

Design of a scheelite production pilot plant for the treatment of spent electroless nickel plating solutions in Field Service Solution S.A.S.: an alternative source for tungsten recycling

Clara Ballesteros ^a, Mauricio Acelas ^a, Jenny A. Oviedo ^b, & Gilberto Quintero ^a

^a Field Service Solution S. A. S. Parque Industrial Río Frio, Floridablanca, Santander, Colombia. cballesteros2104@gmail.com, eacelas@unal.edu.co, proyectos@fes-sas.com

^b Corporación para la Investigación de la Corrosión. Sede de Investigaciones UIS Guatiguará, Piedecuesta, Santander, Colombia. joviedo@corrosioncic.com

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Abstract

In this contribution, the design considerations to assemble a pilot-plant for tungsten recovery and further conversion into synthetic scheelite as raw material, as an eco-friendly improvement for the current electroless nickel process in Field Service Solution (FES) S. A. S., are presented. This low-cost methodology scale-up aims to provide an alternative treatment for spent electroless nickel solutions (Ni-P-W) as a secondary tungsten source. The plant is projected to treat up to 750 L of industrial waste per week, achieving a maximum scheelite production of 9.1 kg per week. This prototype serves as a model of eco-sustainability in the nickel-plating industry by controlling the disposal of heavy metals and by facilitating the production of other tungsten derivatives and their feasible reuse in the process.

Keywords: tungsten recycling; spent solutions; electroless nickel; scheelite.

Diseño de una planta de tratamiento de soluciones de niquelado químico agotadas para la producción de scheelita en Field Service Solution S.A.S.: una fuente alternativa para el reciclaje de tungsteno

Resumen

En este trabajo se muestran las consideraciones para el diseño de una planta piloto orientada a la recuperación de tungsteno y su posterior conversión en scheelita sintética como materia prima; esto, como una mejora ecoamigable para el actual proceso de niquelado químico en Field Service Solution (FES) S. A. S. La metodología de bajo costo mostrada provee una alternativa para el tratamiento de soluciones de niquelado químico agotadas (Ni-P-W) como fuente secundaria de tungsteno. La planta proyecta el tratamiento de hasta 750 L de desecho por semana, con lo que alcanza una producción máxima de 9,1 kg de scheelita por semana. Este prototipo constituye un modelo de ecosostenibilidad en la industria del niquelado mediante el control de la disposición de metales pesados y la producción de otros derivados del tungsteno, así como su posible reutilización en el proceso.

Palabras clave: reciclaje de tungsteno; soluciones agotadas; niquelado químico; scheelita.

1. Introduction

Tungsten is a specialty metal bearing unique hardness properties. Its alloys and products have prompted the development of tools and other technological materials that

are widely used in industry, particularly in the aerospace field [1]. However, the tungsten ores availability has significantly decreased over the last decade, promoting the use of secondary sources such as tungsten carbide scraps, as an immediate alternative to fulfill the global demand for

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tungsten powder and other derivatives [2-5]. Thus, urgent efforts to explore novel secondary tungsten sources are required to mitigate the production shortage.

More recently, tungsten has attained significant importance in electroless nickel (Ni-P-W) coating processes, considering its co-deposition provides exceptional mechanical and chemical resistance to all types of treated surfaces [6,7]. Such properties are of the utmost importance in the oil-gas industry, where the durability of drilling tools, that are constantly exposed to erosive and corrosive environments, is a crucial factor in the operation costs [8]. As consequence, the Colombian oil-gas industry has been incorporating the nickel coating in their drilling and secondary oil recovery operations, where Field Service Solution (FES) S. A. S. is contributing significantly as a leader company in the design and implementation of this cutting-edge technology.

The typical composition of an electroless nickel (EN) bath includes sodium tungstate (Na_2WO_4) in concentrations as high as 25 g/L along with other additives such as sodium hypophosphite, the reducing agent, and organic carboxylic acids acting as nickel ligands and pH stabilizers [9]. The elevated tungsten concentration and its low co-deposition ratio make these types of solutions a suitable source of tungsten for secondary recovery operations.

It should be noted that only nickel recovery strategies have been implemented in spent EN baths [10]. Moreover, after an extensive review in scientific and technical literature, no tungsten-related processes have been explored for this type of industrial waste, whose enormous global generation rates boost the urgency to investigate supplementary recycling treatments.

Tungsten recovery from tungstate ion solutions has been widely discussed in literature. Strategies involving the use of ion exchange resins [11] and the selective precipitation and coagulation with complexing agents [12] and inorganic acids [13] have successfully allowed tungsten separation. However, in the case of EN solutions, the high concentration of other additives and ions may interfere with both the resin exchange process and the organic ligands complexation. Thus, acid precipitation constitutes a viable, selective, and low-cost alternative for the tungsten recovery treatment of EN waste solutions.

In this contribution, the design considerations of a pilot plant for scheelite production, directly from spent EN solutions, is described; this, based on laboratory-scale experimentation. This process will be implemented as a tungsten recycling alternative in the FES S. A. S. electroless nickel plant, located in Floridablanca, Santander, Colombia, to provide a greener character to the vanguard nickel coating process developed by our company.

2. Materials and methods

The electroless nickel solutions were acquired from Dr. Hesse GmbH & Cie KG, and were employed for tungsten recovery after depletion of the nickel content in the electroless nickel plant of FES S. A. S. (Floridablanca, Santander, Colombia). The chemical composition of the EN solution was not disclosed by the manufacturer. For the acid-

Table 1.

Optimal dosage found for analytical-grade acids

Acid	Volume (mL/ 10 mL spent EN solution)	W recovery (%)
HCl (1:1)	40.0	60
HNO_3 (1:1)	35.0	99

Source: authors

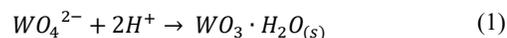
precipitation experiments, hydrochloric acid (37 %, Merck KGaA), nitric acid (65 %, Merck KGaA), calcium hydroxide (>95 %, PanReac AppliChem) and commercial nitric acid (53 %, unspecified brand), were employed.

The tungsten concentration in different batches of spent EN solution was monitored by atomic absorption (AA) spectroscopy (Agilent 240 Series AA spectrometer operating with a nitrous oxide-acetylene flame). Powder XRD experiments were performed on a D8 focus Bruker diffractometer operating in a Bragg-Brentano geometry and equipped with an X-ray Cu tube (40 kV and 40 mA) and a mono-dimensional Lynx Eye detector. The quantitative crystal phases analysis was carried out employing the Rietveld method using aluminum oxide (corundum α -phase) as internal standard. The elemental composition was established with a SEM-EDS JEOL model JCM-6000PLUS microscope using an acquisition voltage of 15 kV, a probe current of 7.5 nA and ZAF correction.

3. Description and optimization of the process at laboratory scale

Preliminary characterization by AA of the spent EN solutions allowed to measure the tungsten concentration range between 6300-14000 mg/L. However, the high variability found among the different spent solution batches is attributed to the operational conditions of the autocatalytic nickel process in plant, the type and thickness of the applied Ni-coating, and the evaporation processes during storage, considering open containers are employed.

The experiments at the laboratory scale were carried out using 10 mL of a 7500 mg/L tungsten spent EN solution. The tungstate precipitation ($\text{WO}_3 \cdot \text{H}_2\text{O}$) (eq. (1)) was initially investigated by the addition of analytical grade hydrochloric and nitric acids in a 1:1 (v/v) concentration, at room temperature. The acid dosages found to provide the maximum tungsten recovery under these conditions are described in Table 1. The solid formed was easily recovered by filtration and dried at 90° C for 12 hours in all cases.



SEM-EDS and XRD characterization confirmed that the yellow solid product obtained after the addition of HCl corresponds to tungstate with a purity greater than 99 % and good crystallinity (Fig. 1). Nevertheless, despite an outstanding tungsten recovery was achieved when HNO_3 is employed, the isolated white product is made of tungsten oxides and phosphite/orthophosphate salts amorphous agglomerates (Fig. 1) [14].

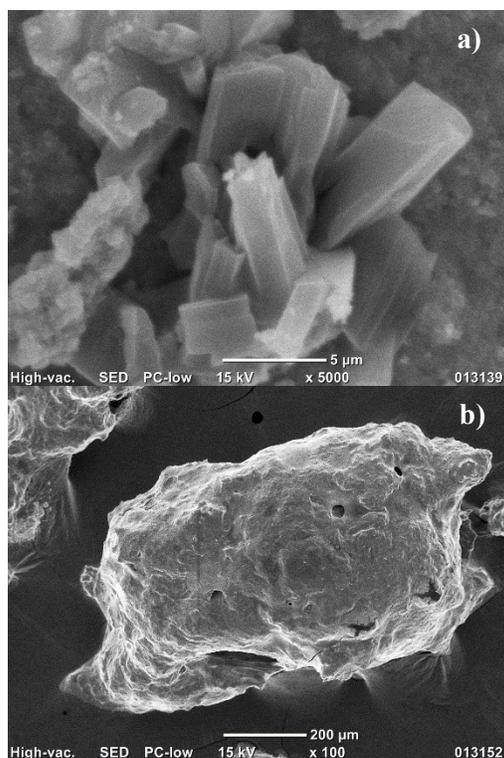


Figure 1. SEM micrograph of the obtained products with HCl (a) and HNO₃ (b)
Source: authors

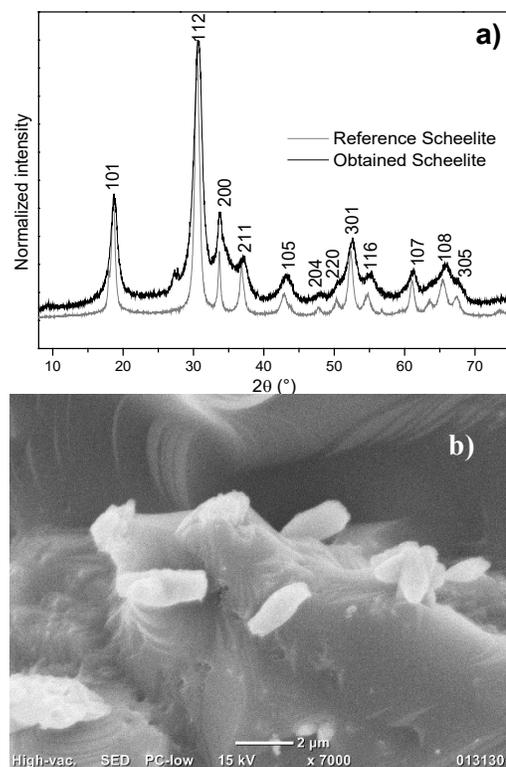
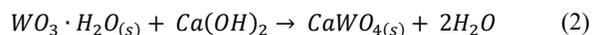


Figure 2. Obtained scheelite XRD profile (a). Obtained scheelite SEM micrograph (b)
Source: authors

The formation of the previously mentioned mixture of tungsten oxides and the simultaneous oxidation of the hypophosphite ions, initially found in the spent EN solution, can be explained in terms of the weaker acidity and stronger oxidizing character of the HNO₃, compared to HCl [15].

Once the addition of HNO₃ was established as the best experimental procedure in terms of tungsten recovery, the amorphous solid was brought into reaction with a Ca(OH)₂ 0.1 M solution at room temperature (eq. (2)). The insoluble scheelite (calcium tungstate, CaWO₄) formed was immediately recovered by filtration with a 73 % purity.



Considering that the combination of tungsten oxides and calcium hydroxide is an acid-base type reaction, the rapid scheelite formation leads to a decrease in the size of the crystals, as corroborated by the peak broadening observed in the XRD pattern, when compared to reference scheelite and to the SEM micrograph shown in Fig. 2.

Because of an operational cost optimization in the scaling-up of the process, the effect of non-analytical grade nitric acid (50 % w/w) was explored. Interestingly, a 1:1.2 ratio (spent EN solution volume: HNO₃ volume) achieved an equivalent tungsten recovery percentage compared to the analytical-grade reagent. Moreover, only a 6 % difference in the final product content of scheelite was observed when comparing the crystal phases composition (Table 2).

Table 2.
XRD characterization of the final products obtained with different types of nitric acid

Compound	Name	Mass composition (%)	
		Analytical-grade HNO ₃	Non-analytical HNO ₃
CaWO ₄	Calcium tungstate	72.8	67.1
Ca(HPO ₄)(H ₂ O)	Hydrated calcium hydroxide phosphate	19.0	32.9
(NH ₄) ₂ HPO ₄	Fosfammite	4.5	0
Ca ₂ (PO ₂ NH) ₄ (H ₂ O)	Hydrated calcium fosfimate	3.7	0
Ca(OH) ₂	Portlandite	<1.0	0

Source: authors

These outcomes indicate that the type of the nitric acids evaluated has no major effect in the final product quality. Hence, the non-analytical grade nitric acid was selected as raw material for the design of the tungsten recovery plant.

4. Pilot plant design considerations

Based on the laboratory scale results and the chemical reactions involved, a general scheme for the pilot plant is presented in Fig. 3. The process starts with the reaction of tungstate ions in the spent EN solution with nitric acid (50 % w/w) in a 1:2.2 volume ratio in the reactor R-101, yielding an insoluble solid, whose composition has been previously

discussed. Since the addition of an inorganic acid is taking place, we propose to maintain the temperature inside the reactor below 30° C employing an aerator-cooler to guarantee product conversion, avoid the generation of by-products and to maintain safety operative conditions. A stirrer is also contemplated to facilitate reactivity.

The insoluble material obtained (mixture of tungsten oxides) is separated in the decanter V-101, where the supernatant unreacted solution is removed and the solid is then transferred to reactor R-102. Next, the Ca(OH)₂ 0.1 M solution is added until a pH of 7.0 is reached. Again, the temperature is kept under 30° C and a stirrer is projected to enhance reactivity. At this point, the scheelite is formed and is subsequently separated in the decanter V-102. Once the supernatant is removed, a wash cycle with distilled water is carried out to eliminate soluble impurities. Then, the solid material is separated again in the V-102 unit and, placed in the drying oven H-101 at 90° C, where the scheelite product is obtained with a minimum purity of 67 %.

To determine the pilot plant capacity, the generation rate of spent EN solution in the FES S. A. S. nickel plant, 750 L/week, was considered as the starting point. As it has been previously pointed out, the tungsten concentration in the spent solutions is variable and depends on operational conditions, among other factors. Therefore, an average

tungsten concentration of 7813 mg/L was specified based on the AA analysis performed for different batches of solution waste. Additionally, a 100 % conversion of the tungstate into scheelite was considered as an overdesign element. With this information in hand, the average production of the pilot plant is projected to be 9178.2 g of scheelite per week. Also, to facilitate the operation and avoid the manipulation of excessive amounts of acid, two batches per day are proposed involving the treatment of 53.6 L of spent EN solution each 12 hours.

The reactor sizing stage considered the total volume of operation, the geometry and the volume change at the end of the chemical reaction. Since the process requires the use of highly corrosive substances, polyvinylidene fluoride (PVDF) was selected as the construction material due to its exceptional chemical resistance. However, only PVDF sheets are distributed by providers, implying that a rectangular geometry reactor must be constructed (Fig. 4). Regarding the volume change, experiments performed at the laboratory demonstrated that after the reaction is completed, no significant modification of the volume was observed. For practical purposes, this volume was assumed to remain constant. After the above-mentioned considerations and a 50 % overdesign element by stirring effects, the proposed total volume for the reactor R-101 is 171 L.

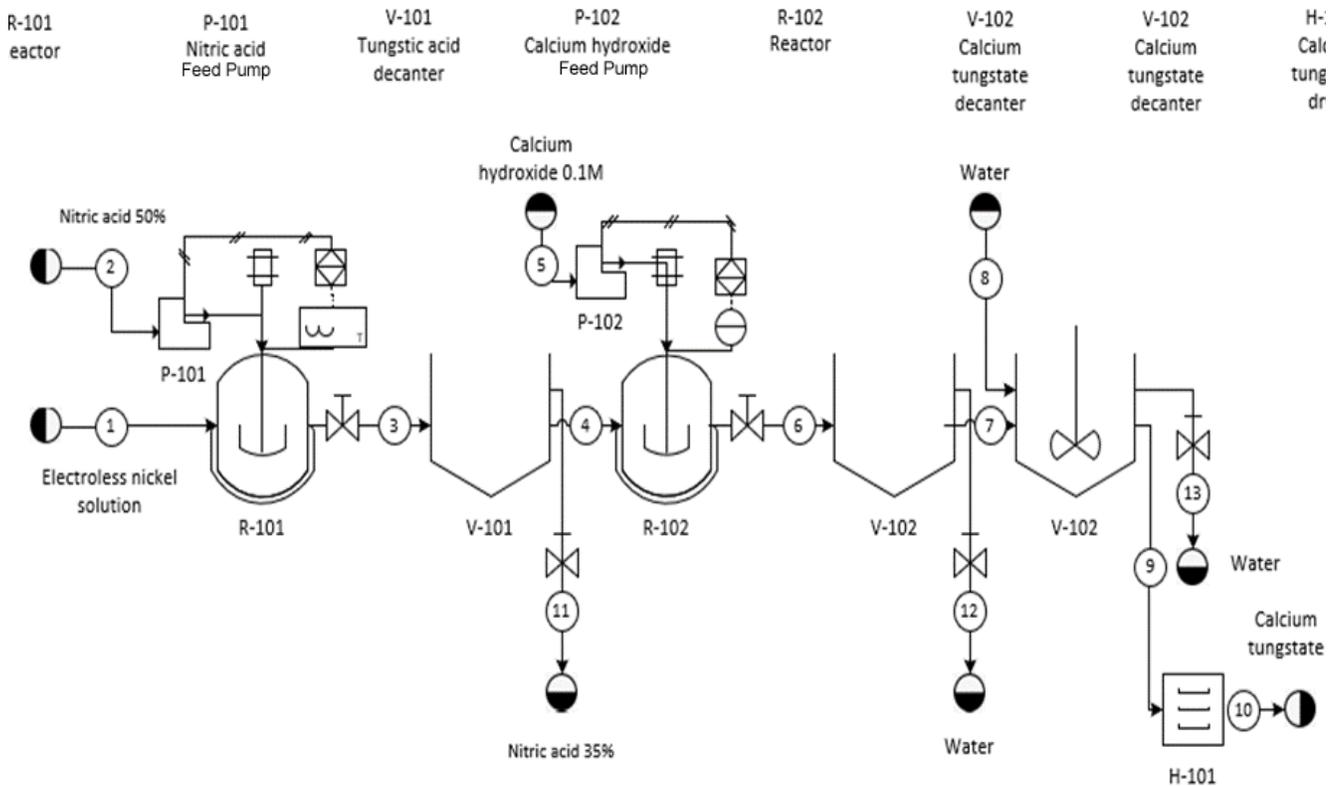


Figure 3. Developed process flowsheet

Source: authors

Note: The decanter V-102 is presented twice to enlarge design details

To ensure the formation of a more crystalline material in the reactor R-101 [16], the acid must be added slowly into the spent EN solution, according to the reaction shown in eq. (3).



The mass balance for this reactor contemplates the mass of the spent EN solution (0.011 % sodium tungstate) (\dot{m}_1), the mass flow of nitric acid (\dot{m}_2) and the accumulation of mass between the final product and the unreacted materials ($\frac{dm_3}{dt}$), as shown in eq. (4). The minor mass loss ascribed to the release of acid gases was not considered.

$$\dot{m}_1 + \dot{m}_2 = \frac{dm_3}{dt} \quad (4)$$

Based in the general energy balance (eq. (5)), the process within R-101 involved the internal energy of the spent solution (u_1), the input enthalpy of the sodium tungstate (h_{e2}), heat of reaction (Q_{rxn1}) (between the tungstate and the acid), the heat of mixture between the acid and the solution (Q_{mix1}), and the energy of the system due to accumulation ($\frac{dE_1}{dt}$), as shown in eq. (6). The schematic global balance diagram for the reactor is depicted in Fig. 4.

$$\dot{E}_{inlet} + \dot{E}_{outlet} = \Delta E \quad (5)$$

$$(\dot{m}_1 * u_1) + (\dot{m}_2 * h_{e2}) + Q_{rxn1} + Q_{mix1} = \frac{dE_1}{dt} \quad (6)$$

It should be disclosed that the enthalpy and internal energy values of the spent solution were approximated to those reported for water, considering the overall composition (>70 % water). From eq. (6), the heat of mixture is calculated to be -19372 kJ leading to a final temperature of 56.4° C if the addition of acid occurs in a single step. To avoid this, and to maintain the reaction temperature below 30° C, heat extraction by direct aeration with cooled air at 5° C and a tube air diffuser is proposed. Moreover, the addition of nitric acid is planned to occur during a 3 h time lapse. The required mass of air (963.8 kg/3h) was calculated from eq. (7), where Q is the heat needed to be extracted from the mixture (19372 kJ), m is the mass of air (kg), C_p the heat capacity of air 1.005 kJ/kg° C [16], and ΔT is the temperature difference of air inlet and outlet. This last one was established in 20° C.

$$Q = mC_p\Delta T \quad (7)$$

The tungsten oxide settling velocity was identified as the main contribution that influences the decanter V-101 design. After the determination of this parameter in the laboratory, the type of sedimentation exhibited characteristics analogous to suspensions with a high concentration of solids with a well-defined limiting layer between the sedimented mass and the supernatant liquid [18,19].

From Kynch method [20], the decanter total area was calculated according to eq. (8), where A_t is the total area of the decanter, 1.5 is an oversdesign factor, ϕ is the hypothetic inlet flow rate (0.171 m³/h), t_u is the experimental retention time 176.32 min, and h_o is the initial vessel height 0.678 m. From these parameters, A_t for V-102 corresponds to 1.109 m².

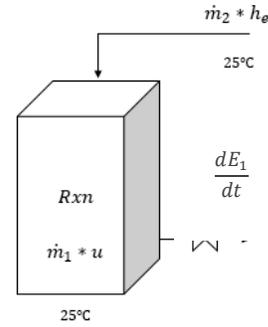


Figure 4. Balance representation for reactor R-101
Source: authors

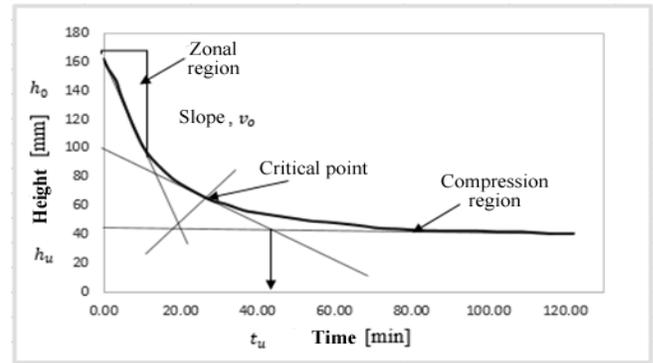


Figure 5. Design parameters for V-101 obtained from the settling velocity curve of tungsten oxides
Source: authors

$$A_t = 1.5 \phi t_u / h_o \quad (8)$$

On the other hand, the sludge area of the decanter depends on the operating time-lapse and the amount of sludge produced. To determine this area (clarification area A_c), eq. (9) was employed, where 2 is an oversdesign factor, ϕ is the hypothetic inlet flow rate (0.171 m³/h), and v_o is the slope of the zonal region (0.0059 m/min) observed in Fig. 5. The clarification area was calculated to be 0.96 m² (an additional heuristic oversdesign factor of 30 % was also considered). It should be disclosed that the clarification area was proposed to have a rectangular pyramidal geometry (Fig. 6) based on the availability of the PVDF material and, as a strategy to improve the settling velocity.

$$A_c = 2 \phi / v_o \quad (9)$$

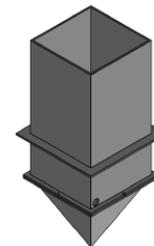


Figure 6. Proposed geometry for decanter V-101
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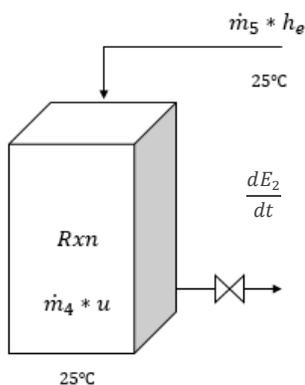


Figure 7. Balance representation for reactor R-102
Source: authors

For reactor R-102, the total volume was established in 206 L, taking into consideration the average volume ratio of $\text{Ca}(\text{OH})_2$ 0.1 M required and the tungsten oxide sludge separated from the decanter V-101. The mass balance in reactor R-102 considered the mass of the tungstic acid produced in the first reactor (\dot{m}_4), the amount of $\text{Ca}(\text{OH})_2$ necessary for the neutralization (\dot{m}_5), and the accumulation of mass between the final product and the unreacted materials ($\frac{dm_6}{dt}$) as shown in eq. (10).

$$\dot{m}_4 + \dot{m}_5 = \frac{dm_6}{dt} \quad (10)$$

The heat balance in reactor R-102 involved the inlet enthalpy of $\text{Ca}(\text{OH})_2$ 0.1 M (he_5), the internal energy of the tungsten oxides (u_4), the heat of mixture that included an estimated composition of water- HNO_3 (Q_{mix2}), the heat of reaction (Q_{rxn2}), and the energy accumulation attributed to the addition of calcium hydroxide ($\frac{dE_2}{dt}$) as shown in eq. (11) and Fig. 7.

The heat of mixture in reactor R-102 was calculated according to eq. (11) to be -5160 kJ. However, this amount of energy can increase the temperature up to 30.7° C only. Thus, a cooling system was not contemplated for this stage.

$$(\dot{m}_4 * u_4) + (\dot{m}_5 * he_5) + Q_{rxn2} + Q_{mix2} = \frac{dE_2}{dt} \quad (11)$$

The decanter V-102 design parameters were established using the same methodology and geometry of decanter V-101. Based on the settling curve for scheelite (Fig. 8), the total area and the clarification area were determined to be 1.14 m² and 0.047 m², respectively.

After scheelite separation in the V-102 unit, an additional washing step is considered to remove soluble impurities that may have been adsorbed on the product surface. This treatment is proposed to involve 40 L of distilled water to wash the 36 L of scheelite precipitate employing an anchor-type stirrer. Once the washing operation has ended, the scheelite is again separated in V-102.

For the last step, the excess of water absorbed by the final product is removed in an electrical drying oven, which is

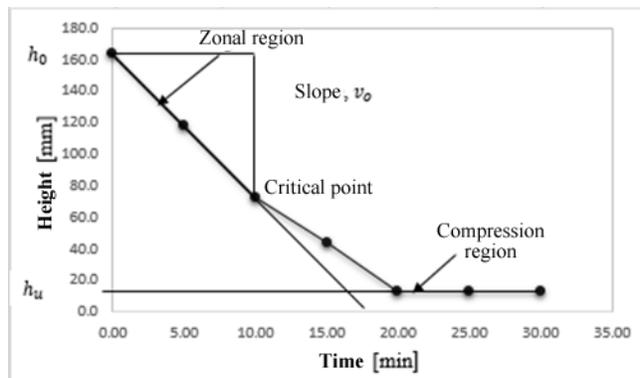


Figure 8. Design parameters for V-102 obtained from the settling speed curve of scheelite
Source: authors

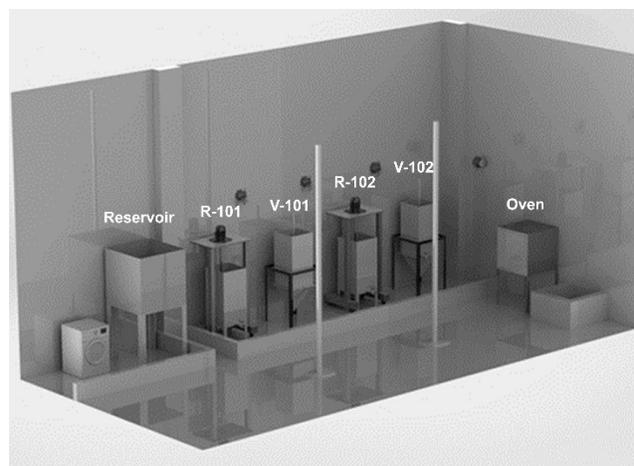


Figure 9. Pilot plant 3D scheme
Source: authors

planned to contain five racks, holding two 0.5m x 0.1m x 0.8m refractory glass trays. The total charge of the oven was dimensioned from the total mass of product obtained in the batches (simultaneous 72 kg of product and 36 kg as overdesign element), and considering that each batch requires three days at 90° C to achieve complete moisture removal, as indicated by the laboratory experiments.

Finally, after consideration of the above discussed elements of design for the scheelite production pilot plant, a 3D prototype of the plant to be installed in the FES S. A. S nickel plant is depicted in Fig. 9.

5. Conclusion

An innovative process for recycling of tungsten as scheelite from spent electroless nickel solutions was described. The implemented process proved to be efficient for the generation of scheelite product with a minimum purity of 67 %; this, when a non-analytical grade nitric acid was employed.

Moreover, the key plant design considerations were successfully established from laboratory scale experiments

and basic operational parameters. The installation of this tungsten recycling plant is projected as a contribution to the national chemical industry and as an environmental strategy to reincorporate raw materials in the EN solution formulation.

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References

- [1] Mishra, D., Sinha, S., Sahu, K.K., Agrawal, A. and Kumar, R., Recycling of secondary tungsten resources, *Trans. Indian Inst. Met.*, 70(2), pp. 479-485, 2017. DOI: 10.1007/s12666-016-1003-8
- [2] Luo, L., Kejun, L., Shibayama, A., Yen, W., Fujita, T., Shindo, O. and Katai, A., Recovery of tungsten and vanadium from tungsten alloy scrap, *Hydrometallurgy*, 72(1-2), pp. 1-8, 2004. DOI: 10.1016/S0304-386X(03)00121-X
- [3] Leal-Ayala, D.R., Allwood, J.M., Petavratzi, E., Brown, T.J. and Gunn, G., Mapping the global flow of tungsten to identify key material efficiency and supply security opportunities, *Resour. Conserv. Recycl.*, 103, pp. 19-28, 2015. DOI: 10.1016/j.resconrec.2015.07.003
- [4] Shemi, A., Magumise, A., Ndlovu, S. and Sacks, N., Recycling of tungsten carbide scrap metal: a review of recycling methods and future prospects, *Miner. Eng.*, 122(September) 2017, pp. 195-205, 2018. DOI: 10.1016/j.mineng.2018.03.036
- [5] Tkaczyk, A.H., Bartl, A., Amato, A., Lapkovskis, V. and Petranikova, M., Sustainability evaluation of essential critical raw materials: cobalt, niobium, tungsten and rare earth elements, *J. Phys. D. Appl. Phys.*, 51(20), pp. 203001, 2018. DOI: 10.1088/1361-6463/aaba99
- [6] Stepanova, L.I., Bodrykh, T.I. and Sviridov, V.V., The effect of tungsten inclusion into nickel-phosphorus films on their thermal and barrier properties, *Met. Finish.*, 99(1), pp. 50-58, 2001. DOI: 10.1016/S0026-0576(01)80560-2
- [7] Ijeri, V., Bane, S., Shah, K. and Goradia, P., The electroless deposition of nickel-phosphorus-tungsten alloys, *Prod. Finish.*, 78(7), pp. 1-7, 2014.
- [8] Arun, I., Duraiselvam, M., Senthilkumar, V., Narayanasamy, R. and Anandakrishnan, V., Synthesis of electric discharge alloyed nickel-tungsten coating on tool steel and its tribological studies, *Mater. Des.*, 63, pp. 257-262, 2014. DOI: 10.1016/j.matdes.2014.06.029
- [9] Eltoun, M.S.A., Electroless and corrosion of nickel, phosphorus, tungsten alloy, *J. Middle East North Africa Sci.*, 2(2), pp. 16-24, 2016.
- [10] Li, L., Takahashi, N., Kaneko, K., Shimizu, T. and Takarada, T., A novel method for nickel recovery and phosphorus removal from spent electroless nickel-plating solution, *Sep. Purif. Technol.*, 147, pp. 237-244, 2015. DOI: 10.1016/j.seppur.2015.04.029
- [11] Srivastava, R.R., Mittal, N.K., Padh, B. and Reddy, B.R., Removal of tungsten and other impurities from spent HDS catalyst leach liquor by an adsorption route, *Hydrometallurgy*, (127-128), pp. 77-83, 2012. DOI: 10.1016/j.hydromet.2012.07.004
- [12] Makino, T., Nagai, S., Iskandar, F., Okuyama, K. and Ogi, T., Recovery and recycling of tungsten by alkaline leaching of scrap and charged amino group assisted precipitation, *ACS Sustain. Chem. Eng.*, 6(3), pp. 4246-4252, 2018. DOI: 10.1021/acssuschemeng.7b04689
- [13] Plattes, M., Bertrand, A., Schmitt, B., Sinner, J., Verstraeten, F. and Welfring, J., Removal of tungsten oxyanions from industrial wastewater by precipitation, coagulation and flocculation processes, *J. Hazard. Mater.*, 148(3), pp. 613-615, 2007. DOI: 10.1016/j.jhazmat.2007.03.016
- [14] Quintero, G., Brunner, E., Jiménez, L-A., Oviedo J-A. and Acelas, M., Scheelite production from spent electroless nickel solutions as an alternative tungsten recycling source, *Hydrometallurgy*, 197, pp. 105453, 2020. DOI: 10.1016/j.hydromet.2020.105453
- [15] Feldstein, N., Lancsek, T.S. and Amick, J.A., Potentiometric analysis of hypophosphite in electroless nickel plating baths, *Anal. Chem.*, 42(8), pp. 945-946, 1970. DOI: 10.1021/ac60290a033
- [16] Chai, Y., Ha, F.Y., Yam, F.K. and Hassan, Z., Fabrication of tungsten oxide nanostructure by sol-gel method, *Procedia Chem.*, 19, pp. 113-118, 2016. DOI: 10.1016/j.proche.2016.03.123
- [17] Cengel, Y.A. and Boles, M.A., *Thermodynamics. An Engineering Approach*, 7th ed. New York: McGraw-Hill, 2011.
- [18] Vanderhasselt, A. and Vanrolleghem, P.A., Estimation of sludge sedimentation parameters from single batch settling curves, *Water Res.*, 34(2), pp. 395-406, 2000. DOI: 10.1016/S0043-1354(99)00158-X
- [19] Harris, C.C., Somasundaran, P. and Jensen, R.R., Sedimentation of compressible materials: analysis of batch sedimentation curve, *Powder Technol.*, 11(1), pp. 75-84, 1975. DOI: 10.1016/0032-5910(75)80026-X
- [20] Concha, F. and Bustos, M.C., A modification of the Kynch theory of sedimentation, *AIChE J.*, 33(2), pp. 312-315, 1987. DOI: 10.1002/aic.690330219

C. Ballesteros, received the BSc. Eng. in Chemical Engineering from the Universidad Industrial de Santander, Colombia. Her research focuses on the development of strategies for metals recycling.
ORCID: 0000-0003-4016-2154

M. Acelas, received the BSc. in Chemistry in 2012 and the MSc. Chemistry in 2016, all of them from the Universidad Industrial de Santander, Colombia. He is currently enrolled as a Chemistry PhD student at Universidad Nacional de Colombia. His research is focused on the synthesis of organic conjugated systems, polymers and hybrid materials, and their evaluation in photochemical applications. Moreover, he has experience in spectroscopic characterization of polymeric coating materials, and in the development of chemical processes for elements and compounds recovery. He is part of the scientific advisory board in FES S.A.S.
ORCID: 0000-0003-1369-4868

J.A. Oviedo, received the BSc. in Chemistry in 2010, the MSc. Chemistry in 2012, and the Sp. in Integrity and Corrosion Management in 2019. She is currently the responsible of the technological line of physical chemistry in Corporación para la Investigación de la Corrosión (CIC). Her experience is based on the physicochemical study of internal corrosion processes in the oil and gas industry, as well as the management of the chemical analysis laboratory at CIC.
ORCID: 0000-0002-0152-715X

G. Quintero, received the BSc. Eng. in Metallurgy Engineering in 1986. His extensive expertise involves materials, special welding, thermal processes, special steels, and their heat and thermochemical treatments. He has received specialized courses in Austria and Germany on these subjects.
ORCID: 0000-0002-4640-9704