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A TRAVEL INTO CERAMIC PIGMENTS MICRONIZING

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An increasing number of ceramic tiles is decorated by inkjet printing, utilizing in most cases pigmented inks. These inks are manufactured by micronizing conventional ceramic pigments, starting from 3-10 μm in size and going down to a median diameter usually ranging from 0.2 to 0.6 μm. The theoretical framework predicts significant changes in both optical and fluid mechanical properties during such a size reduction of pigment particles. However, not all the expected advantages occur and still unanswered questions concern colour strength and particle size distribution of micronized pigments as well as efficiency and actual yield of the milling process. The present contribution is thought as a travel along progressive steps of pigment micronizing, that is aimed at disclosing what happens in terms of particles size, shape and composition in the submicrometric field. For this purpose, industrial pigments were selected to represent crystal structures with different density, hardness, cleavage and fracture toughness: Cr-Sb-doped rutile (orange-yellow), Co-Cr-Fe-Mn-Ni spinel (black), and V-doped zircon (turquoise). Pigments were micronized in a pilot plant (Netzsch Labstar LS1) keeping carrier, solid load, type and concentration of dispersant, rotation speed, amount and size of grinding media, and milling time under control. For each pigment, sampling was carried out at increasing milling time in order to get "instantaneous pictures" at progressive stages of micronizing. Pigments were characterized for particle size distribution (laser diffraction and dynamic light scattering), particles morphology (SEM), specific surface area (BET), degree of solubilization (ICP-OES), phase composition, unit cell parameters and crystallite size (XRD-Rietveld), optical properties (DRS) and colour after application in glazes for porcelain stoneware tiles fired at 1200°C (CIE L*a*b*). Results highlight a different behaviour during micronization: the milling efficiency decreases from zircon to rutile to spinel, in partial agreement with literature data. Crystal structural and optical features are substantially changed once pigment particles turn into submicronic size. A gradually lower crystallite dimension is accompanied by increasing frequency of lattice defects (inferred from variation of unit cell parameters) and sometimes amorphization. The occurrence of amorphous phase can significantly reduce the pigment yield with loss up to 25% wt. These structural changes are associated to decreasing colour strength and increasing brightness through the submicrometric field. This overall trend affects more zircon than rutile and spinel. Microscopic observations reveal unexpected paths during micronization in terms of particles size and shape, by varying process parameters, that are able to influence both pigment milling yield and ink performance. Concluding remarks will address suggestions and recommendations for pigment milling and ink formulation.