Serial No. KN-44-64

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KN-44-64

NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Final Report

Plant Production and Development of A-1-D Semi Works Trial of Substitution of Disodium Phosphate & Ammonium sulfate for Sec. Ammonium Phosphate.

Period Covered: March 1943 - June 1944.

FILE: 210.21

DATE: 11/7/44

210.21

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Title: Plant Production and Development of A-1-D Semi Works Trial of Substitution of Disodium Phosphate & Ammonium sulfate for Sec. Ammonium Phosphate.

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SUBMITTED BY: E. J. Hess E. J. Hess DATE SUBMITTED: 10/17/44

APPROVED BY: V. Chalupski DATE ISSUED: 11/7/44

V. Chalupski Nov. 14, 1944

INTRODUCTION:

This report is a continuation of KN-43-30 and covers the reduction to plant practice of the K-1781 formula for "Impedex" (now coded A-1-D). New standard A-1-D formulas, K-1904 and K-2067, have been written as a result of this work. Results on a total of thirty-six plant batches are discussed in this report.

A series of batches were also made in the Semi Works using modifications of K-1781 designed to give lower ingredient cost, this material is in process of evaluation by the Paint Laboratory.

SUMMARY & CONCLUSIONS:

Plant manufacture of A-1-D (K-1781 type) may be divided into the following categories:

1. Preliminary work and development of the plant formula in final form. All of this work was done using the following equipment (part of the blue unit):

   Vat 383 - lead lined wood vat, fitted with paddle agitator. This vat was used for slurrying the Di-ammonium phosphate for delivery to the strike vat (Vat 281).
   Vat 351 or 356 - lead lined scale tanks. Used for weighing and diluting caustic soda solution.
   Vat 381 - wood dilution vat. The copperas solution is weighed into this vat prior to strike.
   Vat 281 - wood strike vat; capacity 8550 gallons; double paddle agitation, 17 RPM.
   Press 7 - 48" wash press; wood plates; rubber covered iron frames; frame capacity 2.05 cu.ft.; Labour electric pump.

2. Production in the yellow unit - a result of the taking over of Vat 281 for the production of zinc yellow. The following equipment was used:

   Vat 348 - iron scale tank. Used for weighing and diluting caustic soda solution.
   Vat 351 or 356 - lead lined scale tanks. Used for weighing and diluting (if necessary) the copperas solution prior to strike.
   Vat 247 - wood strike vat; capacity 7650 gallons; turbine agitation, 100 R.P.M.
   Press 3 - 48" wash press; wood plates; rubber covered iron frames; frame capability 2.05 cu.ft.; Labour electric pump.

3. Trial production in the large blue unit strike vat. The equipment used follows:

Vat 383 - lead lined wood vat; paddle agitator. This vat was used for slurrying the Di-ammonium phosphate for delivery to the strike vat (Vat 180).
Vat 348 - iron scale tank. Used for weighing and diluting caustic soda solution.
Vat 381 - wood dilution vat. The copperas solution is weighed into this vat prior to strike.
Vat 180 - wood strike vat; capacity 9920 gallons; double paddle agitation, 8 R.P.M.; no vent on vat.
Press 3 - as shown.

On the basis of previous plant experience with an earlier formula, K-1271, in the red unit (Vat 208 - Press 21) a 15 mol batch size (1-1/2 K-1781) was selected as representing the capacity of Press #7. A slight decrease (4%) in the caustic soda amount was the only deviation from K-1781 used in this trial batch. No difficulty was encountered during the strike, control tests and the appearance of the "Impedex" slurry were normal.

Filtration of this batch was accomplished with considerable difficulty, however, and it was finally necessary to set up a stump press (10 frames) after the major portion of the batch (44 frames in 7 Press) had been processed. When the press was dumped four of the wooden plates were found to be cracked and had to be replaced. The most probable reason for the plate breakage was the excessive pressure built up in forcing the press in order to filter the material in the scheduled pressing volume. It had been observed, however, that during the filtration the press frames filled discontinuously (probably due to the plugging up of the ports by the rapid settling of the plate-like particles) and this also may have caused breakage due to the variations in pressure produced on the thin wood plates.

Because of the difficulty with filtration the batch size on subsequent manufacture was arbitrarily cut by 15% and the pressing volume increased so that the resulting overall increase in pressing volume amounted to about 23-1/2%. The next 19 batches, made during the period of March to October, 1943, in the small blue unit (Vat 281) were based on this procedure.

During the course of this production a number of variations from the basic process were tried most of them designed to facilitate production without affecting quality to any great extent. Among the changes used were:

1. Although it was known that high temperature drying tended to give higher ferric iron and lower ammonia analyses the necessity for using the limited amount of dryer space available at its highest efficiency made it desirable to investigate the possibility of increasing the drying temperature of "Impedex". Upon examining available analytical data (the only basis for comparison of the quality of individual batches now in use) it was found that the three plant batches dried 48 hours at 140°F showed an average figure of 8.31% ammonia and 5.26% ferric iron. These figures were well within the specification limits of 7% ammonia minimum and 10% ferric iron maximum on which shipments are based. It was decided, therefore, to dry a few plant batches at 180°F. Satisfactory drying was accomplished in 24-28 hours at this temperature and, although the ferric iron content (the more significant of the specification figures) has varied over a considerable range (4.65% to 10.8%), twenty of the thirty-seven batches tested analysed between 7.4% and 8.9% ferric iron and only two batches were slightly above the passable limit. The only suggestion that can be made as to the reason for the substandard results is over drying. This theory is based on laboratory experience which shows that by far the major portion of the ferric iron present in the material is formed during the drying cycle (see KN-43-96) and any process that tends to release ammonia from the compound increases the tendency to form ferric iron. In any case, it is believed that 180°F drying will give satisfactory results if carefully applied and the higher drying temperature is used in K-1904, the replacement formula for K-1781.

   The substandard material has been consumed slowly by mixing with satisfactory batches.

2. During the course of the early production in Vat 281 a number of batches were made using a strike temperature of 140°F instead of the standard 150°F. This was done on the request of the operating department in order to save the Vinyon cloths then in use on the large Blue presses. A maximum temperature of 140°F had been set up for pressing in order to conserve these heat sensitive press cloths. No difficulty with quality or processing was encountered although the first high ferric content substandard batch was made at this time. The lower strike temperature was not believed to be responsible for this abnormal result, however.

   Subsequent experience with the 140°F strike in Vat 247 (turbine agitation as compared with the paddle type used in Vat 281) indicated that some change in the material (probably smaller particle size) resulted in slow pressing. Excessive pressure was built up in the press thus causing considerable plate breakage. An attempt to straighten out the situation by cutting the batch 10% (11.5 mol batch) was not successful. Succeeding batches in Vat 247 were therefore made at 150°F on both the 11.5 mol and 12.75 mol batch sizes. Pressing was normal with

either batch size. Although no definite reason can be given for the difference in the effect of the low temperature strike variable with the two strike vats if may be concluded that strikes may be made at 140°F in Vat 261 without disadvantage in order to secure maximum filter cloth life but the strike temperatures when using Vat 247 must be maintained within the specified range of 148-152°F.

3. The K-1761 formula called for dilution of the caustic soda solution with approximately an equal volume of water and then heating to the strike temperature (140° or 150°F) before adding to the di-ammonium phosphate. A number of batches made without diluting or heating the caustic soda were satisfactory in quality and it was decided to use this simplification regularly. It was found necessary, however, in subsequent production to shift over from the lead lined scale tanks (Vat 351 or 356) to an iron scale tank (Vat 348) in weighing the caustic soda because of the slight corrosive effect of the strong alkali on the lead lining and because of the difficulty of scheduling these chromatesscale tanks for "Impedex" production. When Vat 348 was used in conjunction with strike Vat 247 it was found that conditions were such that the alkali must be diluted to about 350 gallons in order to obtain enough head for rapid delivery to the strike vat. No heating other than the slight temperature increase resulting from dilution of the alkali was used.

4. Both filtered and unfiltered copperas solutions have been used in making "Impedex" without any definite advantage in favor of the filtered type. However, there is probably some danger of going over the ferric iron specification limit with the unfiltered material and the use of the clarified copperas solution is therefore recommended.

5. Elimination of the heating of the copperas solution to the strike temperature before addition had no effect on quality. The difficulty of controlling the temperature in the strike vat was definitely increased, however, because, under present conditions, none of the "Impedex" strike vats are equipped with a thermometer. The considerable amount of ammonia evolved during the striking procedure makes it desirable to keep the number of samples dipped out for a temperature check to a minimum. The small amount of time saved in eliminating the heating of the copperas solution does not make up for the added disadvantage, therefore the use of cold copperas solution for the strike is not recommended.

6. Because of the soft, friable nature of the dried press cake the possibility of eliminating grinding was suggested. The question of bag-packing "Impedex" instead of using ply wood drums had previously been considered. A number of selected customers were supplied with the unground material in

50 pound bags and asked for comments. As the replies were favorable subsequent production was packed in bags and shipped in lump form. During the course of this work it was found that the lump gave a less bulky package than the ground material resulting in an approximate 20% savings in package cost. An attempt to use a ribbon mixer to mix in substandard material and as a temporary bag-filling machine was unsuccessful. The mixing action seemed to increase the bulk of the material thus increasing package cost. Mixing is now accomplished by sifting in substandard material by hand during the bag-packing operation.

7. A single batch made in the large blue unit strike vat, Vat 180, had a very low ferric iron content (5.91%) even though dried at 180°F. It is postulated that either or both of the following variations are responsible for the improved analytical result:

   a. The very slow paddle agitation (8 RPM) may slow up the release of the NH_3 excess into the atmosphere thus inhibiting the information of ferric salts. Even more likely is the possibility that it promotes the formation of larger aggregates thus decreasing the surface and slowing up oxidation in the dryer.

   b. The absence of a vent to the atmosphere increases the pressure of NH_3 over the slurry thus cutting down the loss of NH_3. This is an unsafe condition because of the considerable volume of ammonia released inside the operating building and production in Vat 180 will have to be stopped until a vent is supplied.

Recently another batch was made using these conditions under plant supervision and a low ferric iron content (6.96%) was again obtained.

The plant is now awaiting definite assurances as to the production rate on "Impedex" in order to justify the construction of a vent on Vat 180. It is suggested that a decision regarding this be made as soon as possible so that the use of Vat 180 in "Impedex" production may act as a deterrent to the manufacture of high ferric iron substandard material.

A few of the process refinements developed during the course of the early work were embodied in a new formula, K-1904 (based on production in Vat 281) which differed from the laboratory procedure, K-1781, as follows:

1. The batch size is increased from 10 mols to 12.75 mols.
2. Small variations in volumes are made based on plant equipment capacities.
3. The caustic soda amount is decreased approximately 4%.

4. The drying schedule is charged from 44-48 hours at 140°-160°F to 24-28 hours at 180°F.

A still more recent formula, K-2067, covers the production in the yellow unit. The principle differences between K-2067 and K-1904 are:

1. The heating of the alkali solution before addint to the strike water is omitted.

2. Grinding is dispensed with and the material is packed out in 50 pound paper bags instead of in plywood drums.

Yields have been satisfactory. Results on 60 plant batches show an average figure of 99.6% of the estimate which is the equivalent of 98.6% of theoretical.

During the course of the development work on "Impedex" some thought had been given to the possibility of lowering ingredient costs. The greatest possibility for saving appeared to involve either a cheaper source of supply of secondary ammonium phosphate or the use of cheaper ingredients as a substitute for this material.

Meanwhile, there had been some discussion with Grasselli as to the possibility of obtaining a cheap ammonium phosphate liquor from them which would give satisfactory results and still show an appreciable savings. Some material was supplied which was found to be satisfactory (as far as could be ascertained with incomplete tests) on a laboratory scale but nothing more was done because of the necessity of setting up semi-plant scale equipment at Grasselli in order to study their process further. This will not be done until some definite assurance can be given by Newark as to the potential amount of ammonium phosphate that will be needed.

In the early work some consideration had been given to the possibility of substituting other sources of ammonia and phosphate for the secondary ammonium phosphate originally selected. It was found at that time that phosphoric acid + ammonia, disodium phosphate and ammonia, or disodium phosphate and ammonium sulfate could be used.

On this basis two Semi Works batches were made using a mixture of disodium phosphate and ammonium sulphate to supply material for evaluation. A batch (Lot 6455) made with an equivalent amount of these two ingredients to replace the diammonium phosphate and dried at 140°F was well within the specification limits. A saving in ingredient cost of approximately 1/4 cent per pound of product is realized by this change. Part of this batch was treated with additional ammonium phosphate before filtration. No advantage was obtained.

In the second batch (Lot 6456) a decreased amount of ammonium sulfate was used corresponding to a lowering of the ammonia excess from 105% to 75%. An overall saving in ingredient cost of about 1/2 cent per pound of product results from this variation. This batch gave satisfactory analytical figures. Part of this material dried at 180°F, however, analyzed slightly above the specification limit in ferric iron content.

The 140°F dried material, lots 6455 and 6456, have been placed on exposure. The appearance of the panels is still satisfactory after about 8 months test but final results are not available as yet. When and if test data warrants it a revision of the "Impedex" formula to take advantage of this saving should be considered.

EXPERIMENTAL DETAILS

DSN-43-15