KN-64-24 PEARL "AFFLAIR" - SF-97 - NEWPORT PROTOTYPE REACTOR PROGRAM By: J. P. Schaeffer 7/1/64-9/30/64

N39881

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Serial No. PT-KN-64-24

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NEWPORT PLANT

PIGMENTS COLORS PROGRESS REPORT

SUBJECT: PEARL "AFFLAIR" - SF-97 - NEWPORT PROTOTYPE REACTOR PROGRAM

PERIOD COVERED: JULY 1 - SEPTEMBER 30, 1964

DATE: APRIL 29, 1965

SERIAL NO. P.T.KN-64-24

DUP050053365

P.T. KN-64-24

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SUBJECT: PEARL "AFFLAIR" - SF-97 - NEWPORT PROTOTYPE REACTOR PROGRAM

PERIOD COVERED: JULY 1 - SEPTEMBER 30, 1964

CHARGE: 5615-961-16

SUBMITTED BY: J. P. SCHAEFFER DATE SUBMITTED: 9/30/64

APPROVED BY: H. H. GYORGY DATE ISSUED: 4/29/65

ABSTRACT

A prototype of the high agitation, coil heated and baffled Newport reactor was used to establish the effect of process variables of the Pearl "Afflair" hydrolysis in this new type reactor.

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INTRODUCTION

A 20 liter glass prototype reactor with baffles sized 1:12 compared with the hydro tank of the plant, was set up in the laboratory. The plant agitator of rubber covered steel was scaled down 1:12 and was made out of titanium metal. The revolutions per minute of the laboratory agitator which should give the equivalent power were evaluated by using the equation

$$\left[\frac{\text{Speed pl.}}{\text{Speed lab}}\right] = \left[\frac{\text{dia. lab}}{\text{dia. pl.}}\right] \quad 0.5\% \text{ (for Reynolds No. from 2000-200,000)}$$

The reproducibility of the laboratory process for Pearl "Afflair" was found to be good on four batches. The initial trials also matched essentially the first plant batch.

OBJECTIVE

To examine the SF-97 process in a prototype of the new highly agitated and baffled reactor, to establish the effect of several process variables. This is an extension of the work covered by G. Grimm's report (Serial No. PT-KN-64-2 - 3/3/64).

SUMMARY AND CONCLUSIONS

The factors having major effects on the quality of Pearl "Afflair" are:

1. TiO_2/mica ratio
2. Strike time
3. Strike temperature
4. Vigorous boiling and loss of water during boiling period
5. Agitation
6. Heating

The factors having minor effects on the quality of Pearl "Afflair" are:

1. Removal of "black particles"
2. H_2SO_4/TiO_2 ratio
3. Sb_2O_3/TiO_2 ratio
4. Filtration temperature
5. Incomplete and thorough washing of presscake
6. Extended hold time before filtration

The factor affecting the yield is:

H_2SO_4/TiO_2 ratio

Further studies should be made on the effect of different concentrations.

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LABORATORY STANDARD PROCESS

Mica NW-10316 Lot #10 7.06 p
Plus H_2O 12.70 p
Increase temperature to 60°C. - 200 rpm - hot plate
Add TiO_2 conc. 60°C. 13.73 p (=1.95 p TiO_2)
Conc. addition time - 30 min. - temp. 58° - 62°
Add 60° Be H_2SO_4 in 2 min. - temp. 62 - 67°
Increase heat to boiling point: 2 hours
Boil for 3 hours
Loss of water added every 30 minutes
Add 17.8 g Sb_2O_3 (2% Sb_2O_3/TiO_2 ratio)
Stir for 5 minutes
Add water to cool down the batch to 80°C.
Filter immediately afterwards to about 1500 ohms
Dry overnight and calcine at 950°C.

Product was compared to a laboratory standard prepared from Runs 12, 16b and 22b which read vs. plant in-process standard AF-800.0

vs dark, vs yellow, vvs opaque

vvs less yellow, vs less luster

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DISCUSSION

A. Major Factors

1. A 10% increase and a 10% decrease of the TiO_2/mica ratio compared with the standard ratio of 0.276 was tested in order to control the color of Pearl "Afflair". A TiO_2/mica ratio of 0.3 causes a slight lightening of color, which is more yellow over black (oblique) and an increase in opacity with almost no effect on luster. The decrease of the TiO_2/mica ratio to 0.25 had the effect of lightening the color with increased yellowness over white, which is less yellow over black (oblique) and an increase in transparency. A less lustrous product was obtained.

2. Under standard conditions, the strike time will be 30 minutes. The effect of a 5 minute strike was investigated. This caused a lighter color and an increase in opacity and slightly less luster.

3. A departure of 2°C. below and above the standard strike temperature of 58°C. (both mica/water and TiO_2 concentrate being preheated to this temperature) was examined. A strike temperature of 65°C. resulted in a slightly lighter color, which was very yellow over black (oblique) and a slight decrease in transparency and luster. A strike at 51°C. caused a very light color with slightly more yellowness, higher opacity and less luster.

4. The effect of vigorous boiling and not returning the evaporated water during the boiling time was investigated. Under standard conditions, there is a gentle boiling and the evaporated water is added every 30 minutes. The test showed a lighter color and an increase in opacity and slightly less luster.

5. Under standard conditions, the laboratory agitator has 200 rpm (equivalent to 58 rpm of the plant agitator). 100 and 300 rpm (equivalent to 29 and 88 rpm) was examined in the prototype reactor. A decrease to 100 rpm had almost no effect, but an increase to 300 rpm showed a lighter product with slightly more opacity and less luster.

6. The prototype reactor was heated by a large hot plate (equivalent to the silver coils in the plant because of the high agitation). A steam sparger was used in the laboratory reactor in order to see whether the silver coils in the plant can be replaced by a steam sparger. The trial showed a slightly lighter color and slightly less luster.

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B. Minor Factors

1. The removal of the black particles of unknown composition seen on the mica slurry surface caused only very slight differences except for a slight change to less yellow over black (oblique).

2. Variations in H_2SO_4/TiO_2 ratio to 0.5 and 2.0% (standard is 1.25%) had only little effect on the final product. The yield for the 0.5 ratio was about 4% lower than usual.

3. The filtration temperature (90° and 50°C.) had only very slight effect on the quality.

4. Incomplete (800 ohms) and thorough (2200 ohms) washing of the presscake changed the product only very slightly except for more luster if washed to 800 ohms.

5. An extended hold before filtering showed only very slight differences, which are within the experimental error.

6. Variation of the Sb_2O_3/TiO_2 ratio to 1% and 3% (standard 2%) had almost no effect on the quality of Pearl "Afflair".

   Durability as measured in 65 hr. Fadeometer exposure showed

   NF-104 DPS 99175 vs break (2% on TiO_2)
   1% Sb_2O_3/TiO_2 no break
   2% Sb_2O_3/TiO_2 trace break
   3% Sb_2O_3/TiO_2 s break

   The indication that increased Sb causes a break should be rechecked.

7. A faster rate of heating to the boiling caused only slightly more transparency.

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		Over White		Over Black			
		(Vertical)	(Vertical)	(Vertical)	(Oblique)	(Oblique)	
Sample	Process	Darkness	Color	Opacity	Color	Luster	Yield
PSA-5	Strike time 5 min.	light	vs yellow	opaque	vs less yel	s less luster	
PSA-20	Strike time 5 min.	s light	equal	s opaque	vs yellow	s less luster	
PSA-14	TiO_2/mica ratio 0.30	s light	equal	opaque	more yel	vs less luster	
PSA-15	TiO_2/mica ratio 0.25	light	yellow	trans	less yel	less luster	
PSA-8a	Strike temp. 65°C. Filtered to 800 ohms	vs light	equal	equal	yellow	vs more luster	
PSA-8b	Strike temp. 65°C. Filtered to 1500 ohms	s light	vs yellow	s opaque	yellow	s less luster	
PSA-8c	Strike temp. 65°C. Filtered to 2200 ohms	s light	vs yellow	vs opaque	yellow	s less luster	
PSA-9	Strike temp. 51°C	v light	s yellow	opaque	vvs less yel	less luster	

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		Over White		Over Black			
		(Vertical)	(Vertical)	(Vertical)	(Oblique)	(Oblique)	
Sample	Process	Darkness	Color	Opacity	Color	Luster	Yield
PSA-7	Vigorous boiling evaporated water not returned	light	equal	opaque	vs less yel	s less luster	4260 g
PSA-27	Vigorous boiling evaporated water not returned	light	vs yellow	opaque	v s yellow	s less luster	4140 g
PSA-12	Agitation 100 rpm	vvs dark	vvs less yel	vvs trans	vs less yel	vs more luster	4100 g (Std.)
PSA-13a	Agitation 300 rpm temperature before filtering: 90°C.	light	vs yellow	s opaque	vs yellow	less luster	-
PSA-13c	Agitation 300 rpm temperature before filtering: 80°C. (Std.)	light	vs yellow	s opaque	equal	less luster	4125 g
PSA-13c	Agitation 300 rpm temperature before filtering: 50°C.	light	vs yellow	vs opaque	vs yellow	less luster	-
PSA-13d	Agitation 300 rpm filtration after 3 days with agitation on	light	vs yellow	s opaque	vs yellow	less luster	-
PSA-18	Heated by a steam sparger	s light	vs yellow	vs opaque	equal	s less luster	4160 g

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		Over White		Over Black			
		(Vertical)	(Vertical)	(Vertical)	(Oblique)	(Oblique)	
Sample	Process	Darkness	Color	Opacity	Color	Luster	Yield
PSA-6	Removal of black particles	vs light	vs yellow	vs opaque	s less yel	vs less luster	-
PSA-10	H_2SO_4/TiO_2 ratio 0.5	vvs light	vs yellow	equal	vs yellow	vvs " "	3940 g
PSA-11	H_2SO_4/TiO_2 ratio 2.0	vvs light	vs yellow	vs trans	vs less yel	vs more luster	4080 g
PSA-16a	Sb_2O_3/TiO_2 ratio 1%	vvs light	vvs yellow	vvs trans	vvs less yel	vvs less "	Note: 65 hrs Fadeometer No break vs. vs break in std.
PSA-16b	Sb_2O_3/TiO_2 ratio 2%	vs light	vs yellow	vs opaque	vs less yel	vs less luster	Note: 65 hr. Fadeometer tr. break
PSA-16c	Sb_2O_3/TiO_2 ratio 3%	vs light	vvs yellow	vvs trans	vvs less yellow	vs less luster	Note: 65 hr. Fadeometer sl. break
PSA-22a	Standard Filterd to 800 ohms	vs light	vs yellow	vs opaque	vs yellow	equal	
PSA-22b$_1$	Sb_2O_3/TiO_2 ratio 1%	vs light	equal	vs opaque	equal	s less luster	

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		Over White		Over Black			
		(Vertical)	(Vertical)	(Vertical)	(Oblique)	(Oblique)	
Sample	Process	Darkness	Color	Opacity	Color	Luster	Yield
PSA-22b$_2$	Sb_2O_3/TiO_2 ratio 2%	vs light	vs yellow	vs opaque	equal	vs less luster	-
PSA-22b$_3$	Sb_2O_3/TiO_2 ratio 3%	vs light	vs yellow	vs opaque	vs yellow	s less luster	-
PSA-22c	Standard Filtered to 2200 ohms	vvs light	vs yellow	vs opaque	equal	s less luster	-
PSA-19	Heating to the boiling point: 1 hour	vs dark	vs less yel	s trans	vs less yel	vs more luster	-

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