Suspension – 3M Fibers Problem

MEMORANDUM

TO: Mr. M. A. Aitken
Mr. G. E. Brant
Mr. L. B. Dampman
Mr. F. D. Dexter
Mr. D. L. Engle
Mr. J. F. Erdmann
Mr. G. K. Graeber
Mr. G. J. Hanks, Jr.
Mr. C. D. Schuman
Mr. M. E. Sutherland ✓

BY: Mr. J. W. Chatfield
Mr. C. O. Steinmetz

DATE: September 12, 1962

SUBJECT: Minnesota Mining and Manufacturing Company Visit of July 31, 1962

The authors made a visit to Minnesota Mining and Manufacturing Company with Mr. C. H. Strong of the Sales Department.

The call was made to discuss the fiber contamination found by this customer in several blends of QYSJ-5 suspension resin. Also we wished to compare film samples made by us to the film specimens made by this customer.

Film specimens were compared and the visual rating made by inspecting either film sample by 3M or UCP personnel were the same. It was agreed that either 3M film or UCP film would permit comparable ratings.

The contamination studies made by 3M consisted of calendering four-mil clear film and visually inspecting for the presence of fibers. Our studies were similar using a milled film sheet 20 mils thick. Previous blends of QYSJ-5 used by 3M without fiber contamination, fiber contaminated blends, and current production samples were compared and ranked.

Personnel of 3M were impressed with the study and the quality control work done by our people. Our studies and presentation were parallel to the investigation by 3M, and all rankings of the degree of contamination were in agreement.

The cause, correction, and elimination of the fiber contamination was discussed with 3M personnel. Sampling of current production by 3M further assured them that the contamination had been eliminated and that the quality had returned to the level of resin prior to the complaint.

During our meeting with 3M personnel they casually mentioned "flow" and "clarity". These are not particular problems now but indications are that these characteristics will be discussed by 3M as their application of QYSJ-5 becomes more critical.

It is the opinion of the authors that the problem of fiber contamination was resolved with the customer.

Attached is a detailed report covering the fiber contamination problem without specifically mentioning 3M but using blends supplied to them as a refer nce for historical investigation of contamination levels. Reference can be made to Mr. C. H. Strong's Report of Call dated June 12, 13, 14, 15, and 16: (2) July 9, 1962; and (3) July 31, 1962. Detailed information given in these reports is correct and materially aid the speedy correction of the problem.

M E M O R A N D U M

BY: Mr. C. O. Steinmetz

DATE: September 12, 1962

SUBJECT: Fiber Contamination of Process-5 Suspension Resins

Introduction

Process-5 suspension resins were found to be contaminated with fibers upon investigation of a customer complaint alleging that such contamination was present in QYSJ-5 resin. Various blends examined were found to differ in the level of contamination. Detection was made by a customer during the manufacturing of a clear film tape, and the fibers were identified as cotton. Several blends were returned by the customer as unacceptable due to the high level of fiber contamination.

Summary

Prior to the customer's detection of the fibers and subsequent complaint, the production unit had made some corrective steps to eliminate a detected source f probable fiber contamination. The steps which were taken lowered the fiber contamination to an acceptable customer level. However, the acceptable level was not as good as the resin used prior to fiber detection by the customer.

The source of fiber contamination was from the cotton filters used to filter vinyl chloride. The cotton filters were removed, and the system was cleaned of fibers. Production continued without filtering vinyl chloride until fiber contamination was nil; then fiber glass filters were selected as replacement filters. No further fiber contamination has been detected since the corrective measures were made.

Customer acceptance by sampling resin blends of current production, has been achieved for their "critical" tape application.

Discussion

Prior to Novmber, 1961 direct charging to the suspension autoclaves from the vinyl chloride unit was standard practice. This charging was accomplished through one set of cotton filters in the line. During November, 1961 the "direct charging" was discontinued to permit a tank repair program to start at the Vinyl Chloride Unit. Vinyl chloride was then received and stored in available tanks at the Suspension Unit. Autoclave charging from the unit tanks through a second set of cotton filters was established as a standard procedure.

The second set of cotton filters deteriorated more rapidly than the first set in the receiving line due to the hydrochloric acid and water present in the stored vinyl chloride. Cotton fiber from the filters entered the production stream and resulted as a "balled up" fiber in a resin particle. Detection of the fibers from quality control checks of the resin or limited colored film tests was not possible.

Adding to the contamination problem, an occasional non-standard practice of receiving vinyl chloride backwards through the cotton filters caused even a greater than normal amount of fibers to enter and pollute the "new" vinyl chloride in the unit storage tanks. Prior to the customer complaint the production personnel had detected the rapid deterioration of the filters and non-standard receiving practice, whereupon more frequent filter changes were made, the tanks were cleaned, and piping changes were made to prevent reverse vinyl chloride flow through the filters. The more frequent filter changes did not eliminate the fiber contamination but did substantially reduce the level of fiber contamination.

The customer complaint prompted the production unit to immediately remove all cotton filters from the vinyl chloride receiving and charging lines. During the period that the vinyl chloride was unfiltered a decrease in contamination level was noted as the system "cleaned itself" of fibers. QYSJ-5 resin was being produced at this time, and an improvement in color was also verified. Subsequently fiber glass

filters were tested and used as a replacement filter. No deterioration or physical changes were found upon examining the fiber glass filters.

To determine the level of fiber contamination a clear 20 mil film was made and then visually inspected using polarized light. Comparison was made with film made from a known fiber-free resin sample and with the most highly contaminated resin reported by the customer.

A solution test was attempted for comparison, but fibers were not detectable as such but were retained as particle contaminates in solution when viewed with polarized light. Fibers were detectable more readily in the clear milled film. The calendering or milling action works the fibers from the resin and stretches the fibers to about 1/4" length, and they are retained lengthwise in the film with the milling direction.

From the historical analysis of the blends used by the customer and production procedures in force at the time of manufacturing, the cause and elimination of fiber contamination can be seen from the attached chronology of events.

Quality control checks are being made for the customer in our labs. Also blend samples are being sent to the customer for their evaluation and blend selection.

Conclusion

Fiber contamination was detected and corrected. The contamination was caused by deterioration of the cotton yarn wound filters by the combination of hydrochloric acid and water present in the vinyl chloride.

REVIEW BY BLENDS

Blends sampled and checked for fiber contamination. These blends were used or sampled by the customer. Ratings indicated are those of the customer which are also in agreement with UCP ratings.

Blend No.	Date of Manufacture	Production Procedure	Contamination Rating
386	12/25/60	Direct Charging	Fiber Free
409	5/9/61	" "	" "
414	5/20/61	" "	" "
428	7/29/61	" "	" "
431	8/2/61	" "	" "
438	9/15/61	" "	" "
442	9/25/61	" "	" "
449	10/31/61	" "	" "
476	2/21/62	Direct Charging	Fiber Free
480	3/6/62	(Direct Charging Discontinued	Suspected of Fibers
482	3/9/62	(VCI Storage in Unit Tanks	Contaminated
483	3/10/62	(	Contaminated
488	3/11/62	(Reverse Charging & VCI	Highly Contaminated
494	4/10/62	(Storage in Unit Tanks	Contaminated
495	4/11/62	(	Contaminated
510	6/23/62	(Reverse Charging Discontinued (and more Frequent Filter Changes	Acceptable but not good
511	6/25/62	(	50 Per Cent Fiber Reduction
516	7/1/62	(	(25 Per Cent Fiber Reduction (over Blend 510 and 511
517	7/2/62	Removed all Cotton Filters	(
518	7/20/62	(VCI Unfiltered - Removed all (Cotton Filters	(Sampled by Customer and (Accepted as good
519	7/21/62	(	(
522	7/30/62	(Fiber Glass Filters Installed	(Sampled by Customer and
523	7/31/62	(	(Accepted as good for critical (application

UNION CARBIDE

INTERNAL CORRESP NDEN E

UNION CARBIDE CHEMICALS COMPANY SOUTH CHARLESTON 3, WEST VIRGINIA

To (Name) Mr. F. D. Dexter
Company
Location NEW YORK OFFICE

Date June 5, 1962
Originating Dept.
Answering letter date

Copy to Mr. A. R. Anderson
Mr. L. B. Dampman
Mr. D. L. Engle
Mr. O. A. Holt
Dr. R. P. Kurkjy
Mr. R. W. Quarles
Mr. D. E. Richardson
Mr. H. H. Savage
Mr. C. O. Steinmetz
Mr. M. E. Sutherland ✓
Dr. D. M. Young

Subject Suspension Polyvinyl Chloride Design Report Comments from Wacker-Chemie (Second Reply)

Dear Mr. Dexter:

Attached is a copy (in German) and my translation of the second reply from Wacker-Chemie on the Design Report information sent them. The German copy is included should you desire to have the translation checked by a translation service. This translation obviously is not presented in the best English, but I have tried to retain the words and phrases in the sense which I feel was intended by the writer, and this does not often provid good English unless a great deal of latitude is taken with the construction, which then causes some distortion in meaning.

The technical content of the letter does not add much more to their first reply, except to point out certain deviations which we knew exist d and felt we could live with. I will review in detail the Wacker autoclave drawings in comparison to our Pfaudler drawings when I return to Texas City next week and comment further on the content of their second reply, if required. (I left all the drawings in Texas City, so Mr. Holt could refer to them if needed).

I feel that Wacker does not fully appreciate our desire to use glassed vessels to avoid cleaning after every run. If we tell them we plan to operate four to six runs without cleaning, they would shake their heads sadly. However, present practice with the C-20 autoclave here in South Charleston indicates that the only parts of the vessel that get seriously coated with "ivory" ar made of stainless steel, and we would be derelict in our responsibilities n t to try in every way to take full advantage of glass, even with the disadvantages cited by Wacker. Most of the points mentioned about the autoclaves fall under Pfaudler's

responsibility to provide vessels which are free of these faults. I believe that our field representative who inspects these vessels at R chester bef re shipment could profit by these comments of Wacker so he could look for some of these items, particularly regarding alignment and imbalance of the agitator mechanisms.

The comments on the side entering agitators in the blowdown tank and the suspending agent solution tank are well taken, but we did not tell Wacker we planned to use existing vessels for these operations. The problem does not appear serious to me since these vessels have been and/or are in similar service without any obvious difficulty. We will be able to observe these points after start-up and take remedial action as required.

The dryer comments were discussed previously and the question of seal leakage has been checked after a similar comment from Mr. A. G. Gibson, based on some low density polyethylene dryers at Institute. Mr. Holt and I have discussed this point with Mr. Gibson with the present seal design in mind and feel that we should obtain satisfactory performance. The instrumentation we showed on the schematic for dryer temperature control apparently was not completely understood by Wacker - we will control by having the outlet air temp rature reset the inlet air temperature controller in a system somewhat like the cascade systems on the autoclaves.

The latter part of this letter answers a separate inquiry on slurry feed arrangement for the Bird centrifuges, which may be of value t all of our operating departments concerned with Bird centrifuges. The drawings I received can be reproduced by photostat and will be furnished upon requ st.

Should you or any of the recipients of this letter have questions r comments, I will do my best to answer them.

Very truly yours,

J. F. Erdmann

JFE/bcm
Attachment

WACKER-CHEMIE GMBH

TRANSLATION OF: Letter to J. F. Erdmann dated May 15, 1962

REFERENCE: "R"/Ka/Ste No. 21031

CONCERNING: E1710: PVC Plant Design, our letter No. 44-326 dated April 26, 1962

Dear Mr. Erdmann:

We have thoroughly reviewed for you the drawings and apparatus lists transmitted to us with regard to the adaptation for our process and have arrived thereby to the following result:

Autoclaves

As you yourself have remarked, the construction and shape of the aut clav s is of important consideration for the successful operation of the plant. We have established this type of equipment therefore to be the answer. Also our special consideration is observed in your letters. Unfortunately, we must interfere in this respect also in the sphere of the work of the Pfaudler Company. We ask, however, that our detailed statements be interpreted not as criticism but entirely to the propositions for a suitable construction according to our opinion. We may th reby accordingly refer, that many of these vessels are in operation for years without noteworthy disturbances.

From your drawings R161-1530-31, especially R161-1532-33 we have noticed that the main measurements are equivalent essentially to those of our 25 cubic meter autoclaves. Yet your Pfaudler vessel is around 580 mm (23 inches) shorter and the reaction volume correspondingly about 3600 liters (950 gallons) smaller. With reference to the purely process disadvantages of these reductions, Dr. Bauer has already pointed (these) out in his above mentioned letter. According to Pfaudler this change is however necessary in order to be able to do without a steady bearing for the stirrer. According to our observations, the acting forces of the stirrer and accordingly of the power drive assume very important values, especially when th stirrer is not balanced unobjectionably (properly) or vibrates. Both are often th case with glassed stirrers on account of the difficulties of fabrication. B cause of the glassing processes, the shaft distorts itself, so that one limits himself t th supplemental working, which is to center both ends. Also difficult is the balance, which first can result with the completely mounted stirrer, whereby on account of the glassing, equalizing weights must be applied only by means of special devices. From this comes an imbalance through the eventual polymerization of product on the piece, which makes removal difficult and often thereby produces glass chipping. Besides, the additional stress of the bearings, sliding seal ring, and transmission attachment comes to the autoclave head. Considered from these viewpoints, the bearing clearance of the angle transmission appears deficient to us in comparison to the free stirrer length and the attachment with only six bolts on a 24" = 600 mm diameter as insufficient.

Of course, we have pointed out these things according to our determination from evaluations, since we have only your assembly drawings, in which are contained no cross section of the transmission and bearing together with the stuffing box.

Likewise the impeller attachment to the stirrer shaft by means of a two-part hub according to drawing R161-1530-33 appears difficult from a glassing technology standpoint. Our Pfaudler stirrers consist of ring shaped hubs, which are secured against turning and on which the impeller flights are welded. According to the drawing the impeller surfaces stand perpendicular to the direction of rotation. In reference to this, a twisting of the impellers in themselves is stipulated since the impeller flights are attached at an angle. Our impeller flights are not twist d in themselves so that these surfaces with increasing distance from the shaft always would be inclined more toward the direction of rotation or the stirrer axis. This supports the action of the baffles by forming the axial component of the stirring movement. As for the rest the measures correspond to our stirrer with the exception of the shaft length which is around 500 mm (20 inches) shorter.

Also the dimensions of the baffles are with the exception of their length essentially equivalent. The latter are about 1350 mm (53 inches) shorter than ours. From the aboved mentioned measurements we can infer that the baffles are too short in order to make possible a second support in the agitator assembly. These we hav provided for throughout our baffles, in order to avoid the strong oscillations f the baffle and with it a tendency for the baffle stuffing boxes to leak.

All the previously named disadvantages can be avoided with a V2A stirrer (Stainless steel), since one allows for the chemical requirements, which is certainly the case with PVC. We have chosen this method in several cases also with our glassed 14 cubic meter autoclaves with success. The stirrer consists of an unobjectionable (suitable) straight shaft which can be well balanced and centered, and unloads therewith (operates from) the bearing, seal, and transmission. This multiple-piec assembly is simple, polymer accumulations on the end of the stirrer are never observed, but can in an emergency be removed easily and without harm to the surface. On account of the unobjectionable operations it would probably be possible also to increase the stirred vessel to 25 cubic meters and thereby to avoid at the outs t the technical process disadvantages mentioned. Of course, it would then be probably suitable to employ in the place of the angle transmission shown, a VS 80 G transmission of the Pfaudler Company.

A drawing No. 1710/1387 of the thermowells described in our last letter we have transmitted to you already at that time. In the tube a double resistanc thermal element for the temperature recorder is installed as well as a mercury contr l thermal element.

We notice from your drawing SK-75627 about the reaction system that you ar using a Cascade temperature control system for the autoclaves. This is to be desired, since these work more accurately and faster than the simple control systems and tend to oscillate less. Also we have in recent times changed over to this to an increased degree and have installed the Cascade regulator system.

Slurry Flash Tank, 184T

Here two stirrers are provided for which are installed down on the sides. We would suggest that both these stirrers be replaced with a stirrer from above, in order to prevent contamination of the suspension and eventual plastification. We work with several sorts of suspension holders (slurry tanks) with rotations f 22 revolutions/minute, with a power requirement of 7 KW, which also would even serve for your holder size of about 38 cubic meters. The same goes for your suspending agent solution stirrer 206T, your drawing SK-75626-B. Also here is the stirring from above to be preferred. Here we employ a cyclic-gear transmission of 56 revolutions per minute with 10.5 KW power rating, which also serves for a 2 cubic meter vess l. Also the suspending agent storage tank 229T (should) contain a stirrer according to the reasons stated already in our last letter. For this we recommend also a drive with 22 revolutions/minute at a power rating of 7 KW.

For the charging of colloid solutions we use a transportable tank with a pump which is filled on a charge weigh scale. In this way the disadvantages which otherwise appear through the standing of the solution in the long headers are avoided and the charging can take place accurately and without adverse mixing. This arrangement appears especially suitable if more protective agents are to be used simultaneously. Our schematic No. 1710/1890, we have attached for your understanding.

Dryer Design

The subject of the performance of the dryer has already been spoken f in our last letter. According to our consideration of it you would obtain from 600 to 650 tons per month (1,345,000 to 1,456,000 lbs./month), whereas in your statement 780 tons per month (1,750,000 lbs./month) are specified. You should obtain a suitable guarantee about this straight from the manufacturer. According to your drawing SK-75680 you intend to regulate the the inlet temperature of the dryer air with your dryer. We have in the past used the same arrangement, but have in recent times gone over with good success in this area to regulate the heat load by means f th outlet air temperature. This method has the advantage that the regulation has fine sensitivity and results almost free of delayed response, and also responds to irregularities of the product flow (centrifuge output, changes in concentration, tc.). A special aim with a Louisville dryer is to be sure the seal of the rotary cylinder holds against the stationary connecting parts. Here especially at the inlet end where the seal diameter is large the danger exists that the product dust (fines) gets into the seal and becomes plasticized, or impurities from the seal in the form of grease or abraded particles get worked back into the product. We know that with the Louisville dryer this problem has been solved, however, it appears fitting always t refer to this and to require a corresponding guarantee of performance.

Just now your letter of May 2, 1962 has reached us in which you request for transmission the information for our feed system to the centrifuge. We gladly agree to this desire and attach our schematic 1710/1381c, from which you will be able to take the necessary piping sections. The inlet vessel is shown on the accompanying drawing 1710/1467. May we note particularly, that all the shut off devices (valves) in the suspension lines are constructed as special flat discs (sliding gate valves), in order to provide a completely clear tube cross section and thereby prevent layering (stoppages). These valves we have provided for ourselves. We assume that you possess similar valves, otherwise we would have transmitted you a drawing.

The functioning f the arrangement is as f llows:

The suspension (slurry) runs into the inlet vessel, in which through the stand-tube a level head is obtained, whereby the suspension not used by the centrifuge flows back again into the holding tank. In this way the suspension, independent of the pressure of the centrifuge, is always in motion and setting will be avoided. The line from the inlet vessel to the centrifuge itself should be as short as possible. Immediately below the inlet vessel is a sliding gate valve, which in normal operation is always open and immediately beyond is a solenoid valve that closes upon the stopping (failure) of the centrifuge. In this way the suspension pump can continue to run upon the stopping of the centrifuge, without the suspension passing through the standing centrifuge into the dryer. Immediately ahead of the centrifuge you will find a sight glass, which has attached to the upper flange an interchangeable nozzle for the purpose of changing the pressure. By means of a ring nozzle built in underneath, the sight glass can be rinsed with water in order to facilitate observation. An additional water rinse is provided to flush the lines f r the purpose of cleaning. Finally there is provided a by-pass line between the feed and return slurry lines, by means of which the inlet vessel can be discharged when the centrifuge stops or at very small pressure.

All the previously described apparatus serves in the end for the purpos of making possible an uninterrupted flow of material and to eliminate the impurities from the product as much as possible. These requirements serve also for all th equipment which in our writing is not especially mentioned, such as filters for raw materials, seals on compressors and pumps, sealing water leads for suspension pumps, filters, respectively the water removal and oil removal from the compressed air, and finally the blanketing (inert) gasses, etc.

Should we by inspection of your information have overlooked one or another point, we ask you to inform us. We stand ready for the answering of still open questions you may have at your disposal.

Best wishes,

WACKER-CHEMIE
GMBH
Burghausen Management

Attachments:

1 Drawing 1710/1890
1 Drawing 1710/1381c
1 Drawing 1710/1467