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TECHNICAL ARTICLE

Flotation Process Water Recycling Investigation for the Complex Draa Sfar Sulphide Ore, Morocco

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Abstract The effect of water quality on the flotation of a complex polymetallic sulphide ore was studied using a Doehlert-based experimental design. Flotation tests were carried out using a sulphide ore provided by the Guemassa-Marrakech mining company, Morocco (MCG), using simulated tailings dam water. The optimal results, with a galena recovery of 86% and selectivity, relative to chalcopyrite, sphalerite, and pyrrhotite at 58, 63, and 68%, respectively, were reached with a water quality of 5 mg/L Cu^{2+}, 13 mg/L Zn^{2+}, 1390 mg/L Ca^{2+}, 140 mg/L Mg^{2+}, 4130 mg/L SO_4^{2-}, and 13 mg/L PAX. These results indicate that fresh water usage could be reduced in lead circuit flotation at the MCG plant by substituting water from the tailings dam.

Keywords Lead flotation · Selectivity · Doehlert experimental design · Optimization.

Introduction

For economic and supply reasons, and to minimize the discharge of wastewater into the environment, mineral processing plants are increasingly reusing water to reduce fresh water demand (Johnson et al. 2002). This use of recycled water can positively or negatively affect selective flotation of minerals (Bıçak et al. 2012; Boujounoui et al. 2015; Dávila-Pulido et al. 2015; Ikumapayi et al. 2010, 2012; Levay et al. 2001; Lutandula and Mwana 2014; Wang and Yongjun Peng 2014). The main sources of the recycled process waters are tailings dams, thickener overflows, dewatering, and filtration units. This recycled water typically contains contaminants such as colloids (silicates, clays, precipitated metal hydroxides, etc.), base metal ions (Cu^{2+}, Zn^{2+}, Pb^{2+}, etc.), sulfite and sulfate (SO_3^{2-}, SO_4^{2-}), magnesium (Mg^{2+}), calcium (Ca^{2+}), sodium (Na^+), and potassium (K^+), as well as residual reagents such as frothers, collectors, and depressants (Levay et al. 2001; Slatter et al. 2009).

The Mining Company of Guemassa (MCG), located 30 km southwest of Marrakech, Morocco, uses a selective flotation flow sheet to produce a galena concentrate, a chalcopyrite concentrate, and finally, a sphalerite concentrate from a complex polymetallic sulphide ore. MCG mostly uses fresh water from the mine and a dam located a few kilometers from the plant. According to data from the Haouz Tensift Basin Agency (HTBA), the Marrakech region has a semi-arid to arid climate, with low and irregular rainfall (250 mm/year) and high evaporation (2500 mm/year). This situation caused us to investigate ways to reduce the use of freshwater in flotation operations. The aim of this study was to assess the possibility of recycling water from a tailings dam for the MCG lead flotation circuit. Flotation tests were carried out simulating plant operating conditions and reagents, using synthesized MCG tailings water. Flotation experiments were performed on the natural complex sulphide ore provided by MCG.

Response surface methodology (Doehlert uniform shell design; Doehlert 1970) and desirability function were used to determine the areas of acceptable galena

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recovery and selectivity toward sphalerite, chalcopyrite, and pyrrhotite. This experimental design was selected because it required fewer experiments than others (composite or Box–Behnken). It also allowed for selection of different levels (e.g. 3, 5, and 7) for each variable, adding new factors without altering the quality of the design, the interpolation of useful responses, and presenting a uniform distribution of experimental points in the experimental domain.

Experimental

Materials

Flotation tests were carried out at the laboratory on a representative sample of a complex sulphide ore of the Draa Sfar Mine (Morocco), processed by MCG. The ore used was the same as Abidi et al. (2014) and Boujounoui et al. (2015). It is composed of about 6.43% sphalerite (Sp: ZnS), 2.22% galena (Gl: PbS), 0.95% chalcopyrite (Cp: $CuFeS_2$), 41.57% pyrrhotite (Po: Fe_9S_{10}), and 48.82% gangue (Gg), consisting mainly of quartz, talc, chlorite, calcite, siderite, and ankerite.

A series of synthetic water samples were synthesized to represent the water currently discarded in tailings dam of MCG site by mixing Marrakech drinking water with appropriate quantities of six components (Cu^{2+}, Zn^{2+}, Ca^{2+}, Mg^{2+}, SO_4^{2-}, and PAX).The reagents used were $CaCl_2$, 97%, and $ZnSO_4 \cdot 7H_2O$, 98%, from Prolabo; Na_2SO_4, 99%, from Fluka; $Cu(NO_3)_2 \cdot 3H_2O$, 99.5%, from Merck and $Mg(NO_3)_2 \cdot 6H_2O$, (99–102% from Panreac). Flotation reagents (Aerophine 3418A, PAX, and methyl isobutyl carbinol, MIBC) were provided by MCG.

Methods

Sample Preparation

A representative sample was taken from the feed belt of the primary MCG ball mill flotation plant and crushed to 2 mm using roll crushers in the laboratory. The sample was then divided into 1 kg batch samples for the flotation experiments. The batch samples were stored in vacuum sealed bags to prevent oxidation of the sulphide minerals. Prior to each flotation test, the batch samples were milled in 500 mL of synthetic water using a Denver carbon steel ball mill of 9.5 L internal volume, for 10 min, which corresponds to d_{80} finer than 100 μm.

Flotation Experiments

Flotation experiments were carried out in a 5 L Denver flotation cell. Initial solid concentration was about 18% by weight. The pulp level was constantly adjusted by the addition of synthetic water of suitable quality.

The natural pH was about 7. NaOH was used to adjust the pH of all tests to 11.3. Sodium cyanide (NaCN) was used as a depressing reagent of Sp, Cp, and Po, for all tests, with a specific consumption of 350 g/t. 40 g/t of diisobutyl phosphinate (Aerophine 3418A) and 40 g/t of MIBC were used as galena collector and frother, respectively. The impeller rotation speed was fixed at 1000 rpm. The flotation time was 10 min for each test and the concentrate was recovered by automatic scraping every 30 s. All concentrates and tails were filtered, dried, weighed, and then analyzed by atomic absorption spectroscopy for Cu, Pb, Zn, and Fe. Metal recovery to the concentrates was calculated according to Eq. (1):

$$R = 100\frac{Ct_c}{At_f} \tag{1}$$

where R (%) is the metal recovery, t_c (%) is the concentrate metal grade, t_f (%) is the feed metal grade, C is the concentrate weight, and A is the feed weight.

Design of Experiments

Response surface methodology (MRE) (Baçaoui et al. 2002; Ennaciri et al. 2014) was used to optimize operating conditions concerning water quality and selective flotation of galena. This method allows one to search for optimum levels of various factors to achieve a desired response level. The Doehlert uniform shell design was used to assess the relationship between the six factors influencing lead flotation (Cu^{2+}, Zn^{2+}, Ca^{2+}, Mg^{2+}, SO_4^{2-},and PAX) (Boujounoui et al. 2015), coded respectively X_1, X_2, X_3, X_4, X_5 and X_6, and the studied responses [recovery of galena (Y1, R_{Pb}) and its selectivity toward chalcopyrite (Y2, difference between Gl and Cp recoveries: $R_{(Pb-Cu)}$), sphalerite (Y3, difference between Gl and Sp recoveries: $R_{(Pb-Zn)}$), and Po (Y4, difference between Gl and Po recoveries: $R_{(Pb-Fe)}$)].

The Doehlert matrixes (Doehlert 1970) are asymmetrical, so the factors do not have the same number of levels and the number of distinct experimental treatments follows Eq. 2:

$$N = K^2 + K + 1 \tag{2}$$

with N being the number of tests and K, the number of factors. In this study, with six factors, Eq. 2 corresponds to 43 tests to be performed. Six additional center tests were performed to calculate both the variance of experimental error and the reproducibility of the testing data. A second order polynomial model was used, which contains only the terms (bi) and (bij), which reflect the different effects (main and interactions) of factors on the four studied responses (Y1, Y2, Y3, and Y4).The model can be written as (Eq. 3):

$$\begin{aligned}Y = {} & b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_5X_5 + b_6X_6 \\ & + b_{11}{X_1}^2 + b_{22}{X_2}^2 + b_{33}{X_3}^2 + b_{44}{X_4}^2 + b_{55}{X_5}^2 + b_{66}{X_6}^2 \\ & + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 + b_{14}X_1X_4 + b_{24}X_2X_4 \\ & + b_{34}X_3X_4 + b_{15}X_1X_5 + b_{25}X_2X_5 + b_{35}X_3X_5 + b_{45}X_4X_5 \\ & + b_{16}X_1X_6 + b_{26}X_2X_6 + b_{36}X_3X_6 + b_{46}X_4X_6 + b_{56}X_5X_6\end{aligned} \tag{3}$$

where Y: studied response; X_i: investigated factor (i varies from 1 to 10); b_0: a constant; bi: main effect of the factor i; and bij: interaction coefficients between i and j factors;

The experimental design was developed using Nemrodw software (Mathieu et al. 2000). The experimental sequence of the Doehlert design was randomized to minimize the effects of uncontrolled factors. The Doehlert experimental design and responses are given in Table 1.

Results and Discussion

From the results of Table 1, one can see that galena recovery ranged between 35.69 and 74.33%, the maximum selectivity between galena and chalcopyrite was about 47.31% (test 29), and the worst separation was −9.86% (test 24). The selectivity of galena toward sphalerite ranged between 21.51 and 58.29% and the selectivity of galena toward pyrrhotite ranged between 11.87 and 62.20%.

The estimation coefficients of the postulated model (Eq. 3) were determined by the least-squares method using Nemrodw software. The interpretation of the coefficients, main effects of factors (bi), and interactive effects (bij), were performed from statistical tests on the coefficients using the estimated variance from replicates and the Student's test (tα/2,d). Factors with a significance level higher than 95% (p value <0.05) were considered statistically significant. Positive coefficients (bi or bij) indicate that the variables increased the studied response (Yi) and vice versa.

The considered mathematical models were validated using analysis of variance (ANOVA), (F-test), and checked by the correlation coefficient (R^2) (Haider and Pakshirajan 2007). When R^2values are close to 1, the model offers an appropriate explanation of the variability of experimental values to the predicted values (Liu and Wang 2007). An adjusted determination coefficient (R^2Adj) was used to measure the proportion of the total observed variability described by the model. After validation, the different models were used to graphically represent the response surfaces in the domain of interest (isoresponse curves) to interpret the results.

Analysis of Galena Recovery (Y1)

Study of factors and estimation of coefficients

From Table 2, one can see that all of the factors are involved in different interactions except for PAX. Moreover, the most important interactions were: Ca^{2+}/Mg^{2+} ($b_{34}=37.118$), $Ca^{2+}/{SO_4}^{2-}$ ($b_{35} = -17.945$), Cu^{2+}/Ca^{2+} ($b_{13} = -17.365$), Zn^{2+}/Ca^{2+} ($b_{23}=15.315$), Zn^{2+}/ Mg^{2+} ($b_{24} = -13.761$) and Cu^{2+}/Zn^{2+} ($b_{12}=12.165$). The galena recovery can so be described by Eq. 4:

$$\begin{aligned}Y1 = {} & 38.532 + 13.198X_1 + 4.810X_2 - 7.325X_3 \\ & - 5.927X_6 + 12.165X_1X_2 - 17.365X_1X_3 + 15.315X_2X_3 \\ & - 13.761X_2X_4 + 37.118X_3X_4 - 17.945X_3X_5 \\ & + 13.873X_1X_1 + 17.217X_2X_2 + 34.519X_3X_3 \\ & + 22.011X_4X_4 + 23.551X_5X_5 + 18.488X_6X_6\end{aligned} \tag{4}$$

The galena recovery model presents a high determination coefficient $R^2=0.93$, explaining 93% of the variability in the response. The adjusted determination coefficient (Adj. $R_A^2 = 0.80$) and F-test = 7.12 were also satisfactory and confirmed the significance of the model.

Three-Dimensional (3D) Response Surface and Isoresponse Curves for Galena Recovery (Y1)

3D response surface plots were used to visualize the relationship between the response and experimental levels of each variable and the type of interactions between variables to deduce the operating conditions leading to optimal response (Tanyildizi et al. 2005). Figure 1a shows that the highest galena recovery (≈74.33%) was achieved with the greatest levels of Cu^{2+} (X_1) and Zn^{2+} (X_2). These two ions have complementary effects; Cu^{2+} activates galena by adsorption on the mineral surface as Cu^{2+} and Cu $(OH)_2$ (Chandra and Gerson 2009; Fornasiero and Ralston 2006), and Zn^{2+}, in the presence of CN^-, depresses chalcopyrite, sphalerite, and pyrrhotite to ensure good galena selectivity (Seke 2005).

Figure 1b shows that when X_3 and X_1 are at opposite levels, galena recovery is relatively high. However, the highest recoveries were reached when the copper level was high and the calcium was at its lowest. This could be due to adsorption of Ca^{2+} and other metal ions from the used process water, which reduces negative surface charges and consequently affects xanthate adsorption on galena (Ikumapayi et al. 2012).

Table 1 Doehlert experimental design and results

No. Exp	Factors, mg/L						Responses, %			
	X1: Cu^{2+}	X2: Zn^{2+}	X3: Ca^{2+}	X4: Mg^{2+}	X5: SO_4^{2-}	X6: PAX	Y1	Y2	Y3	Y4
1	7.0	10	1600	150	3100	15	67.74	33.25	51.09	53.30
2	0.0	10	1600	150	3100	15	37.07	7.38	21.51	19.32
3	5.3	18.7	1600	150	3100	15	71.15	33.84	55.78	57.09
4	1.8	1.3	1600	150	3100	15	49.21	12.56	31.96	32.50
5	5.3	1.3	1600	150	3100	15	55.10	14.60	41.10	34.43
6	1.8	18.7	1600	150	3100	15	44.19	13.38	28.79	26.56
7	5.3	12.9	1926.6	150	3100	15	67.60	−7.27	41.31	41.40
8	1.8	7.1	1273.4	150	3100	15	61.85	−3.17	41.41	47.75
9	5.3	7.1	1273.4	150	3100	15	59.72	17.19	41.83	38.68
10	3.5	15.8	1273.4	150	3100	15	66.62	−9.02	42.58	48.90
11	1.8	12.9	1926.6	150	3100	15	64.20	45.54	49.68	50.76
12	3.5	4.2	1926.6	150	3100	15	53.51	−6.97	34.35	32.30
13	5.3	12.9	1681.6	189.5	3100	15	64.69	37.10	48.76	53.49
14	1.8	7.1	1518.4	110.5	3100	15	56.03	16.50	40.61	31.67
15	5.3	7.1	1518.4	110.5	3100	15	68.99	32.86	54.70	54.33
16	3.5	15.8	1518.4	110.5	3100	15	71.01	20.21	49.04	54.01
17	3.5	10	1845	110.5	3100	15	69.82	34.48	53.61	54.70
18	1.8	12.9	1681.6	189.5	3100	15	59.82	33.01	45.69	49.03
19	3.5	4.2	1681.6	189.5	3100	15	68.86	27.45	47.43	50.37
20	3.5	10	1355	189.5	3100	15	50.82	41.19	37.27	33.07
21	5.3	12.9	1681.6	157.9	5346.3	15	61.78	31.04	47.77	47.81
22	1.8	7.1	1518.4	142.1	853.7	15	53.89	10.71	36.60	34.45
23	5.3	7.1	1518.4	142.1	853.7	15	67.75	37.20	50.73	56.08
24	3.5	15.8	1518.4	142.1	853.7	15	63.17	−9.86	38.00	45.69
25	3.5	10	1845	142.1	853.7	15	68.34	5.29	47.24	47.76
26	3.5	10	1600	181.6	853.7	15	58.28	12.84	42.76	35.66
27	1.8	12.9	1681.6	157.9	5346.3	15	58.14	26.71	39.45	32.67
28	3.5	4.2	1681.6	157.9	5346.3	15	45.98	13.88	28.75	25.77
29	3.5	10	1355	157.9	5346.3	15	74.33	47.31	56.10	59.51
30	3.5	10	1600	118.4	5346.3	15	62.60	45.88	47.41	49.64
31	5.3	12.9	1681.6	157.9	3474.4	18.8	62.05	25.43	44.51	45.90
32	1.8	7.1	1518.4	142.1	2725.6	11.2	57.78	20.43	43.59	36.03
33	5.3	7.1	1518.4	142.1	2725.6	11.2	62.05	30.77	47.83	39.71
34	3.5	15.8	1518.4	142.1	2725.6	11.2	73.51	41.93	58.29	62.20
35	3.5	10	1845	142.1	2725.6	11.2	59.17	20.70	44.42	36.94
36	3.5	10	1600	181.6	2725.6	11.2	60.22	45.87	47.39	50.60
37	3.5	10	1600	150	4971.9	11.2	65.93	37.51	51.13	52.51
38	1.8	12.9	1681.6	157.9	3474.4	18.8	57.98	42.38	42.77	45.14
39	3.5	4.2	1681.6	157.9	3474.4	18.8	45.47	29.23	33.73	32.07
40	3.5	10	1355	157.9	3474.4	18.8	67.42	−2.99	38.67	41.37
41	3.5	10	1600	118.4	3474.4	18.8	61.67	44.00	46.66	48.22
42	3.5	10	1600	150	1228.1	18.8	48.52	−0.49	28.08	27.68
43	3.5	10	1600	150	3100	15	35.69	12.03	22.45	18.96
44	3.5	10	1600	150	3100	15	41.31	11.85	24.87	23.19
45	3.5	10	1600	150	3100	15	38.07	12.65	24.22	23.08
46	3.5	10	1600	150	3100	15	48.05	17.21	30.70	27.68
47	3.5	10	1600	150	3100	15	39.06	13.87	25.10	21.87
48	3.5	10	1600	150	3100	15	37.55	5.00	21.53	11.87
49	3.5	10	1600	150	3100	15	45.00	8.62	26.48	15.58

Table 2 Estimated coefficients for the responses Y1, Y2, Y3 and Y4 (Signif.: significance,***: Statistically significant at the level 99.9% (p value <0.001),**: Statistically significant at the level 99% (p value <0.01),*: Statistically significant at the level 95% (p value <0.05), Coef.: coefficient, Estim. : estimation, Stan.: standard)

Coef	Y1				Y2				Y3				Y4			
	Coef. Estim	Stan. error	t.exp	Signif. %	Coef. Estim	Stan. error	t.exp	Signif. %	Coef. Estim	Stan. error	t.exp	Signif. %	Coef. Estim	Stan. error	t.exp	Signif. %
b0	38.532	2.368	16.27	<0.01***	12.705	1.148	11.07	0.0275***	24.108	0.744	32.41	<0.01***	21.727	2.437	8.92	<0.01***
b1	13.198	2.007	6.58	<0.01***	6.184	1.191	5.19	0.406**	13.163	0.772	17.05	<0.01***	16.229	2.529	6.42	<0.01***
b2	4.810	1.865	2.58	2.01*	3.746	1.107	3.38	1.99*	3.015	0.717	4.20	0.904**	5.829	2.350	2.48	2.29*
b3	−7.325	2.253	−3.25	0.531**	−2.958	1.338	−2.21	7.7	−6.012	0.867	−6.94	0.136**	−10.826	2.839	−3.81	0.145**
b4	−1.177	2.015	−0.58	57.4	3.843	1.073	3.58	1.63*	−1.332	0.775	−1.72	14.5	0.589	2.539	0.23	81.4
b5	−0.027	1.797	−0.02	98.5	19.754	1.190	16.60	<0.01***	1.525	0.691	2.21	7.7	0.953	2.264	0.42	68.1
b6	−5.927	1.996	−2.97	0.930**	−3.407	1.185	−2.87	3.47*	−8.782	0.768	−11.44	0.0248***	−7.841	2.516	−3.12	0.617**
b11	13.873	4.102	3.38	0.410**	7.610	2.296	3.31	2.14*	12.192	1.488	8.20	0.0746***	14.583	4.873	2.99	0.799**
b22	17.217	4.103	4.20	0.0833***	5.317	2.296	2.32	6.7	16.336	1.488	10.98	0.0281***	16.364	4.874	3.36	0.374**
b33	34.519	4.385	7.87	<0.01***	−3.914	2.473	−1.58	17.3	12.223	1.602	7.63	0.0964***	35.275	5.249	6.72	<0.01***
b44	22.011	4.065	5.41	<0.01***	26.427	2.036	12.98	0.0170***	33.015	1.473	22.42	<0.01***	35.287	4.733	7.46	<0.01***
b55	23.551	3.530	6.67	<0.01***	15.931	2.100	7.59	0.0983***	19.992	1.252	15.97	<0.01***	19.376	4.112	4.71	0.0234***
b66	18.488	3.600	5.14	0.0145***	14.644	1.976	7.41	0.107**	17.391	1.272	13.67	0.0147***	14.641	4.208	3.48	0.291**
b12	12.165	5.470	2.22	4.01*	10.635	3.247	3.28	2.23*	10.306	2.104	4.90	0.507**	16.513	6.892	2.40	2.71*
b13	−17.365	7.938	−2.19	4.30*	−71.348	4.712	−15.14	0.0112***	8.786	3.053	2.88	3.46*	−32.116	10.002	−3.21	0.508**
b23	15.315	6.777	2.26	3.75*	42.132	4.023	10.47	0.0327***	−1.616	2.607	−0.62	56.6	21.422	8.539	2.51	2.16*
b14	−5.076	6.488	−0.78	45.2	6.776	3.851	1.76	13.7	−13.001	2.496	−5.21	0.401**	−9.249	8.175	−1.13	27.3
b24	−13.761	6.399	−2.15	4.62*	−2.896	3.798	−0.76	48.4	0.770	2.461	0.31	76.2	−12.269	8.063	−1.52	14.3
b34	37.118	9.143	4.06	0.108**	−20.820	3.825	−5.44	0.339**	−14.274	3.517	−4.06	1.03*	−9.879	11.137	−0.89	39.1
b15	−5.520	6.559	−0.84	41.8	−24.786	5.058	−4.90	0.506**	−7.253	2.523	−2.87	3.47*	0.006	8.265	0.00	99.5
b25	8.689	6.501	1.34	19.9	38.831	4.285	9.06	0.0527***	17.111	2.500	6.84	0.143**	7.992	8.191	0.98	34.5
b35	−17.945	6.620	−2.71	1.55*	−4.130	4.045	−1.02	35.6	−16.271	2.546	−6.39	0.185**	−8.454	8.428	−1.00	33.2
b45	2.101	6.603	0.32	75.2	7.095	4.005	1.77	13.5	8.627	2.540	3.40	1.96*	9.837	8.267	1.19	24.9
b16	14.185	8.579	1.65	11.6	−0.032	3.938	−0.01	99.0	10.608	3.211	3.30	2.17*	24.768	10.809	2.29	3.34*
b26	−7.824	7.281	−1.07	30.0	−26.898	3.899	−6.90	0.139**	−11.482	2.766	−4.15	0.946**	−25.031	9.174	−2.73	1.38*
b36	−5.118	6.953	−0.74	47.9	58.873	5.562	10.58	0.0316***	4.398	2.700	1.63	16.3	11.884	8.785	1.35	19.1
b46	−4.593	6.875	−0.67	52.1	−23.915	4.036	−5.93	0.246**	1.369	2.616	0.52	62.6	−12.121	8.606	−1.41	17.4
b56	3.867	6.720	0.58	58.0	19.017	4.034	4.71	0.586**	6.043	2.573	2.35	6.5	−3.556	8.493	−0.42	68.3

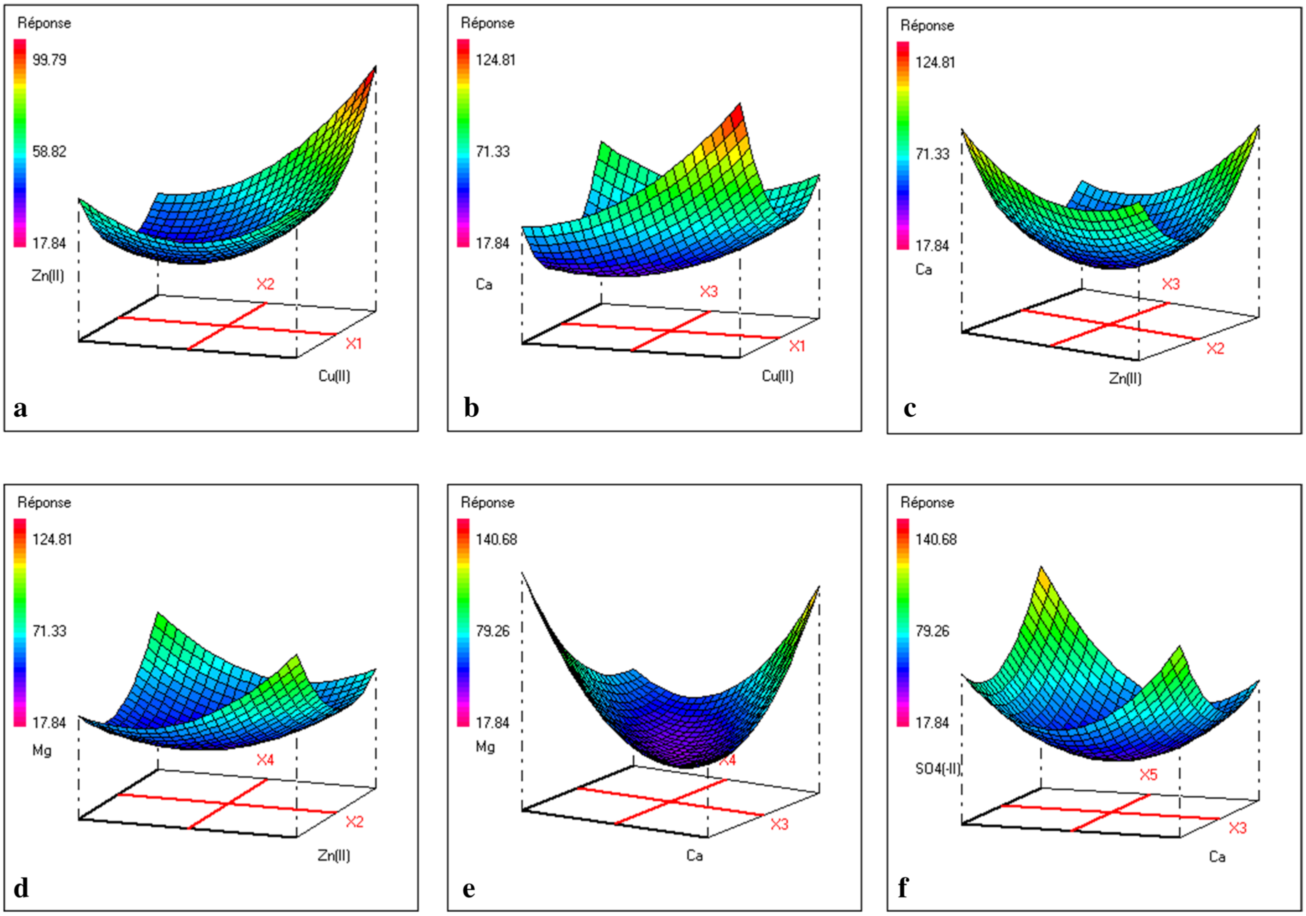

Fig. 1 Galena recovery (Y1; %) vs.: **a** Cu^{2+} and Zn^{2+}; **b** Cu^{2+} and Ca^{2+}; **c** Zn^{2+} and Ca^{2+}; **d** Zn^{2+} and Mg^{2+}; **e** Ca^{2+} and Mg^{2+}; **f** Ca^{2+} and SO_4^{2-}

Galena recovery increased with increased Zn^{2+} and Ca^{2+} and decreased Mg^{2+} concentrations, and with decreased Zn^{2+} and Ca^{2+} and increased Mg^{2+} (Fig. 1c, d). According to these results, Zn^{2+} is considered as an active ion, once it contacts Cu^{2+}, Ca^{2+}, and Mg^{2+}, and with other ions present in the medium. Its effect on galena recovery depends on its concentration, as well as the other coexisting ions.

The maximum galena recovery (Fig. 1e) was achieved with the lowest Ca^{2+} and Mg^{2+} concentrations. This shows that water hardness negatively affects galena recovery (Lascelles et al. 2003).

From the recovery of galena as a function of the interaction X_3 and X_5 (Ca^{2+}/SO_4^{2-}) (Fig. 1f), one can see that greater SO_4^{2-} concentrations and lesser Ca^{2+} concentrations enhance galena recovery. The presence of Ca^{2+} in process water with numerous species, including SO_3^{2-} and SO_4^{2-}, could form hydrophilic layers on the surface mineral ($Ca–SO_4$ or $Ca–SO_3$), reducing collector adsorption on the mineral surfaces (Ikumapayi et al. 2012; Lefèvre and Fédoroff 2006; Wu et al. 2002). The decreased Ca^{2+} concentration prevents $CaSO_4$ formation on the mineral surface and promotes galena recovery.

Analysis of the Selectivity between Galena and Chalcopyrite (Y2)

Study of Factors and Estimation of Coefficients

The coefficients estimated from the results are displayed in Table 2. Analysis shows that all of the factors were involved in different interactions. Furthermore, the more significant interactive effects with a significance level of 99% (p value <0.01) were: Cu^{2+}/Ca^{2+}(b_{13} = −71.348), Ca^{2+}/PAX ($b_{36}=58.873$), Zn^{2+}/Ca^{2+}($b_{23}=42.132$), Zn^{2+}/SO_4^{2-} ($b_{25}=38.831$), Zn^{2+}/PAX ($b_{26}=$ −26.898), Cu^{2+}/SO_4^{2-} (b_{15} = −24.786), Mg^{2+}/PAX ($b_{46}=$ −23.915), Ca^{2+}/Mg^{2+} (b_{34} = −20.820), and SO_4^{2-}/PAX ($b_{56}=19.017$). The least significant interaction was between Cu and Zn (Cu^{2+}/Zn^{2+}) ($b_{12}=10.635$), with a significance level of 95% (p value <0.05). From these results, the selectivity of galena toward chalcopyrite (response Y2), can be described by Eq. 5:

$$\begin{aligned} Y2 = {} & 12.705 + 6.184X_1 + 3.746X_2 + 3.843X_4 \\ & + 19.754X_5 - 3.407X_6 + 10.635X_1X_2 \\ & - 71.348X_1X_3 - 24.786X_1X_5 + 42.132X_2X_3 \\ & + 38.831X_2X_5 - 26.898X_2X_6 - 20.820X_3X_4 \\ & + 58.873X_3X_6 - 23.915X_4X_6 + 19.017X_5X_6 \\ & + 7.610X_1X_1 + 26.427X_4X_4 + 15.931X_5X_5 \\ & + 14.644X_6X_6 \end{aligned} \tag{5}$$

The correlation between the theoretical and experimental responses was satisfactory: $R^2 = 0.94$, $R^2_{Adj} = 0.85$, and F-test = 55.65.

Three-dimensional (3D) Response Surface and Isoresponse Curves for Response (Y2)

From the results of figure (2a), one can see that the lack of selectivity of galena toward chalcopyrite was attained with the maximum concentration of Cu^{2+} and minimum concentration of Ca^{2+}. The best selectivity (47.31%) was obtained with the minimum Cu^{2+} concentration and the maximum SO_4^{2-}concentration (Fig. 2b). This selectivity was more negatively affected when both Cu^{2+} and SO_4^{2-} were at their lowest levels.

The effect of Cu^{2+} on Gl/Cp selectivity depends on Ca^{2+}. Increasing Cu^{2+} and decreasing Ca^{2+} gives good selectivity because it allows good diffusion of Cu^{2+} and the best selective absorption on the mineral surface. Thus, high levels of Ca^{2+} are believed to depress galena and chalcopyrite (Ikumapayi et al. 2010, 2012).

Isoresponse curves (Fig. 2c–h) show that good selectivity of galena toward chalcopyrite was attained when concentrations of Zn^{2+} and Ca^{2+} were similar (high or low) (Fig. 2c), Zn^{2+} and SO_4^{2-} were elevated (Fig. 2d), Zn^{2+} was high and PAX was low (Fig. 2e), PAX and Ca^{2+} were at similar levels (high or low) (Fig. 2f), without PAX and high concentrations of Mg^{2+} (Fig. 2g), and when PAX and SO_4^{2-} concentrations were high (Fig. 2h). The competition of SO_4^{2-} ions with collector (PAX) adsorption on the mineral surface (Bıçak et al. 2012; Ikumapayi et al. 2010, 2012; Lefèvre and Fédoroff 2006; Wu et al. 2002) reduces its negative effect on galena selectivity toward chalcopyrite.

From these results, one can say that selectivity of galena toward chalcopyrite was improved with increased concentrations of Zn^{2+}, Cu^{2+}, and SO_4^{2-} and decreased concentrations of PAX and Ca^{2+}. This could be due to the depressing effects of Zn^{2+}, Ca^{2+}, and SO_4^{2-} on chalcopyrite, sphalerite, and pyrrhotite (Coetzer et al. 2003; Ikumapayi et al. 2010; Lefèvre and Fédoroff 2006; Seke 2005). PAX is a strong collector, and a decrease in its concentration might reduce its adsorption onto other minerals, especially chalcopyrite (Harmer et al. 2008; Wang and Yongjun Peng 2014).

Generally, water hardness (Ca^{2+}, Mg^{2+}, etc.) negatively influences sulphide flotation, either by reducing the hydrophobic surface of the particle (Fuerstenau et al. 1985; Rao and Finch 1989; Somasundaran et al. 2000), or by changing the particle surface charge (Espinosa-Gomez et al. 1987; Ikumapayi et al. 2012; Liu et al. 2013; Rao et al. 1988).

Analysis of the Selectivity between Galena and Sphalerite (Y3)

Study of Factors and Estimation of the Coefficients

From a statistical study of the $R_{(Pb-Zn)}$ (Y3) response (Table 2), all of the factors were involved in different interactions. The most significant interactions were Zn^{2+}/SO_4^{2-} ($b_{25} = 17.111$), Ca^{2+}/SO_4^{2-} ($b_{35} = -16.271$), Ca^{2+}/Mg^{2+} ($b_{34} = -14.274$), Cu^{2+}/Mg^{2+} ($b_{14} = -13.001$), Zn^{2+}/PAX ($b_{26} = -11.482$), Cu^{2+}/PAX ($b_{16} = 10.608$), Cu^{2+}/Zn^{2+} ($b_{12} = 10.306$),Cu^{2+}/Ca^{2+} ($b_{13} = 8.786$), Mg^{2+}/SO_4^{2-} ($b_{45} = 8.627$), and Cu^{2+}/SO_4^{2-} ($b_{15} = -7.253$). The response $R_{(Pb-Zn)}$ (Y3) as a function of these significant factors can be written as Eq. 6:

$$\begin{aligned} Y3 = {} & 24.108 + 13.163X_1 + 3.015X_2 - 6.012X_3 \\ & - 8.782X_6 + 10.306X_1X_2 + 8.786X_1X_3 \\ & - 13.001X_1X_4 - 7.253X_1X_5 + 10.608X_1X_6 \\ & + 17.111X_2X_5 - 11.482X_2X_6 - 14.274X_3X_4 \\ & - 16.271X_3X_5 + 8.627X_4X_5 + 12.192X_1X_1 \\ & + 16.336X_2X_2 + 12.223X_3X_3 + 33.015X_4X_4 \\ & + 19.992X_5X_5 + 17.391X_6X_6 \end{aligned} \tag{6}$$

The correlation between the theoretical and experimental responses calculated by Eq. 6 were satisfactory: $R^2 = 0.95$, $R^2_{Adj} = 0.87$, and F-test = 49.19.

Three-dimensional (3D) Response Surface and Isoresponse Curves for Response (Y3)

Figure 3a shows the 3D response surface curve of the selectivity between galena and sphalerite as a function of the interaction of X_1 and X_2 (Cu^{2+}/Zn^{2+}). It is evident that greater selectivity (58.29%) was achieved with higher levels of Cu^{2+} and Zn^{2+}. This can be explained by the depressing effect of both Cu^{2+} and Zn^{2+} on sphalerite flotation, due to the formation of hydrophilic layers ($Cu(OH)_2$, $Zn(OH)_2$) on the sphalerite surface in an alkaline medium (Cao and Liu 2006; Chandra and Gerson 2009; Fornasiero and

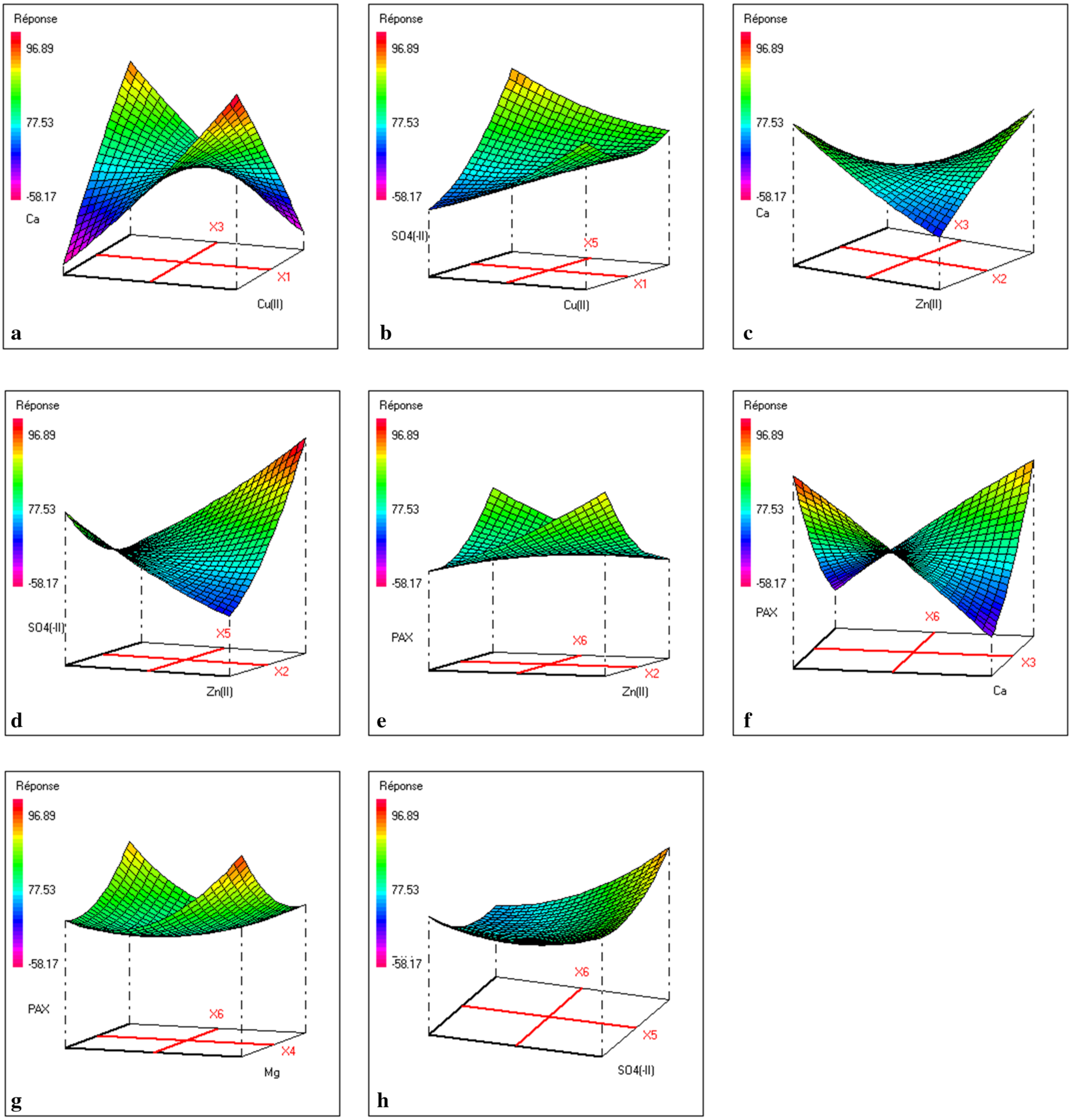

Fig. 2 Galena selectivity toward chalcopyrite (Y2; %) vs.: **a** $Cu^{2+}$and Ca^{2+}; **b** Cu^{2+} and SO_4^{2-}; **c** Zn^{2+} and Ca^{2+}; **d** Zn^{2+} and SO_4^{2-}; **e** Zn^{2+} and PAX; **f** $Ca^{2+}$and PAX; **g** Mg^{2+} and PAX; **h** SO_4^{2-} and PAX

Ralston 2006; Liu et al. 2013; Popov et al. 1989; Prestidge et al. 1997; Seke et al. 2005).

From Fig. 3b, c, it can be seen that selectivity between Gl and Sp increases with increased SO_4^{2-} and Zn^{2+} and decreased Ca^{2+} concentrations. Zinc sulfate ($ZnSO_4$) is well known to selectively depress sphalerite during complex sulphide ore flotation because it deactivates the sphalerite surface by preventing absorption of activating metal ions (Cu^{2+}) and by the formation of hydrophilic zinc hydroxide layers on the surface (Cao and Liu 2006; Seke et al. 2005). The presence of SO_4^{2-} in the process water with other species such as Ca^{2+} could form hydrophilic layers on the mineral surface (Ca–SO_4 and Ca–SO_3; Bıçak et al. 2012; Ikumapayi et al. 2010, 2012).

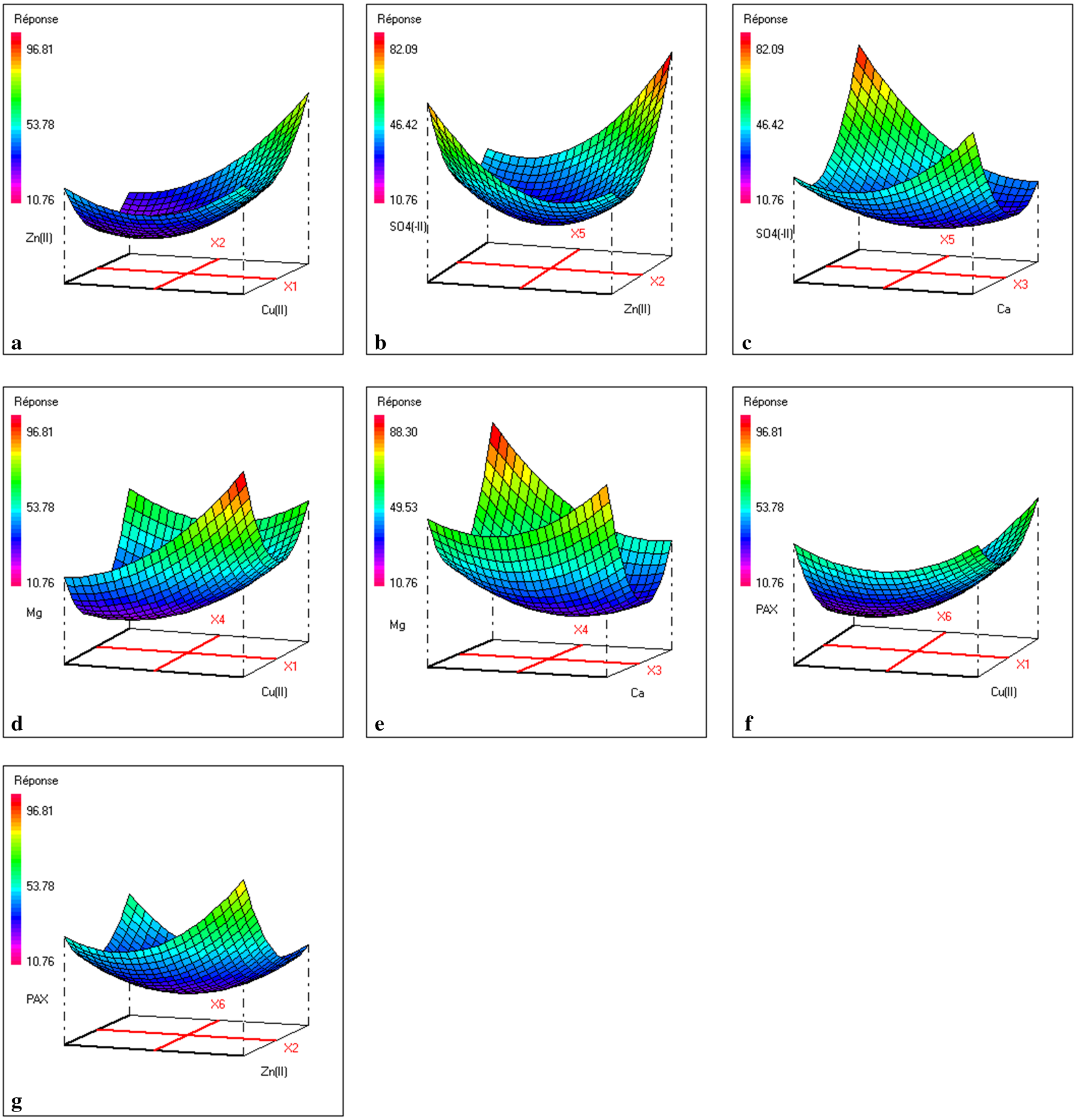

Fig. 3 Galena selectivity toward sphalerite (Y3; %) vs.: **a** Cu^{2+} and Zn^{2+}; **b** Zn^{2+} and $SO_4{}^{2-}$; **c** Ca^{2+} and $SO_4{}^{2-}$; **d** Cu^{2+} and Mg^{2+}; **e** Ca^{2+} and Mg^{2+}; **f** Cu^{2+} and PAX; **g** $Zn^{2+}$and PAX

Figure 3d, e show that the highest selectivity of galena/sphalerite was achieved with the highest level of Mg^{2+} and lowest of Ca^{2+}, and the highest level of Cu^{2+} and the lowest of Mg^{2+}. Lascelles et al. (2003) showed that sphalerite recovery was significantly reduced in the presence of Ca^{2+} and Mg^{2+}, due to the formation of metal hydroxides on the sphalerite surface. The decrease was more pronounced for Mg than for Ca at an alkaline pH. According to Bıçak et al. (2012) and Ikumapayi et al. (2012), increasing the Ca^{2+} concentration negatively affected sphalerite recovery, and its negative impact on galena recovery was more intense. Moreover, Dávila-Pulido et al. (2015) showed that Ca^{2+} in the form of $Ca(OH)^+$ could promote sphalerite flotation at alkaline pH because it increases collector absorption on the sphalerite surface.

Figure 3f, g show that decreased PAX concentrations and increased Zn^{2+} improved the selectivity between galena and sphalerite. For the interaction between Cu^{2+} and PAX, the best selectivity was reached when PAX concentrations were low and Cu^{2+} was high, or when both concentrations were high.

As mentioned above, $ZnSO_4$ is used as a sphalerite depressant in the separation of lead–zinc and copper–lead–zinc ores, especially in the presence of NaCN. Alkyl xanthates are commonly used as collectors for sphalerite flotation, to separate sphalerite from other valuable minerals (Harmer et al. 2008; Wang and Yongjun Peng 2014). Therefore, to increase selectivity between galena and sphalerite, one must increase the concentration of Zn^{2+} and decrease that of PAX.

At high pH, Cu^{2+} can absorb onto the sphalerite surface as $Cu(OH)_2$ colloids (Prestidge et al. 1997), depressing it by preventing the adsorption of the collector (Chandra and Gerson 2009; Fornasiero and Ralston 2006; Popov et al. 1989). To summarize, selectivity of galena from sphalerite was optimal with high Cu^{2+}, Zn^{2+}, and SO_4^{2-} concentrations, and low Ca^{2+}concentrations.

Analysis of the Selectivity between Galena and Pyrrhotite (Y4)

Study of Factors and Estimation of the Coefficients

Table 2 shows that all factors were involved in galena and pyrrhotite selectivity except for Mg^{2+} (X_4) and SO_4^{2-} (X_5). The five important interactions were: Cu^{2+}/Ca^{2+} (b_{13}= −32.116) with a significance level of 99% and Zn^{2+}/PAX (b_{26} = −25.031), Cu^{2+}/PAX (b_{16} = 24.768), Zn^{2+}/Ca^{2+} (b_{23} = 21.422), and Cu^{2+}/Zn^{2+} (b12 = 16.513),with a significance level of 95. The selectivity between galena and pyrrhotite can be described by Eq. 7. The R^2 coefficient of 0.91, the adjusted one (R^2_{Adj}) of 0.77, and F-test result of 6.55 confirm its significance.

$$\begin{aligned} Y4 = {} & 21.727 + 16.229X_1 + 5.829X_2 \\ & - 10.826X_3 - 7.841X_6 + 16.513X_1X_2 \\ & - 32.116X_1X_3 + 24.768X_1X_6 + 21.422X_2X_3 \\ & - 25.031X_2X_6 + 14.583X_1X_1 + 16.364X_2X_2 \\ & + 35.275X_3X_3 + 35.287X_4X_4 + 19.376X_5X_5 \\ & + 14.641X_6X_6 \end{aligned} \tag{7}$$

Three-dimensional (3D) Response Surface and Isoresponse Curves for Response (Y4)

Figure 4a–c show the 3D response surface curve of the selectivity between galena and pyrrhotite vs. the interactions X_1X_2 (Cu^{2+}/Zn^{2+}), X_1X_3 (Cu^{2+}/Ca^{2+}), and X_1X_6 (Cu^{2+}/PAX). It is obvious that the best selectivity between galena and pyrrhotite was achieved at higher Cu^{2+}, Zn^{2+}, and PAX concentrations and with less Ca^{2+}. Interactions of Zn^{2+}/Ca^{2+} and Zn^{2+}/PAX indicate that greater selectivity of galena toward pyrrhotite (Fig. 4d, e) was attained when both $Zn^{2+}$and Ca^{2+}were at high or low levels and when PAX was high.

Generally, pyrrhotite flotation strongly depends on pulp pH, the activator concentration (Cu^{2+}), and the presence of depressant elements (Zn^{2+}, Ca^{2+}, CN^-, SO_3^{2-}). Leppinen (1990) and Finkelstein (1997) showed that the recovery of pyrrhotite in alkaline conditions is low compared to acidic conditions. This could be due to the formation of hydrophilic layers ($Cu(OH)_2$, $Ca(OH)_2$, $CaCO_3$, etc.) on the mineral surface.

Multi-criteria Optimization using the Desirability Function

The objective of this study was to optimize the experimental conditions regarding water quality (Cu^{2+}, Zn^{2+}, Ca^{2+}, Mg^{2+}, SO_4^{2-}, PAX) for better galena flotation and good selectivity toward chalcopyrite, sphalerite, and pyrrhotite. To determine an acceptable compromise, responses were simultaneously optimized by using the desirability functions approach (Derringer and Suich 1980). This method first converts each estimated response Y_i into an individual scale free desirability function d_i that varies from 0 (outside of the desired limits, $Y_i(x) \le Y_i$; min) to 1 the target (desired) value (if $Y_i(x) \ge Y_i$; max), where Y_i; min and Y_i; max are the lower and upper acceptability bounds for response i, respectively. Once the function d_i (partial desirability of response Y_i) is defined for each response of interest, an overall objective function (D), representing the global desirability function, is calculated by determining the geometric mean of the individual desirability. Therefore, the function D over the experimental domain is calculated using Eq. 8 (Bruns et al. 2006):

$$D = \left(d_1 \cdot d_2 \cdot d_3 \cdot d_4\right)^{1/4} \tag{8}$$

The maximum of the function D gives the best global compromise in the studied domain and corresponds to optimal experimental conditions. The targeted values of galena recovery, and its selectivity toward chalcopyrite, sphalerite and pyrrhotite were 100%. Minimal acceptable values were 67, 40, 50, and 55%, of galena recovery, and its selectivity toward chalcopyrite, sphalerite, and pyrrhotite, respectively. The global desirability function D was calculated using Nemrodw software, yielding a global degree of satisfaction for the four responses of 38.30%.

The response surface corresponding to the global desirability function D is presented as contour and

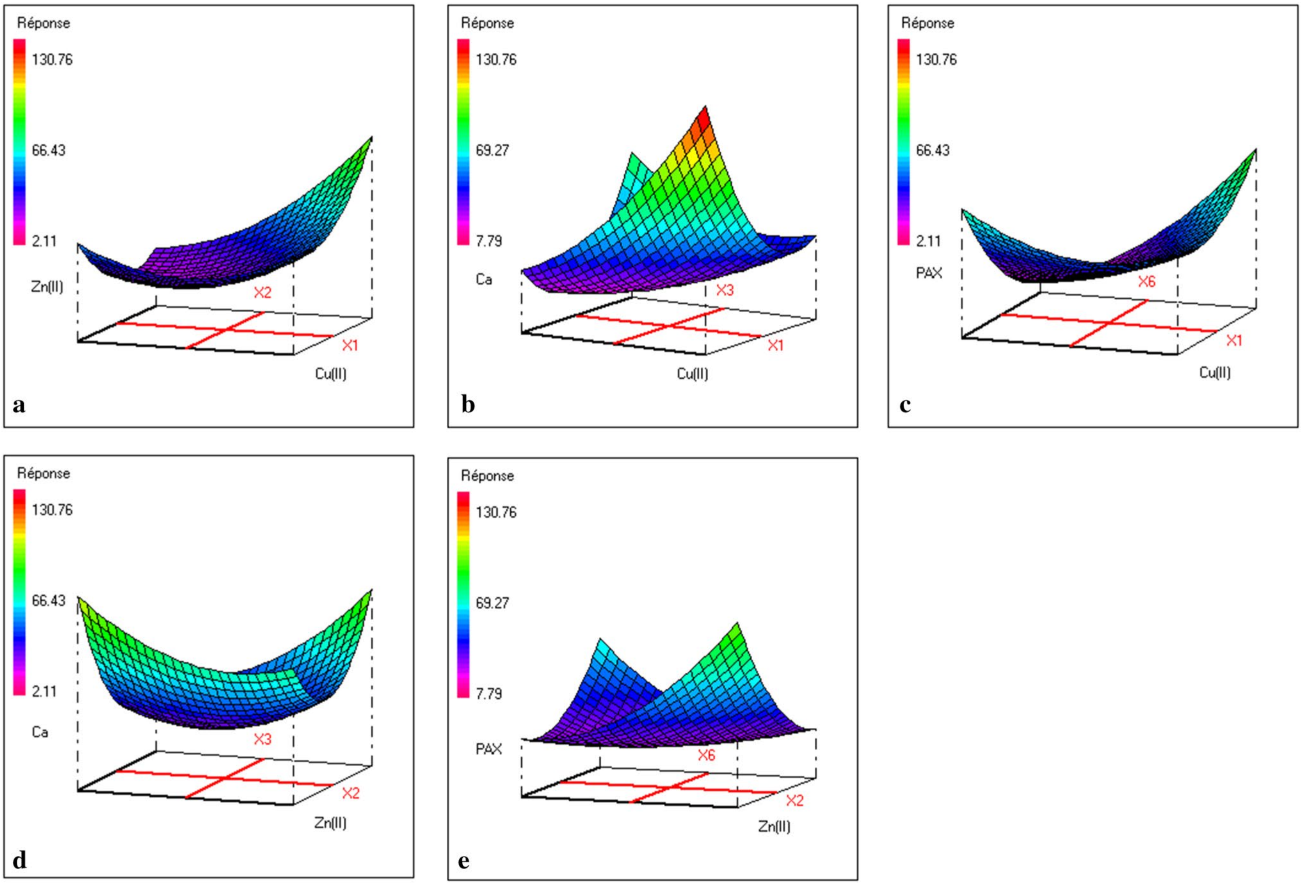

Fig. 4 Galena selectivity toward pyrrhotite (Y4; %) vs.: **a** Cu^{2+} and Zn^{2+}; **b** Cu^{2+} and Ca^{2+}; **c** Cu^{2+} and PAX; **d** Zn^{2+} and Ca^{2+}; **e** Zn^{2+} and PAX

three-dimensional plots. It can be seen from Fig. 5 that the acceptable compromise domain for better recovery of galena and its good selectivity relative to chalcopyrite, sphalerite, and pyrrhotite, was where concentrations of Ca^{2+}, Mg^{2+}, and PAX were low and those of Cu^{2+}, Zn^{2+}, and SO_4^{2-} were high.

The maximum recovery of galena (86%) and its maximum selectivity relative to chalcopyrite (58%), sphalerite (63%), and pyrrhotite (68%), were reached in the optimal experimental conditions of Cu^{2+} (5 mg/L), Zn^{2+} (13 mg/L), Ca^{2+} (1390 mg/L), Mg^{2+} (140 mg/L), SO_4^{2-} (4130 mg/L), and PAX (13 mg/L).

Conclusions

The Doehlert experimental design was used to assess the possible use of tailings dam water at the MCG site. Our optimization study of galena recovery and its selectivity toward chalcopyrite, sphalerite and pyrrhotite, using synthetic process water showed that:

- High concentrations of Zn^{2+}, Cu^{2+}, and SO_4^{2-} and low concentrations of PAX, Ca^{2+}, and Mg^{2+} ensure good galena floatability and better selectivity toward Cu, Zn, and Fe sulphides.
- Doehlert design, response surface methodology, and multi-criteria optimization using the desirability function were used to determine the best compromise zone for galena recovery (Y1), selectivity between galena and chalcopyrite (Y2), selectivity between galena and sphalerite (Y3), and selectivity between galena and pyrrhotite (Y4).
- The optimal operating conditions of galena flotation and its selectivity toward chalcopyrite, sphalerite, and pyrrhotite were: 5 mg/L of Cu^{2+}, 13 mg/L of Zn^{2+}, 1390 mg/L of Ca^{2+}, 140 mg/L of Mg^{2+}, 4130 mg/L of SO_4^{2-}, and 13 mg/L of PAX. At these conditions, galena recovery and its selectivity toward chalcopyrite, sphalerite and pyrrhotite were respectively 86, 58, 63, and 68%.

Based on these results, it can be seen that the use of fresh water can be reduced by using tailings dam water. The next step will be to assess the lead flotation process

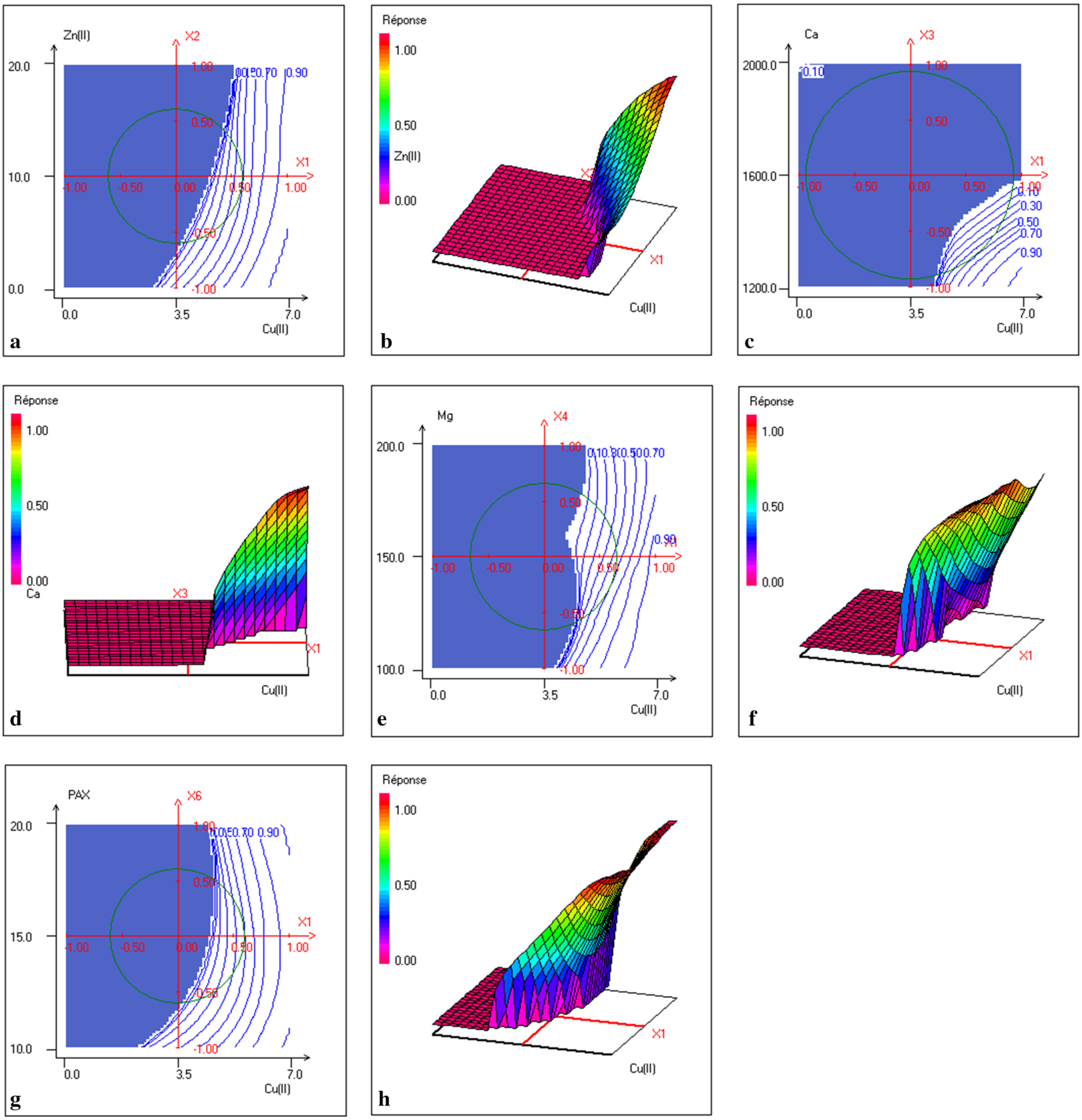

Fig. 5 Desirability function vs.: **a, b** $Zn^{2+}$and Cu^{2+}; **c, d** Ca^{2+} and Cu^{2+}; **e, f** Mg^{2+} and Cu^{2+}; **g, h** PAX and Cu^{2+}

with water made by mixing fresh water with water from the MCG tailings dam. Results will determine how much water can be effectively recycled.

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