Blue copper phthalocyanine (CuPc) ink concentrates – existing processes

Traditionally many procedures are needed to carry out the multitude of different chemical and physical transformations required in the change from non-pigmentary to pigmentary material, with good dispersion properties that consume water and solvent as well as occupying many different pieces of equipment, all of which increase costs and/or require recovery of liquids.

For example, conventional processes for the production of inks or ink concentrates containing copper phthalocyanine pigments (see Figure 10 for molecular structure) have made it necessary to subject the crude copper phthalocyanine to prolonged treatment in a ball mill or kneader in the presence of an inorganic salt followed by further conditioning, such as solvent treatment, and isolation to convert the crude metal phthalocyanine into a pigmentary form, which is then dispersed, usually with further grinding, in an ink vehicle.

An alternative procedure for, primarily, the North American and Far Eastern markets involves limited integration of the processes by flushing the pigment presscake into the ink vehicle to form a concentrate. However this technique still requires various stages of process equipment, and chemicals to be used and recovered.

Physical changes involved

Figure 13 depicts the change in crystal size when converting from crude to ball milled intermediate (containing aggregated crystals) and then to final pigmentary form.

Blue CuPc concentrate – intensified process

General

The process described in this paper carries out dispersion, crystal size and shape change, controlled change of polymorphic structure, surface chemistry and final dispersion manipulations on a solid material all within the confines of the barrel of an automated co-rotating twin screw extruder.

Ink concentrates containing a pigmentary copper phthalocyanine (CuPc) can be obtained by firstly milling crude CuPc to reduce its particle size and then extruding the modified crude CuPc together with a mixture of heatset ink vehicle and compatible solvent.

This concept is described in detail by Langley et al in a European Patent Application¹ and US Patent². In addition,
there is also a patent application pending on the intensification of pigment/paint processing.

An outline of the intensified ink concentrate process together with the traditional processes is shown in Table 1. The degree of process intensification can be seen clearly.

**Principles of co-rotating twin screw extrusion**

A general arrangement of a Co-rotating Twin Screw Extruder is shown in Figure 1.

**Figure 1: Twin screw extruder, General arrangement**

A variable speed electric motor is coupled to a gearbox which reduces the speed and splits the output to two shafts. Power is transferred to the twin shafts through thrust bearings to protect the gearbox from the large axial forces experienced during pressure generation.

The extruder barrel consists of segmented sections up to a total length of 40:1 L/D allowing flexibility. Temperature control is by electric resistance heaters and/or oil/water circulation. Trials were carried out on 30mm and 65mm machines with lengths of 25D and 40D. The co-rotating agitator screws intermesh closely and are self-wiping. The screw configuration is built up using slip-on agitator components to allow flexibility in designing a screw profile for a particular application.

Feed screw elements convey material downstream. (Figure 2) The positive conveying action pushes material through regions of low forwarding capacity eg mixing sections consisting of kneading paddles (Figure 3).

**Figure 2: Operating principles, conveying. Conveying feedscrew.**

Paddles are the primary working component of an agitator assembly which may contain several of these kneading paddle sections. Paddles can be set at varying offset angles down the shaft to vary the mixing intensity. Paddle pairs (ie paddles on opposite shafts) must always be orientated at 90° to each other (Figure 4 shows this clearly).

**Figure 4: Operating principles, mixing. Interaction of paddle pairs**

The smearing action of the bilobe mixing paddles relative to each other and to the barrel wall causes high shear regions to occur in the mix. The closely intermeshing arrangement of the agitators causes the compression/expansion effects shown in Figure 5, which clearly highlights the figure of eight transportation (3 dimensional). This unique action leads to a very homogeneous product.

Agitator configurations are characterised by their mixing intensity. This is dependent on:

1) Number of paddles
2) Relative offset angle of paddles in mixing/kneading zone
3) Degree of flow restriction to hold mix in kneading zone eg use of orifice plug or reverse feedscrew section.
4) Number of mixing zones

**Figure 3: Operating principles mixing, Paddle orientation.**