INCO Industria Colori has developed a specific range of ceramic pigments for ink production with exceptional technical characteristics. These products are aimed at ceramic glaze and colour producers and ceramic tile manufacturers interested in starting up their own independent production of pigmented inks.

The technical characteristics are a result of complete pigment reformulation, meticulous selection of the raw materials and a thorough review of the production process.

This completely new range of dedicated pigments is already used in the production of INCO INX and can produce excellent quality inks quickly and with considerable savings. For this reason the range has been named QUICK STAIN.

Pigmented ceramic inks are dispersions of a solid inorganic pigment phase in a liquid phase (solvents with varying degrees of polarity, dispersants, rheology modifiers). The production of these inks is based on a process of reducing the pigment particle size (a “top down” process) through high-energy grinding in special mills with grinding chambers filled with microspheres of diameter of 0.3-0.4 mm (Photo 1).

During the process, a homogeneous mixture of solvent and pigment is circulated for several hours through the grinding chamber, while a special rotor imparts a high peripheral velocity, and hence high energy, to the spheres. This continues until the inorganic pigment has an average particle size of 250-350 nm and a d99 of 0.8-1.3 µm (Photo 1b).

Unlike the organic pigments used for production of inks for printing on paper and fabrics, inorganic pigments have a high Mohs hardness (>6-7) and a high specific gravity (>4 g/cm³) (Photo 2).

This high degree of hardness means that a lot of energy (2-4 kWh/kg) and long milling times are needed to achieve the requisite ink particle sizes. This results in low levels of plant productivity, high energy costs of production and considerable wear of the grinding spheres and mill chamber components.

Due to the high specific weight of the pigment and the larger particle size with respect to other types of inks, ceramic pigmented inks are more...
susceptible to sedimentation. The properties of the inorganic pigment play a key role in the design of a high-quality ink. In particular, the particle size distribution, morphology and homogeneity of the particles have a significant influence on the end characteristics of the ink in terms of colour gamut, filterability and sedimentation.

PRODUCTION OF CERAMIC PIGMENTS FOR INKS

The inorganic pigments used in ceramics are synthetic products obtained from a high-temperature (1000-1400°C) solid-state reaction of mixtures of transition metal precursors (Fe, Cr, Ni, Co, Mn, Zr, etc.). This high-temperature process produces coloured crystalline structures (spinel, zircon, sphene, etc.) that can be used for colouring ceramic glazes or as a solid component for the production of inks.

The pigments used for ink production must have very specific characteristics:

1) Low percentage of salts: a washing process is therefore required.
2) High colour intensity: a limited quantity of pigment can be introduced as solid phase.
3) Colour gamut: each pigment must produce the maximum specific colour range.
4) A narrow particle size distribution: the narrower the particle size distribution at the entrance to the grinding mill, the narrower the distribution curve in the final ink.

This is preferable as it allows for the production of inks with superior colorimetric properties and stability.

Pigments for ink production currently undergo a dry pre-milling process using Jet Mills. These consist of compressed air counter-jet mills (up to 6 atmospheres) in which the grinding action is achieved by impact between the particles. The narrow and fine distribution of the particles is guaranteed by a classifier that rotates at different speeds according to the desired particle size (Photo 3).

This grinding technology has certain particle size limitations in terms of the coarse fraction, especially as regards the particle fineness specifications required for ink production. Laser diffractometer measurements of the particle size distribution of products obtained from Jet Mills reveal values of $d_{50} = 2-3 \mu m$ and $d_{99} = 5-7 \mu m$, which are certainly very good values.

However, if we analyse the particle size distribution in greater detail, we find that $d_{100}$ can reach values as high as 10-14 $\mu m$, revealing the presence of a grinding tail consisting of coarser particles which are sometimes not detected by the instrument.

The situation proves to be even worse when analysing the pigment under the scanning electron microscope (SEM), which is capable of measuring the real size of the particles as well as analysing their morphology. Photo 4 shows the particle size distribution of a yellow pigment milled in a Jet Mill as measured using a laser diffractometer. Although 100% of the particles would appear to be smaller than 10 micron, this is simply not the case. Analysing the photo taken under the scanning electron microscope, we find particles larger than 10 microns and with dimensions as large as 25-30 micron. Furthermore, the pigment displays a significant degree of nonuniformity in terms of particle size and shape. Although these particles are not...
large in number, their presence can nonetheless create problems during the submicronic milling process. According to milling theories, these larger size crystals would require grinding spheres of diameter 1.0-1.5 mm, which are too large to produce the required ink particle sizes of 0.2-0.3µm. These particle size values can be obtained using spheres of diameter 0.3-0.5 mm. With these sphere dimensions it takes a very long time to reduce the size of the large particles and these long milling times have the negative consequence of creating a fine particle size tail in the final ink. These particles result in a loss of colour in the ink and rheological problems.

Exploiting its more than 25 years of experience in the field of inorganic pigments, INCO has conducted a study aimed at developing a new series of pigments with significantly improved particle size and morphological properties compared to those produced in a conventional Jet Mill.

The Quick Stain pigments have a particle size distribution with an average value of 0.7-0.9 µm and a real d_{100} of below 3 micron measured from images made with a scanning electron microscope, as well as particles with more uniform shape and size. Photos 5a and 5c compare pigments obtained with Jet Mill grinding technology (left) with Quick pigments (right). As can clearly be seen in the photos, the Quick series pigment particles are considerably finer and more uniform (Photos 5a, 5b, 5c). These properties of Quick pigments result in an ink with a narrower particle size distribution than a pigment deriving from a Jet Mill process, as can be seen in Chart 6. The image for Quick Yellow IQ 10/Q110 appears to show a fused, spongy, almost sintered mass. In reality, as can be seen in the following image taken at a larger magnification, it is an aggregation of primary particles created by electrostatic attraction between very fine particles, with average particle size values of around 600-700 nm.

THE ADVANTAGES OF QUICK

Due to their finer size and narrower particle size distribution, Quick technology pigments are most suited for the production of high-quality ceramic inks (Chart 6).

They give the following advantages:

1) More intense colour.
   Narrowing the Gaussian grinding curve reduces the quantities of the finest and coarsest particles, automatically increasing colour intensity.

2) Lower sedimentation and creaming.
   A more uniform particle size results in a reduction in sedimentation and creaming. The product remains rheologically stable for a longer period of time.

3) Greater colour stability.
   More uniform particles result in production batches with more consistent colour while maintaining the same fire resist-
4) **Improved filterability.**

Quick pigments have a more regular shape, which is maintained even after Ink Mill grinding. The particles therefore always have the same size at the filter. Other kinds of pigments are liable to create inks with lenticular shaped particles and thereby give rise to problems of filterability on decorating machines.

This brings savings of at least **30%** for ink producers.

5) **Energy saving during grinding.**

Due to the very fine particle size, the narrow Gaussian curve and the controlled shape of the particles, it is possible to reduce the grinding times by **30-40%**.

6) **Reduction in wear of grinding media.**

The expensive grinding media (spheres) are used for less time.

7) **Reduction in mill wear.**

8) **Smaller plant investment.**

All the above-mentioned savings lead to a reduction in investments in milling machinery as a result of the considerable increase in production capacity.

---

**THE QUICK RANGE**

The Quick Stain range consists of 14 high-saturation pigments with a particle size below 3 microns. They cover a wide gamut and enable ink manufacturers to offer a wide range of colours.

In keeping with the INCO tradition (Sarti del colore dal 1988 - Colour tailors since 1988), specific colours can naturally be developed to order using Quick technology.

Inject high quality into your ink system!