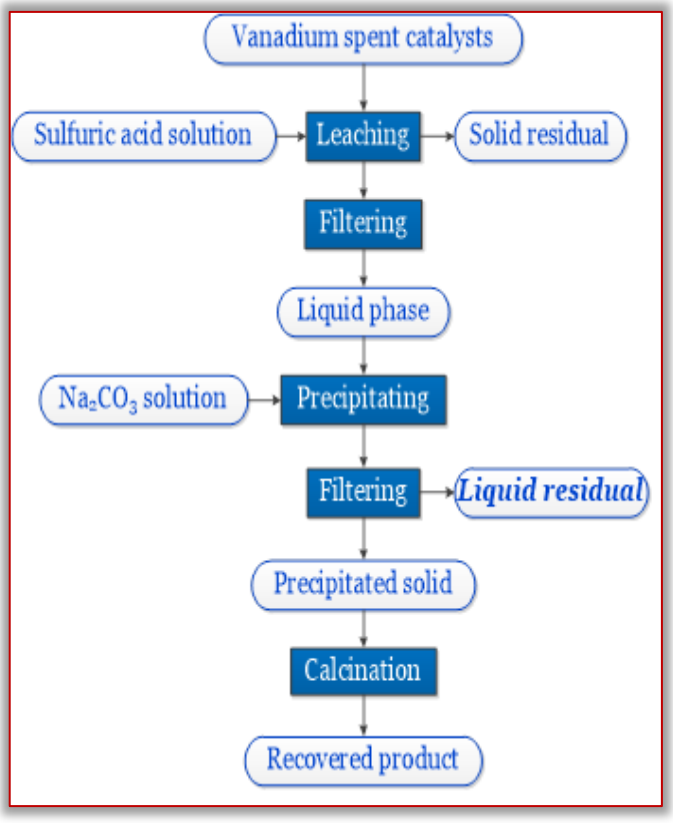

Figure 2. General processing scheme

The proposed processing consists of a first leaching stage, in which the catalyst is mixed with a diluted H_2SO_4 solution (leaching agent) in order to separate the elements capable of dissolving (leachable) from the rest of the compounds that make up the residual, which remain as insoluble residues. Once the leaching time is over, the sample is filtered, separating the liquid phase that is used for the next stage of processing. In addition, it is separated a solid residual containing the elements that were not able to dissolve in the sulfuric acid solution [18, 20].

The next stage is precipitation, in which an insoluble solid, called a precipitate, is formed from a chemical reaction between two solutions. This reaction results in the formation of solid particles that separate from the liquid medium. It is the final operation in the processing of the vanadium residue and can be performed in several ways. Proper pH control is key to vanadium separation and recovery. Selective precipitation can be used to remove unwanted impurities and metal separation [16, 20]. This stage results in a recovered solid containing the elements of interest and a liquid residual that is separated after filtration.

By using a sulfuric acid solution as a leaching agent and a sodium carbonate solution for precipitation, sodium sulphate is formed as part of the chemical reactions that occur in the process, which becomes part of the liquid waste that is generated. This process can be represented by the following chemical reaction [20]:

$$M_2(SO_4)_x + Na_2CO_3 \rightarrow M_2O_x + CO_2 + Na_2SO_4 \quad (1)$$

Where:
M is the metal,
x is the formal oxidation state of the metal.

In addition, this liquid residue may contain metallic elements such as Zn, Ni, Mg, Fe, Cr, among others, depending on the residue and its composition.

Determination of sulphate concentration in the liquid residue

For the determination of sulphate content, it was used the turbidimetry method, proposed by the Standard Methods [6], for water and wastewater analysis.

Reagents (pure for analysis):
— magnesium chloride ($MgCl_2 \cdot 6H_2O$),
— sodium acetate ($CH_3COONa \cdot 3H_2O$),
— potassium nitrate (KNO_3),
— acetic acid (CH_3COOH),
— Barium chloride crystals ($BaCl_2$),

A buffer solution was prepared for which it was necessary to dissolve 30 g of magnesium chloride, 5 g of sodium acetate, 1.0 g of potassium nitrate, and 20 mL of acetic acid (99 %), in 500 mL of distilled water and it was made up to 1000 mL. A standard solution of sodium sulphate was prepared from which a calibration curve was prepared with the points 10, 20, 25, 30, 40 mg/L. For the determination of the sulphate content, 100 mL of sample was taken and added into a 250 mL erlenmeyer, 20 mL of buffer solution was added and mixed on shaker. While stirring, a tablespoon of $BaCl_2$ crystals was added and timing was started and stirring was maintained for 60 seconds. After stirring was completed, the solution was poured into an absorption cell of the spectrophotometer. Turbidity was measured at a wavelength of 420 nm. The described procedure is performed on the calibration curve and the test sample.

- **Determination of the density of the liquid residual**

Among the analyses performed for the characterization of the liquid waste generated from the processing of the spent catalysts is the determination of its density, which will increase as the concentration of salts in the waste increases.

To determine the density of the liquid residual, a pycnometer is taken and weighed empty on the analytical balance, then the liquid residual is added, rooted and then weighed [3]. The expression to determine the density of the liquid is shown in Equation 2.

$$\rho = \frac{m_{(p+l)} - m_p}{V} \quad (2)$$

where:

m_{p+l}: mass of pycnometer and residual liquid.

m_p: mass of the empty pycnometer

V: volume of the pycnometer

- **Determination of the chemical composition of the liquid residue**

The liquid residue used was characterized at the Research Center for the Metallurgical Industry (CIPIM). The characterization was carried out by inductively coupled plasma spectroscopy (ICP).

- **Preparation of grinding wheel samples**

Among the possible uses that were evaluated for the liquid residue was its incorporation into the mixture during the manufacture of grinding wheels agglomerated with P–35 cement, in order to evaluate the effect that this residue could have on their behaviour. For this purpose, two working conditions were studied, one where the grinding wheel specimens were manufactured in laboratory conditions, and the other where they were manufactured in workshop conditions, similar to those in which the grinding wheels are manufactured in the Construction Materials Company of Villa Clara, in order to see the influence of the preparation conditions.

For the manufacture of the grinding wheel prototypes, both in laboratory and workshop conditions, the same ratio between the components was maintained.

The formulation used in the preparation of the grinding wheel prototypes is shown in Table 1. In this case, it corresponds to the samples prepared under laboratory conditions.

Table 1. Shaping of grinding wheels with residual under laboratory conditions (in g)

Mixture	Water	Liquid residue	Abrasive	Cement
P_L	17,50	–	15,00	35,00
RL_LC	–	17,50	15,00	35,00
RL_LD	8,75	8,75	15,00	35,00

For the preparation of the grinding wheels, P–35 cement was used as a binder and abrasive powder with a grain size between 0.2 – 0.1 mm [12]. The water/solid ratio evaluated was 0.5 and the amount of abrasive was kept constant. All the components, once weighed, are mixed with a mechanical stirrer for two minutes at 1,600 rpm and placed in the oven for seven days at 30 °C in a closed mold. The P_L mixture corresponds to the test tube used as a standard under laboratory conditions. The concentrated residual (RL_LC) and the residual diluted by half (RL_LD) were used.

The manufactured grinding wheels were reproduced by modifying the preparation conditions, simulating shop conditions. The solid components were mixed for seven minutes in a porcelain rotary mixer, and once the components were removed from the mixer, the residual liquid was added, continuing the mixing manually, and then the mixture was poured into test tubes with dimensions of 4.2 cm in diameter and 3 cm wide, and allowed to stand for 21 days for further evaluation. The grinding wheels were immersed in water daily for a short period of time to maintain surface moisture and promote cement hydration [1]. The formulation used for the mixtures under workshop conditions was double the quantities used under laboratory conditions (see Table 1). The P_i sample corresponds to the standard sample under the above conditions. As in the

previous analysis, the concentrated residue (RLiC) and the residue diluted by half (RLiD) were used.

A diagram of the grinding wheel preparation process is shown in Figure 4.

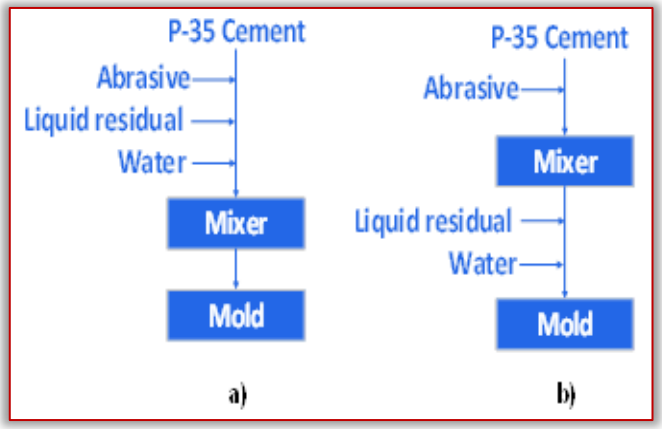

Figure 4. Grinding wheel preparation, a) laboratory scale, b) workshop scale

The grinding wheel samples manufactured were measured for compressive strength in a mechanical testing machine of the Center for Research and Development of Structures and Materials (CIDEM) of the Universidad Central "Marta Abreu" de Las Villas using the TestExpert.NET software. In the case of the samples manufactured under laboratory conditions, images of the surface were taken with an optical microscope.

RESULTS AND DISCUSSION

Chemical composition of the liquid residual

The results of the determination of the chemical composition of the liquid residual by ICP spectroscopy are shown in Table 2.

Table 2. Composition of the liquid residual

Ca	121,56	mg/L
Mg	135,60	
Mn	10,54	
Cu	0,29	
Pb	0,22	
Fe	<0,50	
Ni	11,50	
Al	0,76	
Cr	<0,20	
V	0,30	
Na	13,63	g/L

From Table 2 it can be determined that the sodium sulphate content present in the liquid residual is equivalent to 42.06 g/L (4.2 %) of Na_2SO_4. This concentration of sulphate contained in the residual allows us to evaluate possible applications of this product in cementitious materials.

On the other hand, if we add the rest of the metallic elements, we can see that they represent 0.028 % of the metals present in the liquid waste, including the most dangerous chemical elements such as vanadium and lead, which represent 0.52 mg/L.

The maximum permissible limit of vanadium in wastewater varies according to the regulation and the specific country or region, and in many cases the regulations do not report specific values for this metal.

In Cuba, Cuban Standard NC 27/2012 does not include vanadium among the elements of interest [17]. For this reason, it is taken as a reference for the analysis to be carried out what is stated in the Ecuadorian Environmental Regulations (Ribadeneira, 2001).

In the liquid waste analyzed, the vanadium content gave a value of 0.30 mg/L, which is below that established in the Environmental Regulations for Hydrocarbon Operations in Ecuador [23], which establishes a maximum permissible limit of vanadium <1 mg/L for effluent discharge, complying with the requirements of the standard.

Considering that the amount of liquid residual added to the test tubes is in the range of 17.5 to 35 mL, it is obtained that between 0.005 and 0.01 mg of V are added to the mixture, which represents between $3.9x10^{-6}$ and $7.8x10^{-6}$ % in the total mass of the formulated mixture, so the concentration in the mixture is between 0.039 and 0.078 mg/L, relatively low contents.

As for lead, it can be observed in Table 2, that the residual contains 0.22 mg/L, complying with the requirements established by Cuban Standard NC 27:2012 [17], which establishes 1 mg/L as the maximum allowable limit of lead in wastewater. This concentration value indicates that part of the lead present in the catalytic residual passed to the leached phase and subsequently under the precipitation conditions developed did not precipitate all the lead present, remaining in the liquid residual.

Regardless of the levels of vanadium and lead contained in the residue, work is being done to inertize it in a cementitious matrix, which further reduces the possible polluting effects.

Sulphate content in the residual liquid

To determine the sulphate concentration in the liquid residual sample, the method described in section 2.3 was developed, obtaining the calibration curve shown in Figure 5.

The model describing the calibration curve is as follows:

$$A = 0{,}0058\ c + 0{,}0046 \quad (3)$$

Where:
A is the absorbance
c is the concentration

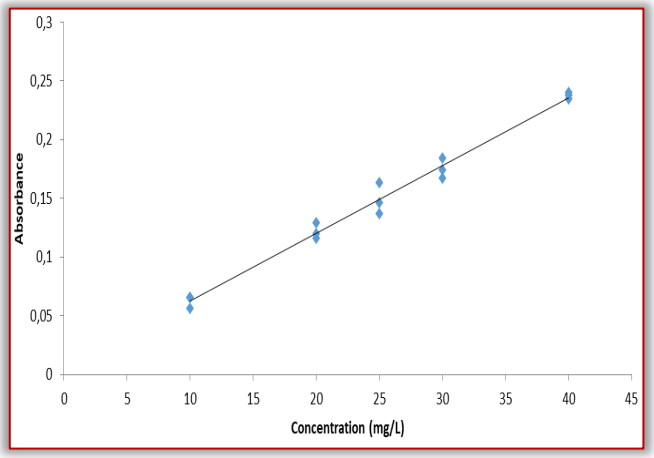

Figure 5. Calibration curve for sulphate determination.

Simple linear regression analysis between absorbance and concentration revealed a highly significant relationship ($p < 0.0001$), with a coefficient of determination (R^2) of 98.61%, indicating that the model explains almost all of the variability in the data. The slope of the regression line (0.005783) was statistically significant ($p \approx 0.0000$), confirming a positive linear dependence between the variables.

The lack of fit test showed no significant evidence ($p = 0.8771$), supporting the adequacy of the linear model. In addition, the correlation coefficient ($r = 0.993$) reinforced the strong linear association between the two variables. The residuals showed a random distribution without autocorrelation (Durbin–Watson = 2.215, $p = 0.5553$), validating the model assumptions.

As a result of the analysis performed, the sulphate concentration in the liquid residual sample was 5.49 %, which was analysed by triplicates.

Table 3. Results of sulphate determination in liquid residue

Sample	Concentration SO_4^{2-} (mg/L)	Concentration SO_4^{2-} (%)	σ
RL	54915,25	5,49	0,0479

σ: Standard deviation

As can be seen in Table 3, the sulphate concentration presents in the liquid waste analysed is 5.5 %, which is higher than the sulphate content required by the sodium determined in Table 2. This means that the rest of the sulphate present in the waste is forming sulphates with the other metallic elements contained in the waste, such as: Ca, Mg, Al, Mn, Cu, Pb, Ni, etc.

The sulphate content determined can be considered high, so it is feasible to evaluate alternative uses for this waste. In this case, it was carried out an evaluation of the influence that this residual can have in the manufacture of materials for construction, where Na_2SO_4 is frequently used as an alkaline activator.

Density of liquid residual

To determine the density of the liquid residual, the technique described in section 2.4 was used, obtaining the results shown in Table 4.

Table 4. Results of residual density determination

Residual	Density (g/cm³)
$\bar{x}$	1,053
σ	0,003

As can be seen in Table 4, the density of the liquid residual is 1.053 g/cm³, which confirms that it is a liquid with a significant concentration of dissolved solids.

Possibilities of using liquid residue on grinding wheels

It is generally accepted that alkalis, including sodium sulphate, accelerate cement hydration, which usually leads to increased strength in the early stages [2, 14].

The early acceleration of hydration by alkalis has been attributed to improved dissolution rates of the calcium silicate phases and the precipitation of the hydrated phases [124. Studies have shown that sulphates can result in a shorter induction period, a higher hydration rate during the acceleration regime, an increase in the intensity of the main heat peak, and a faster deceleration rate [15].

However, alkalis can have a detrimental impact on strength in later stages, for which there is currently no clear explanation [12]. For this reason, when analysing the uses of liquid waste, the manufacture of abrasive wheels was selected, since once these are produced, they are sent to production and consumed shortly after manufacture. Therefore, the long–term effects that the addition of sodium sulphate may cause would not be taken into account.

In addition, heavy metals can be confined in cement through different solidification mechanisms, such as adsorption, ion exchange, physical encapsulation, symbiosis, and chemical incorporation, with differences between them in their effect on hydration reactions [5].

Taking into account these criteria, it is evaluated as a possible outlet for this residual for use in grinding wheels agglomerated with Portland 35 (P–35) cement.

As a result, were obtained the grinding wheels shown in Figure 6, produced under laboratory conditions. The image shows that the grinding wheels have a high degree of compaction and do not show porosity or defects to the naked eye, the latter mainly due to the mixing conditions and the quality of the processing mold, in addition, the samples shown considerable hardness, being able to scratch glass and other metal surfaces.

Figure 6. Grinding wheels under laboratory conditions

Figure 7 shows images of the surface of the specimens obtained, which were taken with an optical microscope.

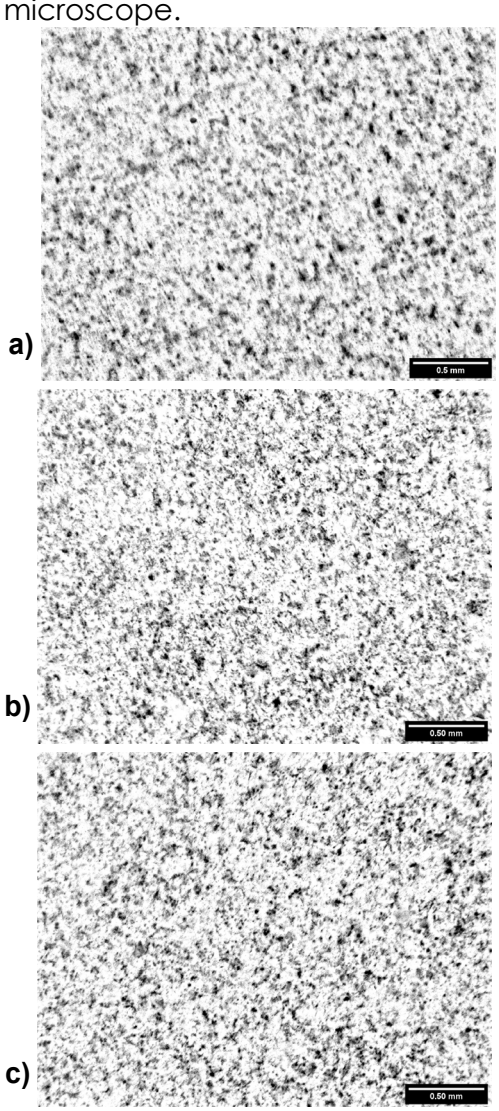

Figure 7. Image of the surface of grinding wheels. a) P, b) RL_LC, c) RL_LD

Figure 7 reveals a homogeneous distribution of the components of the mixture regardless of its composition, which is important because it shows that the abrasive was distributed uniformly throughout the specimen (grinding wheel), ensuring a stable behaviour of the grinding wheel during the grinding or polishing process of the surfaces.

To evaluate the performance of the grinding wheels, it was carried out a compressive strength test. Table 5 shows the mechanical compressive strength data obtained with the prepared samples. This study was carried out 30 days after the grinding wheel samples were made.

Table 5. Compressive strength of grinding wheels (in MPa)

Grinding wheels	Resistance (MPa)
P	18
RL_LC	28
RL_LD	20

Table 5 shows that the addition of 1.109 g of sulphate as part of the liquid residual to the mixture slightly increases the mechanical compressive strength of the RL_LD sample, with the greatest effect being observed with the addition of the concentrated solution (2.218 g of sulphate) in the RL_LC sample, which increases the strength by 55 % with respect to the original mixture.

On the other hand, as described in section 2.6, grinding wheels were manufactured in workshop conditions, obtaining the prototypes shown in Figure 8.

Figure 8. Grinding wheels in workshop conditions.

The new samples were tested for compressive strength to evaluate their mechanical properties, obtaining the results shown in Table 6.

Table 6. Compressive strength test (in MPa)

Grinding wheels	Resistance (MPa)
P	24,0
RLiC	30,1
RLiD	25,5

It can be observed in Table 6 that the highest resistance corresponds to the sample with concentrated residual (RLiC), showing an increase in resistance of 25 %; however, the sample prepared with the diluted liquid residual showed an increase in resistance of only 6 %. The trend observed in the behavior of these grinding wheels is similar to that shown by the samples manufactured under laboratory conditions, where the incorporation of the concentrated liquid residual showed the best results, confirming its positive effect.

According to the data obtained for compressive strength, shown in Tables 5 and 6, higher values can be observed for the grinding wheels manufactured under workshop conditions, in relation to those manufactured under laboratory conditions.

These results may be associated to a longer curing time for the grinding wheels manufactured in workshop conditions, which was 21 days, as opposed to the grinding wheels prepared in laboratory conditions, which was 7 days, which allowed a more complete hydration of the P–35 cement, increasing its resistance.

On the other hand, in the case of the grinding wheels prepared in workshop conditions, the natural environmental humidity could have acted as an uncontrolled moist curing, benefiting the hydration of the cement, since they were immersed in water daily, unlike those in the laboratory, which were in a closed container.

Taking into account the results obtained in the characterization of the residue and its preliminary evaluation in the manufacture of grinding wheels, Figure 9 shows the working scheme to be applied with the liquid residue generated.

Once the liquid residue is generated, if the vanadium concentration is less than 1 mg/L [23], then the residue is used in the manufacture of grinding wheels. On the other hand, if the vanadium content is higher than this value, sulfuric acid can be added to the residue, acidify the solution and then reuse the liquid in the preparation of the initial leaching solution, so that the residue is not discharged into the environment.

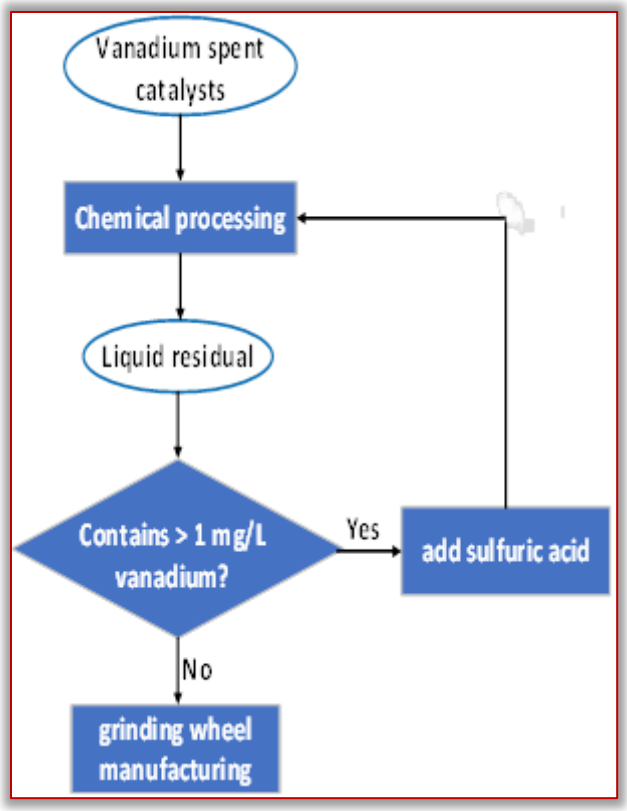

Figure 9. Sequence of work for the liquid residue

CONCLUSIONS

— The liquid residue generated from the chemical processing of the spent catalysts is constituted by 13.6 g/L of sodium, 0.22 mg/L of lead and 0.30 mg/L of vanadium as fundamental contaminating elements, a density of 1.053 g/cm^3 and 5.5 % sulphate, which allows it to be considered for use as an alkaline activator.

— The use of the liquid residual, generated during the processing of the catalysts, in the substitution of 50 % and 100 % of the water used in the formulation of grinding wheels bonded with P35 cement, increased the compressive strength of the formulated specimens, the effect being greater in the case of total substitution.

— The possibility of using the liquid residue generated from the processing of spent catalysts in the development of new materials allows improving the properties of the new product obtained and avoids its discharge into the environment.

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