



E. I. DU PONT DE NEMOURS & COMPANY
INCORPORATED

PIGMENTS DEPARTMENT

Handwritten initials
NEWARK, N. J.

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J. H. C.

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NEWARK

AQUEOUS MILLING OF CPC

(A-II-C-261 - AJS-75%, OJCK-35%)

REPORT FOR THE PERIOD
OCTOBER 18, 1951 to NOVEMBER 15, 1951

During this period, the first batch ground in the 100 gal. Cowles Dissolver was finished to a pulp ready for standardization, and four additional batches were completed to the same stage. Products of excellent quality were obtained in every case, and no unexpected operating problems encountered. The experience will be summarized step-wise as follows:

a) The Cowles unit has continued the excellent performance noted in the last report. Subsequent to the first run all charges have contained 75 lbs. of fresh CPC Green, 7.5 lbs. fresh Blancol, and 750 lbs. total borax. Three charges were made with the 18" Cowles toothed disc with a maximum power consumption of about 22 H.P. The power is quite sensitive to small amounts of water near its maximum. The 18" smooth disc was tried on the fourth run, but did not give sufficient agitation to the charge. It was replaced with the 22" smooth disc, which provided excellent agitation to the whole charge and used the full 30 H.P. available. Control tests (both spectrophotometric curves and actual brushouts) indicate that a grinding time of about 2 hours is adequate, and may be more than is required. In every case the charge pumped easily to the dilution vat, though it is preferred to dilute slightly before pumping.

b) Dilution prior to Classification.

A dilution volume of 350-400 gallons before classification has been successfully used. This point has not been carefully studied and it is believed less volume can be used. It has been demonstrated that it is important to avoid stoppage of the agitator in this dilution tank, since it is very difficult to start again with a charge in the vat.



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c) Classification

After the second charge, dilution has been done in a vat so that there is a continuous gravity flow into the Dorr rake classifier. It has been possible to classify at an overflow rate of 3.5-4.0 gal. per minute with a negligible loss of crystalline borax. The borax sludge leaving the classifier averages close to 74% borax, with only minor variations resulting from changing conditions. The speed of the rake and the speed of feed seem to be the most important variables, but it is believed that no significant further reduction in water content can be obtained without a filtration step. The present operating condition is acceptable as long as dry pigment is charged to the Cowles, though a drier borax sludge would be much easier to reload.

Since a true equilibrium of repetitive use has not been attained because of variables applied, it is difficult to estimate the probable consumption of borax. The actual amounts required to compensate for losses from previous runs and to make up to 750 lbs. have varied from 110 lbs. to about 250 lbs. The best estimate at present is a consumption of about 2 lbs. per lb. of CPC Green charged.

Several mechanical refinements are needed to put this process on a sound operating basis; but it is clearly established that the procedure is practical.

d) Extraction of the Pigment

The procedure to date has used a 2% H_2SO_4 extraction, with the addition of about 7.5% of Diphenylguanidine as the sulfate salt for the purpose of flocculating the Blancol suspension. The filtration and washing cycles have been quite normal and press cake solids have averaged 27-28%. The press cake is quite thixotropic and breaks down on stirring to a fluid paste.

e) Finishing and Standardization

The press cakes finished thus far have been well stirred and treated with NH_4OH to a pH of about 7.5-8.0. At this point the material is quite fluid, but shows evidence of non-uniformity and settles rather badly. One batch was put through a colloid mill in the laboratory with some improvement in appearance. It was then treated with $BaCl_2$ and a quite thick but smooth paste resulted. It appears that too much $BaCl_2$ was used, and it was necessary to thin to a solids content well below 20% to obtain an acceptable fluidity.



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Contrary to experience with the previous process on GT-675-P, this treatment did not result in any loss in strength. It is believed that a suitable treatment with BaCl_2 can be worked out to give an acceptable smooth paste of any desired solids content up to about 25%. This can probably be done in the Abbe-Lenart mixer.

f) Quality

The three batches tested after finishing have been quite close to each other, about 20% stronger than GT-675-P, Lot 74295, blue and intense. They are about 5% stronger than an available sample of Heliogen Green paste (from Staso) and slightly blue and intense when tested by brushout.

g) Program

It is planned to make runs from at least four batches of CPC crude green (two have already been used). This may be followed by one run using 5% Blancol for possible trial in lakes. Attention will then be turned to milling CPC-LB with 5% Blancol for lakes and 10% Blancol for dispersed pastes.

Two laboratory studies on Green during the period deserve mention. It has been proposed to reduce the borax loss by using the slurry from a previous charge as the dilution liquid. This was demonstrated to be workable on a laboratory scale, but seemed to be a somewhat doubtful operation for routine plant use, if carried to the equilibrium point. If, however, it were done on a campaign basis, say three or four times and then start over, it would appear to offer economies in several ways.

The laboratory also tried reductions in the borax CPC ratio to as low as 2:1. The spectrophotometric curves indicated successful grinding, but the finished pastes were markedly inferior to the control. They were, however, still superior to present standards and this deserves more study, since economies will result both from decreased borax use and a larger batch size.

Attempts to prepare WD lakes by adding Blancol and dextrin to dispersed pastes, followed by drying, gave products of fair dispersion but poor strength. It is proposed to give this further study, using plastic milling during the drying cycle.

During this period a dextrin-free CPC Green WD prepared in #14 building from grinds made several months ago was tested and found to be of good strength and dispersibility. This product is being tested by the Sales Service laboratory and there is about 150 lbs. available for sampling purposes.

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