Synthesis optimization and characterization of Na-jarosite and K-jarosite using hydrothermal method

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Introduction

$AM_3(SO_4)_2OH_6$

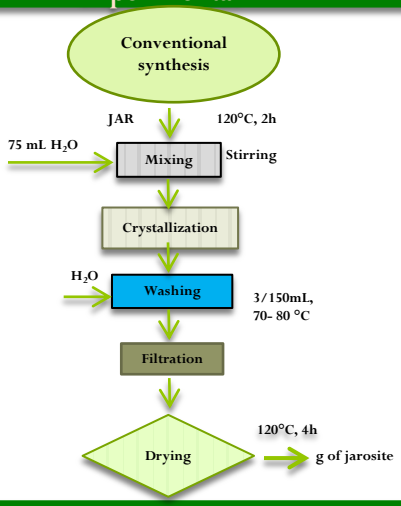
Where :
A: can be cations: Na^+, K^+, Rb^+, NH_4^+, H_3O^+, Ag^+, ½Pb^{2+} and ½ Hg^{2+}.
M_3: can be Fe3+ , V3+ , Al3+ otro.
SO_4: exchanged for hydroxyl (OH-) and M+ can be exchanged for (AsO_4^{3-}), F^-, and heavy metal cations[1].

"Jarosites are promising materials for formulating low-cost adsorbents and ionic exchangers to remove regulated contaminants in underground water".

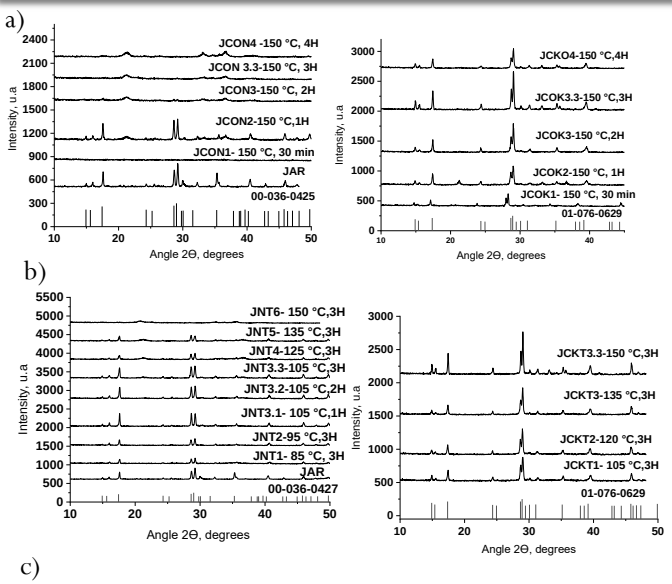
According to literature, the synthesis of jarosite involve conventional heating at a temperature range between 60-100 °C in acid media and long times (4 to 24h). Typically, an aqueous solution of Fe_2SO_4 and MOH (where M = Na or K) with a pH range of 1.5 to 2 is stirred at 95 ºC for 4h; however, the reported methods are imprecise [2].

Therefore, in the present work, we assess the effect of time, temperature and the molar ratio on crystallinity and yield of Na-jarosite and K-jarosite reaction to finding the optimum synthesis conditions maintaining high yield [3].

Experimental



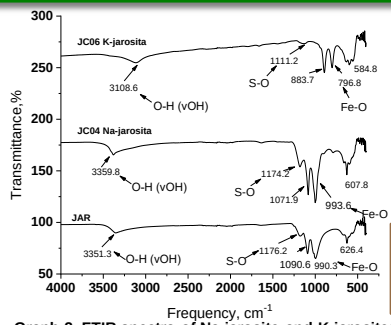
Results and Discussion



Graph 1. Optimization of synthesis; a) effect of time, b) effect of temperature and c) effect of molar ratio of MOH: Fe_2SO_4 on the crystallinity of Na-jarosite and K-jarosite

Table 1. Effect of time, temperature and molar ratio on the yield of Na-jarosite and K-jarosite.

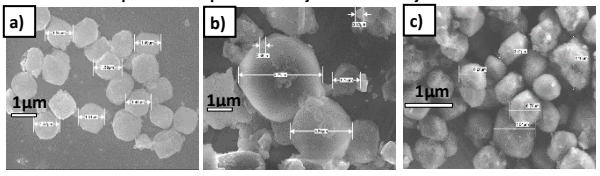
Sample	Yield %	Sample	Yield %	Sample	Yield %
Time		Temperature		Molar ratio of MOH: Fe_2SO_4	
JCON1	38.4	JNT3.3	87.5	JCN1	45.2
JCON3.3	87.5	JNT6	87.5	JCN5	81.1
JCOK1	86.2	JCTK1	85.0	JCK1	75.8
JCOK3.3	84.8	JCKT3.3	84.8	JCK5	99.8



K-jarosite FTIR spectrum has all bands with lower intensity and shifted towards lower frequencies[4].

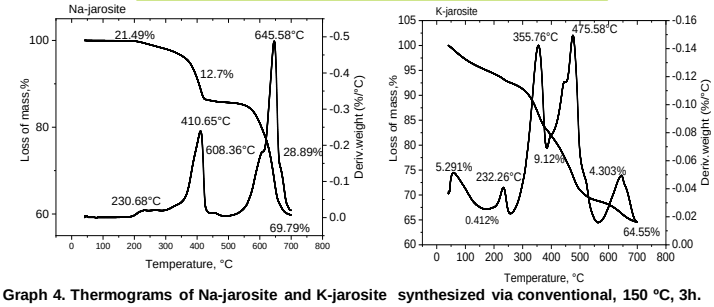
Na-jarosite and K-jarosite are particles with homogeneous pseudospheric morphology of approximately 1 to 1.5 µm.

Graph 2. FTIR spectra of Na-jarosite and K-jarosites



Graph 3. Micrographs of jarosites; a) JAR at 10000 X b) JCO4 Na-jarosite and c) JCO6 K-jarosite, both taken at 25,000x.

Na-jarosite is more stable than K-jarosite, its process of desyhroxylation and transformation of sulfates in to SO_2 occurs 510-700 °C, and in K-jarosite these occurs 260-550 °C, [5].



Graph 4. Thermograms of Na-jarosite and K-jarosite synthesized via conventional, 150 °C, 3h.

Conclusions

-DRX analysis indicated that the time,temperature and molar ratio have an important influence on the formation of jarosites and on its hydrothermal stability.
-Na-jarosite and K-jarosite were synthesized at 105 and 150 °C, both in 3h with yields of 87.5 and 84.8%, respectively.
-K-jarosite presented an advantage over Na-jarosite, it was synthesized in a wide interval of time, temperature, and KOH / Fe_2SO_4 molar ratio.
-The crystallinity and yield confirm that Na-jarosite and K-jarosite are hydrothermally stables.

References

[1] González-Ibarra AA, Nava-Alonso F, Fuentes-Aceituno JC, Uribe-Salas AJ. Min. Metall. Sect. B-Metall (2016) 52 135-142.
[2] D. Baron, C. D. Palmer. Solubility of jarosite at 4-35° C. Geoch. Cos. Acta (1996) 60185- 195.
[3] Zhao R, Li Y, Chan C. K, Synthesis of Jarosite and Vanadium Jarosite Analogues Using Microwave Hydrothermal Reaction and Evaluation of Composition-Dependent Electrochemical Properties. J. Phys. Chem. C (2016) 120 9702−9712.
[4] Islas H, Patiño F, Reyes M, Flores M.U, Reyes I.A, Palacios E. G, Ordoñez S, Flores VH. Rev. Latin. Am. Metal. Mat (2017) 37 228 – 236.