NANOCOLLOIDAL DISPERSIONS FOR IN SITU GENERATION "IN SITU" OF MAGENTA PIGMENTS OF CR-PSEUDOBROOKITE IN INKJET APPLICATIONS.

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1. INTRODUCTION

Pseudobrookite $TiFe_2O_5$ is a rare mineral, the name designating an AB_2O_5 structure for a variety of mineral and synthetic phases. The name of the mineral derives from its similar appearance to brookite, one of the titanium dioxide polymorphs. It is isostructural with armalcolite (lunar mineral named for *Arm*strong, *Al*drin and *Co*llins, the first moon explorers) $(Fe_{0.5}Mg_{0.5})\ Ti_2O_5$ and with 'karrooite', $MgTi_2O_5$ with ions coordinated by six oxygen atoms. The stability at high temperatures is associated with high entropy due to the disorder of cation mixing between two different cation-oxygen octahedra. Although these phases are metastable at low temperatures, kinetic transformation to stable oxides is generally so slow that phases like armalcolite can persist for billions of years. There are two synthesis strategies in the preparation of inkjet inks:(a) top-down for the generation of submicrometric pigment dispersions by means of ultragrinding, with high costs in equipment and energy, and (b) bottom-up for the generation of dispersions by non-conventional chemical procedures in ceramics involving aqueous or hydroalcoholic methods (1). This study describes inks generated by the following methods: aqueous CO (colloidal sol-gel), MOD (metal-organic colloid with polycarboxylate acids) and alcohol GP (sol-gel colloids from alkoxides).

2. EXPERIMENT AND RESULTS

The optimum sample (2) $(Fe_{1,9}Cr_{0,1})TiO_5$ was prepared by the conventional ceramic (CE) method from oxides (anatase in the case of Ti) and non-conventional CO methods (from nitrates and titanium butoxide peptized in an ammoniacal solution), MOD (with CO precursor with O.5 mol citric acid/mol pseudobrookite prior to its ammoniacal peptization) and GP (by hydrolysis in a closed atmosphere at ambient temperature of the stirred ethanolic solution of the above precursors). Figure 1 shows the appearance of the dispersions and the results and characterizations obtained by the deposition and glazing indicated.

3. CONCLUSIONS

The results indicate the generation of high intensity red-brown films by direct deposition of the inks, as well as of the colloids stabilized at 500ºC glazed on polyglycols applied on a 48 thread. The powders obtained through drying of the inks (Fig.1.a) crystallize NH_4NO_3 (AN in Fig.1.f) by the effect of the ammonium added on the nitrate solution except the GP powder which hydrolyses in the absence of ammonia producing an amorphous powder. Despite the CE sample presenting free hematite (indicated as H in the X-ray diffractograms (XRD) in Fig.1. at 1000ºC with lightly sintered particles (Fig.1.d), better shades of red are obtained (a*=18.8) (Fig.1.e) than in the highly sintered particles of the other methods.

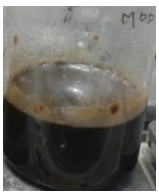
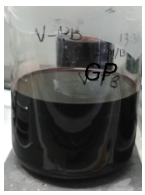
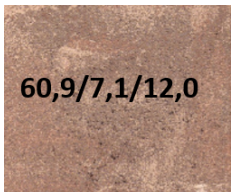
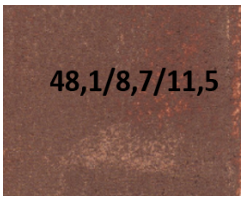
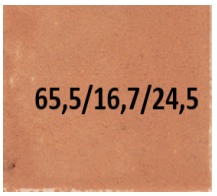
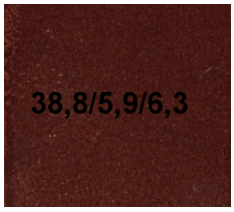
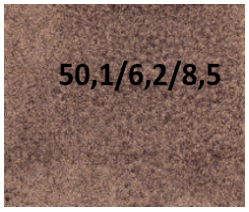
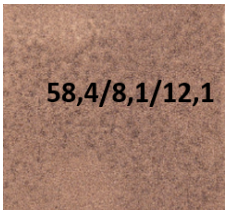
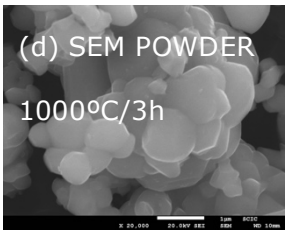
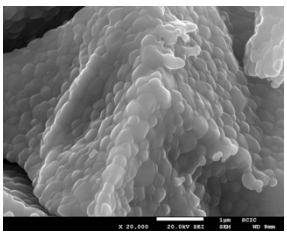
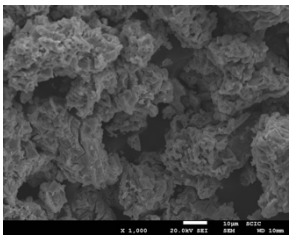
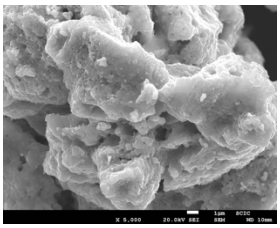
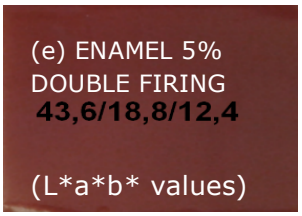
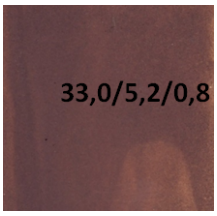
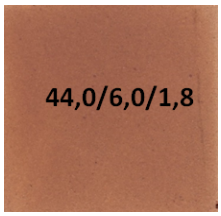
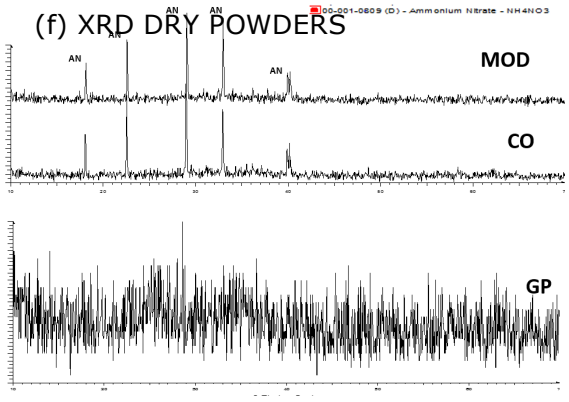
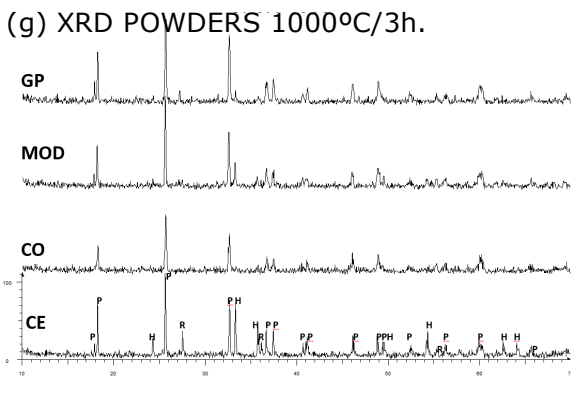
CE	CO	MOD	GP
(a) DISPERSIONS			
(b) DIRECT DEPOSITION 48 threads (L*a*b* values)	 60,9/7,1/12,0	 48,1/8,7/11,5	 65,5/16,7/24,5
(c) POWDER DEPOSITION 500ºC/1h ON A WHITE BODY DOUBLE FIRING CYCLE 1050ºC (L*a*b* values)	 38,8/5,9/6,3	 50,1/6,2/8,5	 58,4/8,1/12,1
 (d) SEM POWDER 1000ºC/3h			
 (e) ENAMEL 5% DOUBLE FIRING 43,6/18,8/12,4 (L*a*b* values)	 41,2/16,1/15,6	 33,0/5,2/0,8	 44,0/6,0/1,8
 (f) XRD DRY POWDERS MOD CO GP		 (g) XRD POWDERS 1000ºC/3h. GP MOD CO CE	

Figure 1. *Results obtained described by rows in phrases a, b, c, d, e, f and g.*

4. ACKNOWLEDGEMENTS

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5. REFERENCES

[1] Zhidong Pan, YanminWang, HuiningHuang, ZhiyuanLing, Yonggang Dai, ShanjunKe, Recent development on preparation of ceramic inks in ink-jet printing, Ceramics International 41(2015)12515–12528.

[2] R. Galindo, C. Gargori, S. Cerro, A. García, G. Monrós, New red pigment based on Cr doped pseudobrookite., VI Reencuentro Franco-Español de Química y Física del estado Sólido, Book of Abstracts, Tarragona 2010.