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SPECIAL GRINDING STUDIES

The following summary of the HF grinding studies is classified according to proposed end use of the product.

Dry Water Dispersible Lakes

The semi-works grinds made in February have been finished as GP-511-D of approximately standard (30%) CPC content. The strengths are in line with indicated relative particle size (by optical means) with one sample distinctly stronger than standard and the others of about standard strength. Six of the batches were combined in a mix which is very close to GP-511-D. However, some evidence is pointing toward a deficiency of wetting in these colors, and this will need attention. The material is superficially wet, but becomes quite gummy and difficult to disperse completely.

Another semi-works grind was carried to 8 hours; but the grinding rate continues very much inferior to laboratory cycles and it is obvious that a careful study of conditions in this unit is required. Nevertheless, it is well demonstrated that the desired quality as judged by strength can be obtained.

An expert from the Experimental Station spent one day at Newark going over the separation problem and the ultimate conclusion was very optimistic. When grinding is as successful as most of the laboratory grinds, filtration appears quite feasible and can probably be done through conventional filter membranes such as cotton duck. A relatively thick bed of borax seems to offer no bar to filtration under these circumstances. When grinding is less efficient, either because of the physical conditions or by reason of the use of less agent, filtration is always ineffective until substantial dilution is accomplished. This suggests some tendency to flocculation at high concentrations, but becoming dispersed on dilution. In any event centrifugal separation was shown to be effective in these cases after dilution and appears to be the method of most promise for such situations. In view of the optimism on finding means of separation, it is felt that continuation of the study of grinding conditions can proceed with the study of separation being carried on in a limited way at the same time, but to be ultimately proved after grinding conditions are established.

An attempt to use a batch of Newport Crude Green in presscake form resulted in the observation that this batch of crude contained a substantial amount (ca.10%) of pellet-like material not readily broken up on grinding with borax. Since this material is found to be present in varying degree in most batches of crude, there is now a serious question of the feasibility of grinding the crude presscake unless the pellets can be eliminated.

Near the end of the month a substantial increase in the charge in the laboratory unit was made with the hope of more completely covering the discs and possibly getting improved operation. The hope has been fully justified and the charge was increased from 60 gm to 100 gm of crude, with proportional increase in borax, without any significant reduction in rate of grinding as judged by spectrophotometric transmission curves. Lower speeds of agitation give some increase in cycle, but still give good grinding.

One attempt to prepare a water dispersible lake of RT-428-D was made without success. There was little evidence of particle size reduction; the resulting products were much lower in pigment content than desired and were not completely re-dispersible in water. Also they were weaker than desired, even when compared with present standards at equal toner content. No further study in this field is planned at present.

#### Dispersed Pastes

One series was run with 10% Blancol re-using borax 3 times after filtration on a porous steel funnel. Even after substantial dilution, filtration was poor. However, the pigment pastes compare favorably with GT-689-P and the recovery of borax indicates a loss of about 1 lb. per lb. of pigment.

In a subsequent grind the amount of borax was reduced to as low as 2 lbs. per lb. of pigment. The time of grind was increased in proportion to the pigment charge and acceptable strength was obtained. However, in two subsequent grinds at standard charge in which the particle size reduction was followed by spectrophotometric transmission curves, it has been shown that there is re-aggregation after about one hour of grinding. Experiments are under way to show whether there is also an optimum in strength at the same point, and it is obviously in order to study means of preventing this re-aggregation.

#### Linoleum Lakes

The preparation of acceptable BL-288-D has again been accomplished on a laboratory scale from aqueous ground pastes. However, there seems to be a limit in the amount of Blancol which can be tolerated, since products containing 10% Blancol do not take up the Resinous Oil from the emulsions and do not exhibit the strength advantage anticipated from the observed strengths of the pastes based on brushouts.

Attempts to prepare linoleum lake directly from HF grinds without isolation of the paste have had only moderate success. In the case of low Blancol grinds, the separation from borax without actual extraction is difficult, although a reasonably satisfactory lake was obtained. One trial of grinding with whiting instead of borax followed by simple drying of the slurry after adding the resinous oil emulsion gave weak, dull products by Armstrong rubout. Linoleum tests are not yet complete on any of these lakes.

#### Ball Mill Grinding

The question has always been before us as to whether the HF process for aqueous grinds is peculiar to the mill used or whether it can be modified to work in a ball mill. It has been demonstrated that substantial amounts of borax are ground by the balls and can no longer be separated from the pigment by filtration or decantation. With small amounts of borax which could probably be tolerated in WD lakes, some particle size reduction appears to be accomplished, as judged by spectrophotometric transmission curves. However, the rate of grind is very slow and, in 1/2 pt. jars, is less effective in five days than in 15 minutes in the HF mill. At the moment there seems little hope of accomplishing the desired grinding in water in ball mills, but the goal probably justifies some further studies.

#### Analysis by Optical Means

Since chemical analysis of WD lakes for CPC content is cumbersome and time consuming, the theoretical possibility of obtaining the same information from spectrophotometric transmission curves has led to considerable effort in that direction. The first attempt on CPC Green using samples analyzed by chemical means did not appear hopeful. However, analysis of the actual solutions used for the transmission curves suggests that these solutions were not made up with proper care, and that, when a curve is drawn based on this actual analysis, an orderly pattern appears which justifies the theoretical hopes. Some subsequent tests based on this curve fall in line very well with CPC content estimated by other means. This method of analysis clearly deserves further study.

  
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