Roasting of sulphide using carbothermal reduction

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Abstract

The carbothermic reduction process is a direct reduction of sulfides. It allows a roasting in which higher concentrations of metals such as molybdenum are obtained by using both a reducing agent (in this case, carbon C) and a sulfur scavenger, such as CaO so as to prevent air toxic gases pollution such as SO_2. In this paper, we analyze sulfur concentrates rich in copper, iron, and molybdenum that are obtained through rougher flotation and differential floats, which give rise to different laws. The concentrates were subjected to a carbothermic reduction process in which the working parameters were studied. The values of both the capturing agent and the reducing agent were adjusted to retain higher concentrations of sulfur. Analyzing the results obtained, the temperature was set and we reached the optimal working time and optimum molar ratio of CaO and C for the greatest recovery of metals such as molybdenum.

Keywords: carbothermic reduction; sulfides; roasting.

Tostación de sulfuros por reducción carbotérmica

Resumen

El proceso de reducción carbotérmica es una reducción directa de sulfuros. Permite realizar una tostación en donde se obtienen concentraciones mayores de metales como molibdeno, empleando un agente reductor (en este caso, el carbón C) y un captador de azufre como el CaO, evitando la contaminación del aire con gases tóxicos como el SO_2.
En este trabajo se analizan concentrados sulfurados ricos en cobre, hierro y molibdeno, obtenidos por flotación rougher y flotaciones diferenciales, dando origen a diferentes leyes. Los concentrados se sometieron al proceso de reducción carbotérmica en donde se estudiaron los parámetros de trabajo. Se ajustaron los valores del agente captador y reductor, para retener las mayores concentraciones de azufre. Analizando los resultados obtenidos se fijó la temperatura, el tiempo óptimo de trabajo y optimas relación molar del CaO y C para los cuales se alcanzó la mayor recuperación de metales como el molibdeno.

Palabras clave: reducción carbotérmica; sulfuros; tostación.

1. Introduction

1.2. Obtaining Molybdenum

Molybdenum is recovered mainly as molybdenite (MoS_2) from minerals or concentrates that have been obtained by using "rougher" or "differential" flotation. A roasting or leaching is later carried out using nitric acid or nitrate/sulfuric acid, technical grade molybdenum trioxide (MoO_3) is thus obtained [1,2].

However, within the framework of the conventional metallurgical processes, the treatment of concentrates includes one or more oxidation stages to obtain a high-temperature metal, generally by using roasting furnaces. In general, such operations involve SO_2 formation, which generates environmental pollution problems. From an economic standpoint, it is seldom feasible to abate this completely, and as such a considerable part of this gas frequently leaks into the atmosphere as chimney gas or flue gas [3,4].

Oxidizing roasting is a traditional pyrometallurgical method that is not always possible for the whole variety of metallic sulfides, especially if these are low-grade concentrates or when they are forming disseminated mineral

deposits or polymetallic sulfide deposits. This is generally due to the complexity of treatment flow charts, followed by the high cost of eliminating the SO_2 gas that is produced in these pyrometallurgical methods.

An alternative method for the treatment of sulfide concentrates that prevents air from being polluted by SO_2 is the direct reduction of sulfides with appropriate reducing agents in the presence of a desulfurizing agent such as CaO, $CaCO_3$, or Na_2CO_3 [7,8]. Such processes are generally applied to MoS_2 concentrates with grades higher than 21%. Carbon C is the reducing agent that is used worldwide to obtain reduced sulfide [6, 8]. The general equation (1) for carbothermic reduction of metallic sulfides in the presence of CaO and C can be represented by the following global reaction:

$$MeS + C + 2CaO + O_2\,(g) \leftrightarrow MeO + CaS + CO_2\,(g) \quad (1)$$

In this work, a method is presented to obtain a greater molybdenum recovery. This method may be applied to concentrates produced directly through various flotation processes, the concentrates containing a Mo percentage that ranges from 0.4-3.0 %.

1.2. The thermodynamics of carbothermic reduction roasting

Carbothermic reduction reactions have been shown to occur mainly through intermediary gaseous compounds, and, to a lot lesser degree, through reactions among solids [6,7,8]. Therefore, it is to be expected that the reduction reactions of metallic sulfides in the presence of CaO may also occur through the CO_2 and CO gaseous species. Based on the above, it can be stated that the chemical equation 2 dominates the carbothermic reduction process for molybdenum:

$$MoS_2 + C + 2CaO + 1.5O_2(g) \leftrightarrow MoO_3 + 2CaS + CO_2(g) \quad (2)$$

Figure 1. Stability diagram for the Mo-CaO-S_2-O_2 at 600 °C for the 0<Mo/Mo+Ca<0.5 metallic fraction.
Source: The authors.

Compounds used in the carbothermic reduction such as CaO and C may be analyzed in detail through the Me-CaO-S2-O2 systems phase equilibrium diagrams. For a 600° C condition and a 0<.Mo/Mo+Ca<0.5 metallic fraction, the phase diagram shown in Fig. 1 may be obtained. The area of interest for molybdenum reduction is the superior right area. This is in line with chemical reaction 2, which is carried out at relatively high temperatures.

2. Experimental Procedure / Method

The samples under study come from two different flotation processes, namely, rougher flotation and differential flotation. Each concentrate presents different chemical compositions from those of elements such as Cu, Fe and mainly Mo. The aim is of this research is to study the behavior of each concentrate in the roasting process by using carbothermic reduction for different chemical compositions. The most favorable conditions of the raw material are determined so that these are fed into the roasting furnace.

The mineralogical characterization was obtained using a Philips Diffractometer WP 1011. Chemical composition was determined through volumetric techniques by acid digestion attack and quantification with atomic absorption spectrophotometryby using a Perkin Elmen AA 100 equipment. Total sulfur quantification was performed with the ASTM C-25 technique. For each of the chemical elements, Cu, Fe and Mo, specific techniques were used [9-11].

Based on the results of chemical and mineralogical composition, various experimental tests were designed to simulate the roasting process by using carbothermic reduction. Tests were conducted on a 1g sample with CaO and C for various stoichiometric ratios: these are always referred to as Mo, that is, Mo:CaO:C. The sample was placed in a porcelain crucible that turned out to be inert to the chemical reactions involved in the roasting process through carbothermic reduction. After, the sample was placed in a muffle furnace with an oxidation atmosphere. Finally, the roasting product was analyzed by means of an acid digestion attack and was read with an atomic absorption spectrophotometer.

To summarize, on the laboratory scale, the concentrates are treated with carbothermic reduction for the aim of finding the optimal operational conditions for which the highest percentage of molybdenum recovery can be obtained.

2.1. Tests conducted on concentrates produced with rougher flotation

2.1.1. Temperature variation – Time variation

The samples are introduced into a muffle kiln at a constant temperature with CaO and C to Mo molar ratios of 1:1:1 (Mo:CaO:C) for an established variable time. The sample exposure was evaluated at: 500 ℃, 600 ℃, 700 ℃ and 800 ℃. For each temperature, the same roasting times were run, that is, from 15 min, 30 min, 60 min, 90 min, and 120 min to 180 min.

2.1.2. Variation of molar ratios (Mo:CaO:C)

Once the roasting temperature was set up, for which the maximum molybdenum value was obtained, work was started with molar variations. Initially, CaO varied with respect to Mo, and the C concentration was left fixed. Once the molar ratio for CaO was established, the stoichiometric ratio of C started to vary with respect to Mo.

2.2. Tests conducted on concentrates obtained with differential flotation

2.2.1. Temperature Variation – Time variation – Variation of molar ratios (Mo:CaO:C)

For concentrates produced with differential flotation, the same working conditions that were applied to concentrates obtained with rougher flotation were taken as a basis as the mineralogy of both flotations is quite similar.

The samples exposure was evaluated at different temperatures: 500 ℃, 600 ℃, 700 ℃ and 800 ℃. For each temperature, the same roasting times were run, from 15 min, 30 min, 60 min, 90 min, and 120 min to 180 min. It should be pointed out that the tests were carried out rendering the molar ratios fixed for CaO and C with respect to Mo (Mo:CaO:C).

3. Results

3.1. Mineralogical characterization

Copper and iron ores such as chalcopyrite (CuFeS2) and pyrite (FeS2) were determined from the mineralogical study and smaller proportions of minerals such as molybdenite (MoS2) were observed. The diffractograms obtained are shown in Figs. 2 and 3.

3.2. Chemical characterization

Results from the chemical characterizations of each of the concentrates are shown in detail in Tables 1 and 2.
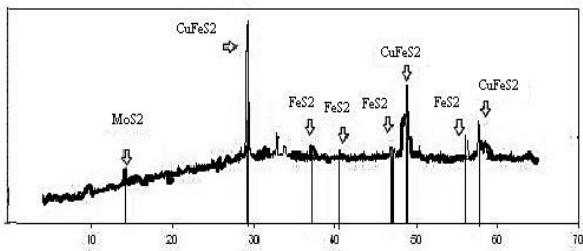
Figure 2. Diffractograms of sulfide products produced with rougher flotation.
Source: The authors.
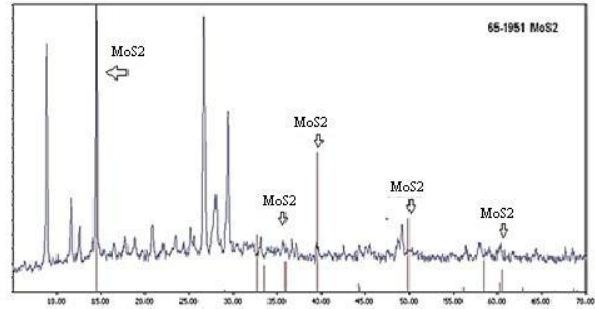
Figure 3. Diffractograms of sulfide products from differential flotation.
Source: The authors.

Table 1.
Chemical composition of concentrates obtained through rougher flotation – "R" samples

Mtras.	Mo (%)	Cu (%)	S (%)	Fe (%)	CaO (%)	MgO (%)	Other (%)
R1	0,43	27,69	32,51	30,61	0,18	0,17	7,83
R2	0,50	26,81	31,30	28,01	0,18	0,08	12,52
R3	0,64	26,09	31,01	30,45	0,18	0,17	11,35
R4	0,83	25,77	31,01	31,89	0,18	0,10	9,40
R5	0,94	24,71	29,91	31,14	0,22	0,36	12,54

Source: The authors.

Table 2.
Chemical composition of concentrates obtained through differential flotation – "D" samples

Mtras.	Mo (%)	Cu (%)	S (%)	Fe (%)	CaO (%)	MgO (%)	Other (%)
D1	1,22	9,54	33,57	17,00	7,59	5,89	24,34
D2	2,11	9,98	19,53	17,51	6,49	7,69	36,17
D3	2,89	10,5	25,06	11,35	7,75	6,45	35,25

Source: The authors

3.3. Kinetics of roasting at various temperatures

Figs. 4 and 5 show the comparative results of roasting through carbothermic reduction applied both to R1 concentrates obtained with rougher flotation and D1 with differential flotation. R1 and D1´s behavior are taken as a basis.
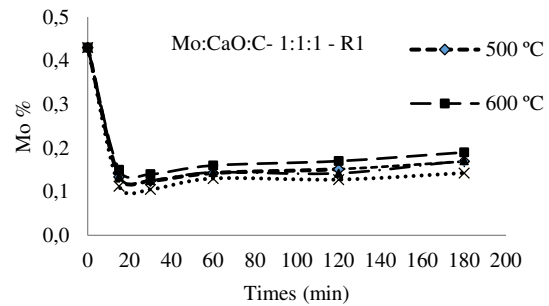
Figure 4. Temperature variation for R1 samples.
Source: The authors.
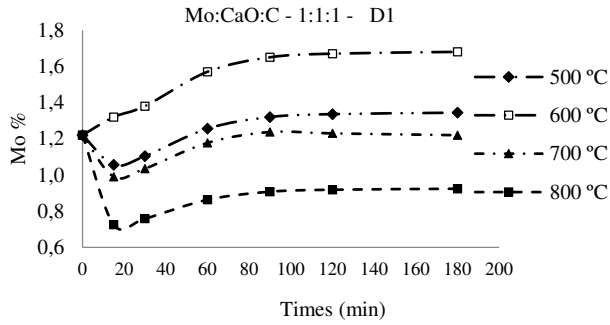
Figure 5. Temperature variation for D1 samples.
Source: The authors.

3.4. Molar ratio variation for CaO with respect to Mo and C (Mo:CaO:C) for R samples.

Once the working temperature has been established, work with molar variations is started. Figs. 6 to 9 show CaO molar variations with respect to Mo, C molar variation is left constant.

There are a variation of CaO molar ratios with respect to Mo and C (Mo:CaO:C). Variations are: 1:1:1; 1:0.5:1; 1:2:1; 1:3:1 at a constant temperature of 600° C for R1 to R5 samples.
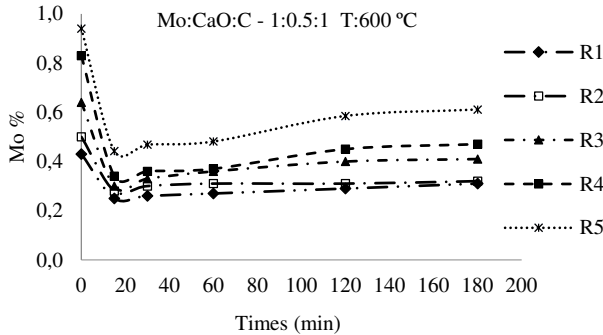
Figure 6. Mo percentage of variation versus time at 600° C for R samples.
Source: The authors.
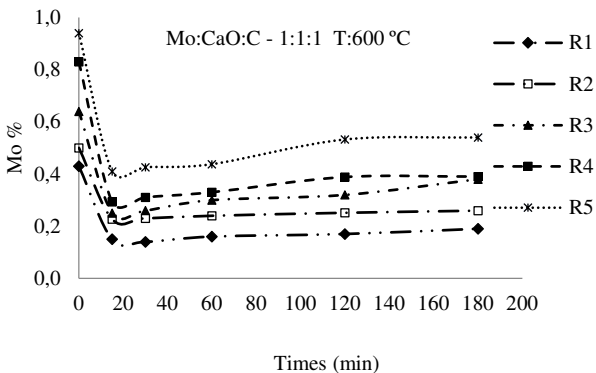
Figure 7. Mo percentage of variation versus time at 600° C for R samples.
Source: The authors.
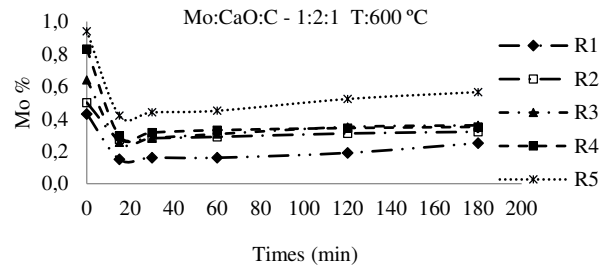
Figure 8. Mo percentage of variation versus time at 600° C for R samples.
Source: The authors.
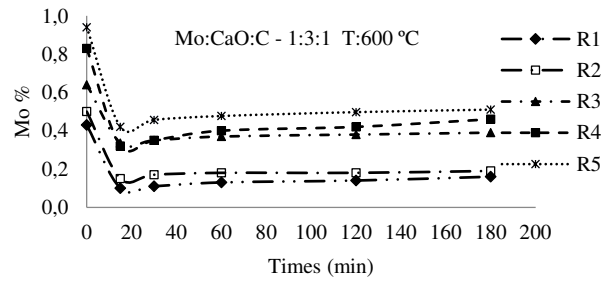
Figure 9. Mo percentage of variation versus time at 600° C for R samples.
Source: The authors.

3.5. Variation of molar ratios for C compared to Mo and CaO (Mo:CaO:C) for R samples

Once the CaO optimal molar ratio has been set up as 0.5, at which point a better molybdenum recovery is obtained, the molar ratio for C starts to altered.

Variation of molar ratios for C with respect to Mo and CaO (Mo:CaO:C) are: 1:0.5:1; 1:0.5:0.5; 1:0.5:2; 1:0.5:3, as shown in detail below in Figs. 10 to 13.

3.6. Variation of molar ratios for C, with respect to Mo and CaO (Mo:CaO:C) for D samples

For concentrates produced with differential flotation, we kept the same optimal conditions as those imposed on concentrates produced with rougher flotation. These conditions are the following: temperature: 600° C, molar ratio: 1:0.5:1, and an optimal time of 90 minutes. The tests performed are shown in Fig. 13.
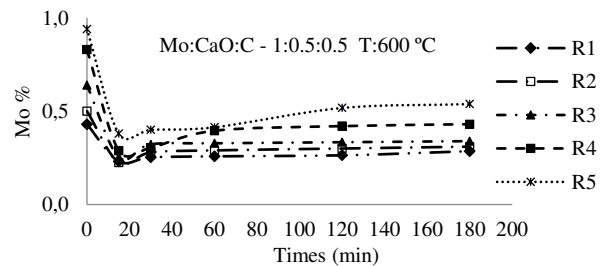
Figure 10. Variation of the Mo percentage vs time at 600°C for R samples.
Source: The authors.
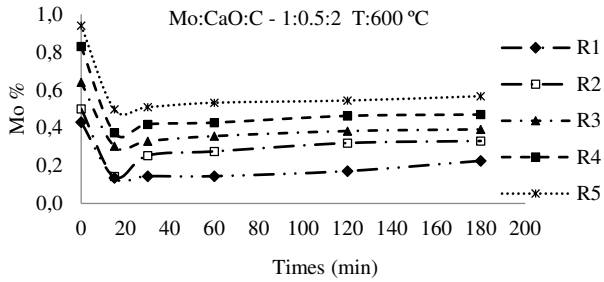
Figure 11. Variation of the Mo percentage vs time at 600 °C for R samples. Source: The authors.
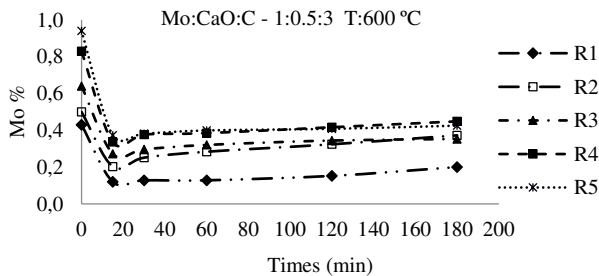
Figure 12. Variation of the Mo percentage vs time at 600° C for R samples. Source: The authors.
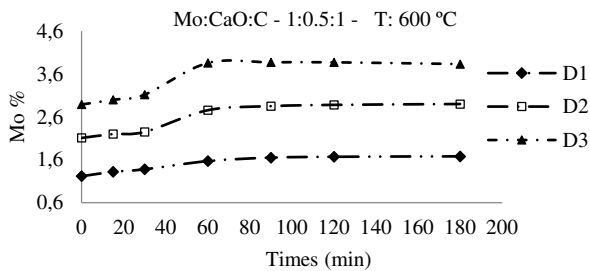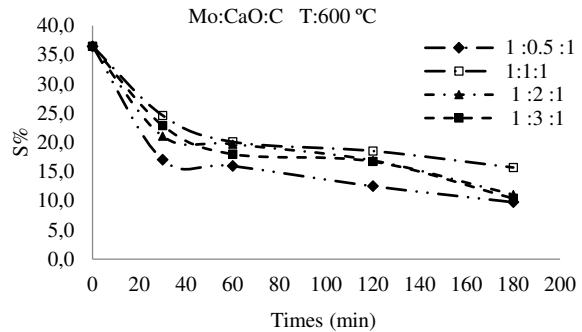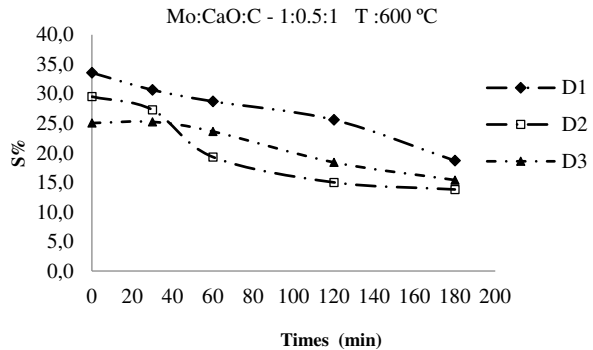

Figure 13. Variation of the Mo percentage vs time at 600° C for D samples.
Source: The authors.

The response from the application of roasting to differential flotation is quite different from that obtained from its application to rougher flotation; rather good recoveries are obtained. This indicates that separation of molybdenum with high concentrations of copper and iron leads to better results when obtaining MoO₃.

3.7. Results of Mo recovery in D samples

The results obtained show that there is no increase in productivity in those concentrates treated with simple flotation productivity (R1 to R5), whereas those treated with differential flotation (D1 to D3) show an increase higher than 35 % with respect to the initial Mo value. These results are shown in detail in Table 3.

Table 3.
Recovery values for "D" samples

Samples	1.1.1	Recovery (%)
D1		35.25
D2		33.64
D3		32.66

Source: The authors.

Figure 14. Variation of the S percentage vs time at 600° C for R samples. Source: The authors.

Figure 15. Variation of the S percentage vs time at 600° C for D samples. Source: The authors.

3.8. Results for sulfur values of samples R and D

Below are ~~Then~~ the results of chemical analysis for simple flotation productivity, Fig. 14, for the concentrates obtained from the differential flotation, Fig. 15.

When the sulfur capture agent, CaO, range was analyzed for each sample "R", variations were observed and studied.

The results are shown in Fig. 14. The figure shows molar CaO variations under the following stoichiometric conditions Mo:CaO:C: 1: 0.5:1; 1:1:1; 1:2:1 and 1:3:1, all evaluated at 600°C.

The graphs indicate that an increase in CaO stoichiometric conditions does not increase the value of the reactant fraction. Because the function of retaining the CaO is S, once the optimum conditions for which maximum retention is reached is found, increases in molar ratios do not cause increases in the reactant fraction of Mo. It is worthwhile mentioning that the recovery that remains is due to the conditions set by 1:0.5:1, with respect to other variations.

4. Discussion and analysis of results

4.1. Temperature and time variations

Temperature is one of the determining variables in the roasting process to obtaining Mo. On the basis of a thermodynamic analysis we defined the possible and dominant chemical reactions, shown in Fig. 1, as well as in Equation 2. This agrees with the cited references [12, 13].

Fig. 4 shows that, initially, a considerable decrease exists in the Mo %, with values decreasing from 0.87% to 0.17%. Remarkable decreases take place in the first 20 min when roasting with carbothermic reduction was applied. This indicated that the formation of the various compounds quoted by other authors [7] took place.

Later, the Mo percentages are seen to oscillate from 20 to 80 min. These are specific for each of the temperatures studied (500 to 800 °C). The temperature that responded best was 600° C. As time elapses, beginning at approximately 80 minutes the Mo percentage starts to stabilize and its value becomes more constant. The Mo values obtained reach 0.20 %.

The situation is quite different for D samples, which show another behavior, as can be seen in Fig. 5. Higher percentages of Mo are obtained with D samples than they are with R samples.

The variations found for R samples are due to the formation of various compounds such as molybdates, molybdenum carbides, and molybdenum sulfates. Each of the compounds formed produce oscillations in the weights obtained. According to the various references [6, 8], this is accounted for by a complicated chemical reaction mechanism.

From the time and temperatures that are stated, the kinetic curves slowly increase. However, the 1% Mo value in the R samples does not prove to be optimal.

From the results obtained, it may be stated that in the roasting process, Cu and Fe high concentrations interfere with carbothermic reduction. This leads us to consider the roasting results as favorable.

4.2. Discussions on the behavior of variations in molar ratios for CaO with respect to Mo (Mo:CaO:C) for R samples.

When analyzing the sulfur scavenging agent CaO, oscillating variations may be observed for each of the R samples. The results obtained are shown in Figs. 6 to 9.

The function of CaO is to retain S. However, in order to find the optimal operational conditions in the value of its equimolar ratio, the % of Mo is always regarded as a reference. Therefore, in this work, sulfur retention values are not dealt with.

The graphs indicate that an increase in CaO stechiometric conditions does not improve the values of the reactant fraction or the reaction rate.

The recovery shown is for the 1:0.5:1 conditions in comparison with other variations. These conditions are shown in Fig. 6. The highest Mo values obtained ranged from 0.3 to 0.6 %. For the remaining molar ratios (Figs. 7 to 9), the

values found ranged from 0.1 to 0.5 % Mo.

When analyzing the C reducing agent, the work we undertook was similar to that performed with CaO: the results are shown in Figs. 10 to 12. A response quite similar to that of CaO can be observed. The difference noted when the amounts of C are increased is that Mo recovery improves for the1:0.5:1 condition and it is possible to obtain Mo values that range from 0.3 to 0.6%. For the remaining stoichiometric ratios of 1:0.5:0.5; 1:0.5:2 and 1:0.5:3, no improvements exist in Mo percentages higher than those obtained for the 1:0.5:1 conditions. Mo percentages are not higher than those obtained for the 1:0.5:1 conditions.

The similarity lies in there being a reduction in the Mo percentage during the first 20 minutes of the roasting process. This percentage then starts increasing and finally stabilizes for times longer than 30 minutes. According to the literature [13,14,15], this oscillation occurs because both the CaO and C work synergistically for molybdenum as well as for Cu and Fe.

4.3. Discussion of the molar ratio behavior for CaO and C with respect to Mo for D samples.

The roasting response when applied to concentrates produced with the use of differential flotation (D) is different from that obtained from concentrates produced with rougher flotation (R). These reach higher recoveries with values ranging from 32 to 35% and are shown in Table 3. For rougher (R) flotation, comparison is not possible as the results in the recovery values are not satisfactory.

This indicates that molybdenum separation from copper and iron gives rise to better results in MoO_3 recovery.

The optimal operational conditions for R samples are shown in Fig. 6. Those for D samples are shown in Fig. 12.

The results show that concentrates that used simple flotation or roughers (R1 a R5) do not increase productivity after being subjected to different operational conditions for roasting with carbothermic reduction. However, those concentrates obtained with differential flotation (D1 to D3) show a 35% increase -with respect to Mo- after being subjected to roasting with carbothermic reduction. This can be seen in detail in Table 3. Best recoveries were obtained for differential flotation.

This indicates that the behavior of differential flotation is very different to that of rougher flotation; the best recoveries are achieved in the former. We can conclude that the separation of high concentrations of molybdenum in copper and iron give rise to better results in the production of MoO_3.

4.4. Discussions on the sulfur values of samples R and D

Chemical analysis indicates that there is a decrease in the amount of sulfur in the roasting samples. This decline that can be seen is related to the mass loss of MoS_2 from the total system.

We can say, therefore, that the loss in the R% S in samples where carbothermic reduction was applied is greater, starting with a 34-40% of S and ending at approximately 10% (Fig. 14). This leads us to consider that the R has samples that are easily volatilized compounds throughout the temperature

range for which tests samples were performed. This is of great importance in explaining the differences in reduction.

For sample D we can refer to Fig. 15.It begins with a 30-35% S and ends with a 15-18% S. This indicates that sulfur retention was higher by raising the optimal working conditions and it is shown to contain lower concentrations. Cu% and Fe% weight losses are very different and contain no more volatile R samples.

5. Conclusions

Use of concentrates obtained with differential flotation processes, in comparison with rougher flotation processes, considerably improved molybdenum recovery after the concentrates were subjected to roasting with carbothermic reduction.

The recoveries obtained from the application of carbothermic reduction to concentrates produced with differential flotation were higher than those obtained with rougher flotation. Both samples (R y D) were subjected to identical operational conditions. As such, it can be concluded that a remarkable difference exists that favors the roasting process when concentration of elements such as Cu and Fe are reduced while attempting to recover Mo.

It was found that R samples subjected to toasting with carbothermic reduction had optimal operational conditions, for this condition the recovery the Mo was higher. The conditions were the following: Molar ratio specified for CaO and C was 1:0.5:1, working temperature was 600 °C, and optimal roasting time was 90 minutes. This indicates that the kinetics is coincident with the optimal values of work, for which higher recoveries are obtained for both samples as R such as D.

The reagent most easily affected by molar ratio variation when applying roasting with carbothermic reduction is CaO and not C. CaO plays a most important role as a sulfur scavenger agent.

On analyzing the results, it can be seen that another variable that it is very important to take into account is the separation of copper and iron from molybdenum prior to the roasting process in order to obtain the desired recovery. This results in the capture of sulfur that is most favored for concentrates from differential flotation that obtained for the roughers flotation, being coincident with behavior of Mo.

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