PILOT PLANTS

SODIUM PALCONATE PRODUCTION

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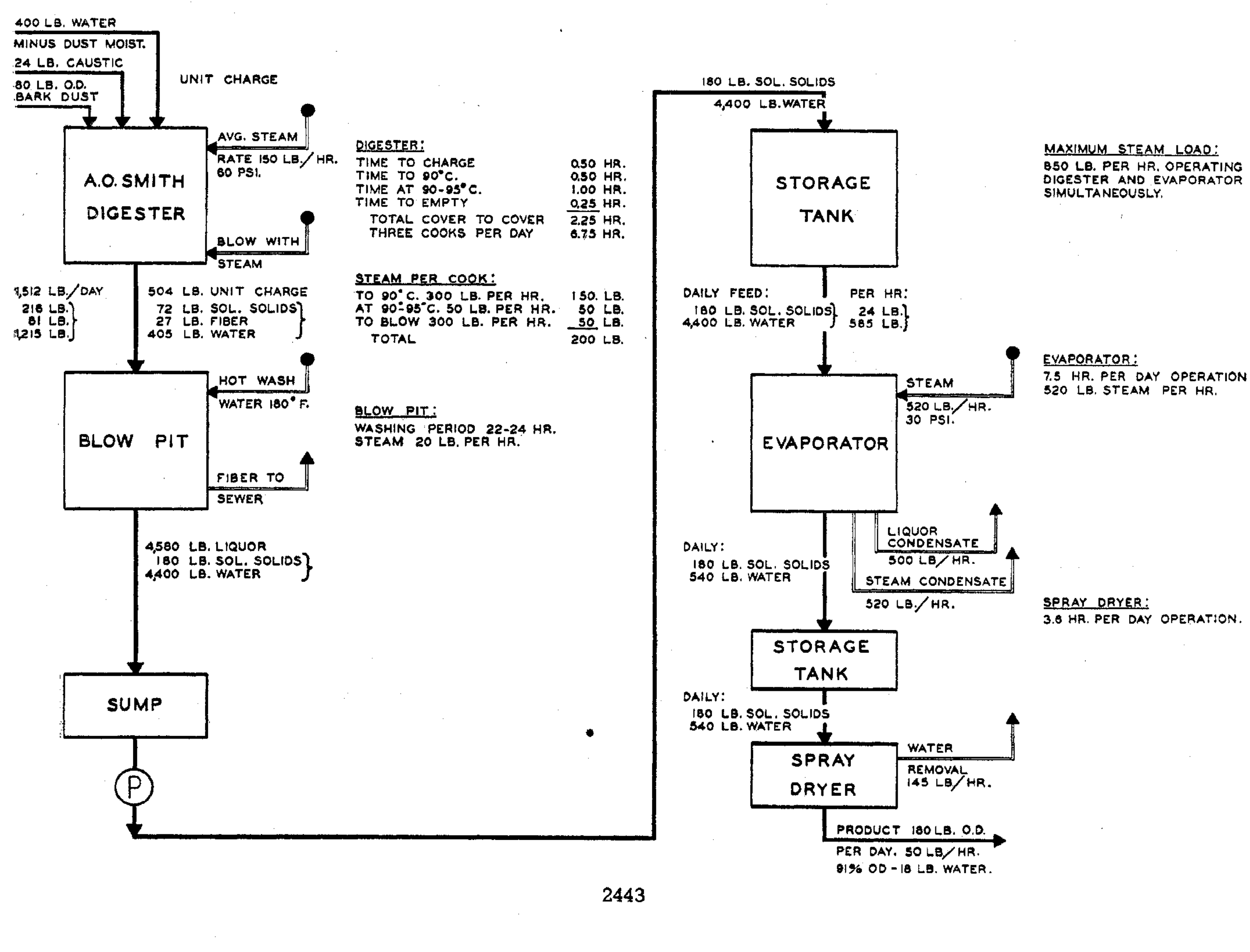
Sodium palconate is an effective agent in controlling viscosity and water loss in drilling; it has other applications as a dispersing agent. Pilot plant production of powdered sodium palconate by alkaline extraction of redwood bark dust followed by concentration and spray-drying of the extract was conducted to extend laboratory methods and prove a tentative process for preparation of the caustic extract, to obtain samples of the material for field testing, and to provide engineering data for use in the design and operation of a commercial plant. Flow sheets and descriptions of the equipment, process, and material are included in the paper.

REDWOOD bark, available in large amounts as a by-product of the redwood lumbering operations in California, is a complex material made up of fibers and the dead tissue of thin-walled sieve cells, parenchyma cells, and cork cells. About 25% of the weight of the dead bark represents redwood bark fiber; the remainder is recovered as an amorphous, finely divided residue better known as redwood bark dust. An examination of the dust shows that the cork cells are at least partially filled with a dark reddish-brown alkali-soluble material; the same material appears within the ray cells and as a thin deposit in the walls of the other cells. This alkali-soluble material has been isolated and shown to consist mainly of a partially methylated phenolic

Figure 1. Flow Sheet for Pilot Plant Production of Sodium Palconate



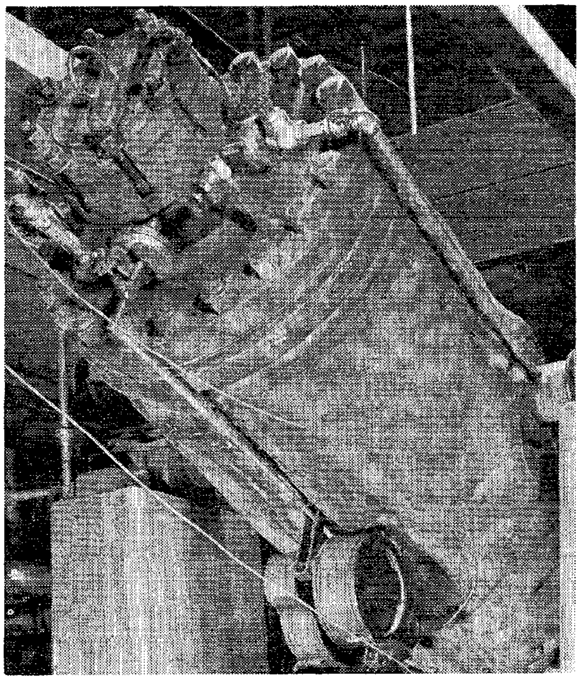


Figure 2. A. O. Smith Experimental Tumbling Digester

acid containing aliphatic hydroxyls, phenolic hydroxyls, and carboxyl groups in the ratio of 2:4:3. This acid has been called palconic acid. An alkaline extract of the bark dust may contain, in addition to the sodium salt of this acid, a significant amount of pectin, which is present in the dust to the extent of 8 to 9%.

Commercial grade sodium palconate, prepared by the alkaline extraction of the bark dust and followed by concentration and spray drying of the filtered extract, has some interesting properties. The viscosity of the aqueous solutions rises rapidly with concentration—the viscosity of a 20% solution at 25° C., determined with a MacMichael viscometer, is 40.1 cp. Solutions of higher concentrations (25 to 30%) are gels. Dilute solutions of sodium palconate remove zinc and aluminum ions from solution. Sodium palconate may be converted to the free acid by passage through a cation exchange column. Sodium palconate has been found to be an effective agent for use in controlling viscosity and water loss in many types of drilling muds. It has other applications in line with its action under certain conditions as a dispersing agent.

PURPOSE OF THE PILOT PLANT

Production of powdered sodium palconate has been made on a pilot plant scale. These operations have been conducted with a threefold purpose: first, to extend the laboratory methods and prove a tentative process flow sheet to be workable for preparation of the caustic extract of redwood bark dust; secondly, to prepare fractional or small tonnage quantities of the product for field testing purposes; and thirdly, to provide engineering data, optimum operating conditions, and information of potential use in the design and operation of a commercial plant.

DESCRIPTION OF PROCESS AND EQUIPMENT

Extensive laboratory investigations proved of great value for pilot plant layout and must be credited for the evolution of a plant virtually identical with expectations. The pilot plant process consists essentially of a caustic cook (redwood bark dust extraction), followed by a washing operation in which the soluble materials are separated from the insolubles, an evaporator concentration of the soluble solids, and a final spray-drying operation to produce the finished product. This sequence is best visualized by reference to the flow sheet, Figure 1.

Most of the units used in the plant as outlined on the flow sheet are general purpose, experimental-type equipment originally intended for and usually employed in the application of pulping and related recovery processes. The design and installation have been in compliance with the rules of simplicity and flexibility. These principles permit the use of selected general-purpose units for more specific applications. The sodium palconate pilot plant is of such selective application, supplemented by certain essential additions.

The digester is a 13-cubic foot experimental tumbling unit manufactured by the A. O. Smith Corporation; it represents a type encountered in various fields of the pulp and paper industry. It consists basically of a steam-jacketed cylinder which can be revolved end over end about trunnion fittings which are piped for the use of direct and for indirect steam for heating and direct steam for blowing (discharge). In its stationary vertical position, the digester is filled through an opening at the top fitted with a removable cover and is emptied through a bottom fitting and valve which are coupled to the blow-tank piping before blowing. Stainless-clad construction permits use of certain acidic pulping agents. This feature, however, is not essential for the present process. Instrumentation is provided for measurement and recording of internal temperature and pressure. The digester revolves slowly about a horizontal axis as pictured in Figure 2; it makes one revolution in approximately 15 minutes. This action permits uniform wetting and pulping at high consistency (ratio of raw material to water). Accessory equipment includes a scale and a crane-mounted, cone-bottom hopper for weighing and charging raw material to the digester. A small steel tank with a gravity feed to the digester is used for caustic cooking liquor preparation. This equipment is situated on a mezzanine floor above the digester and

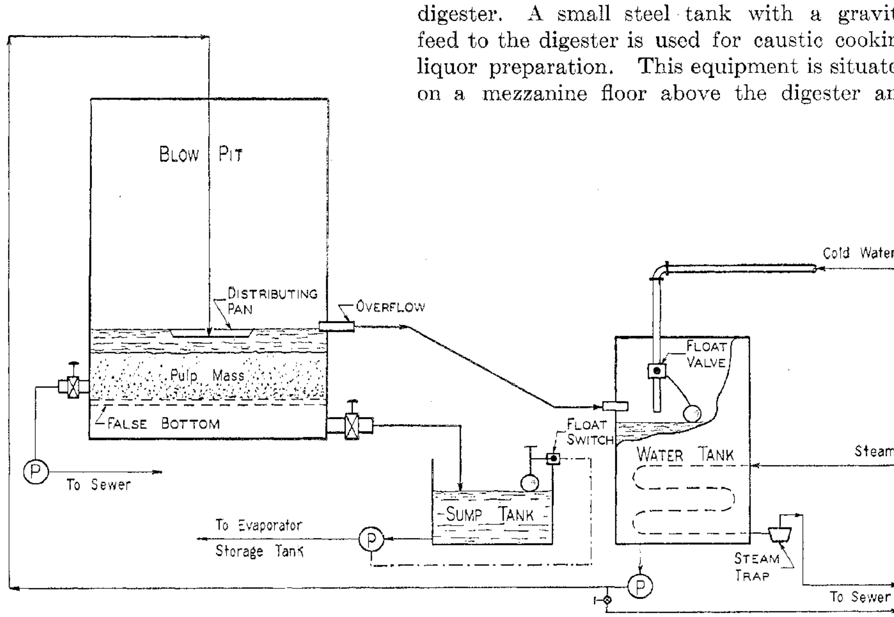


Figure 3. Automatic Washing Equipment

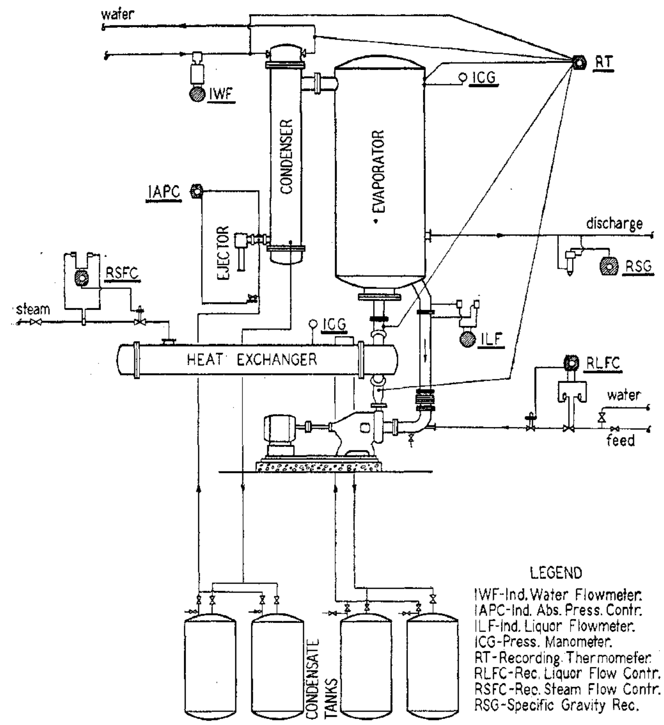


Figure 4. Schematic Layout of Experimental Evaporator

blow pit location. Such an arrangement facilitates preparation of the charges and filling of the digester.

Two concrete based, wooden wall tanks (5 feet in diameter and 7 feet high) make up the blow pits (tanks) into which the cooked pulp is blown through a 2-inch IPS blow line. Entrance is made into each pit on a horizontal tangential run about halfway up the wall of the tank. These pits have stainless steel false bottoms made from plates perforated with $^3/_{32}$-inch drainage holes, and a stainless steel target situated on the wall lining in such a manner as to receive the impact of the digester discharge. The tanks, like the digester, were installed originally and used for ordinary experimental pulping operations. In connection with the digester blow pit arrangement, which is usually considered as a unit, washing equipment has been installed as illustrated in Figure 3. This addition is specific for the present process providing for the automatic maintenance of a small head of hot water over the pulp surface and for the collection of wash liquor effluent draining from the pulp being washed in the pits.

The next unit in sequence, the single-effect Swenson evaporator, is a vertical body, forced circulation, external horizontal heat exchanger type designed specifically for experimental use on acidic and alkaline liquors. The average rated capacity is 500 pounds of water removal per hour but, under certain operating conditions, its capacity is considerably greater than that figure. The evaporator condensate is passed to a vertical tube, surface-type condenser. Accessory equipment includes: pumps for liquor circulation, feed, and discharge; vacuum ejector; calibrated tanks for the collection of evaporator and steam condensate; storage tanks for handling feed and concentrated liquor discharge; and gages and thermometers. All piping, valves, and other equipment elements which are contacted by liquor or evaporator condensate are of stainless steel or stainless-clad construction. Instrumentation is provided for feed liquor, steam, liquor circulation, and condenser water rates in addition to temperature at various points and the recording of the discharge liquor density. An automatic vacuum regulator controls evaporator body pressure. Runs at pressures up to 30 pounds per square inch gage pressure or under vacuum are made with equal ease. The liberal supply of instrumentation makes simulation of multiple-effect operation possible, studying one effect at a time. A schematic layout of this equipment with its instrumentation is shown in Figure 4. Figure 5 shows the evaporator taken from the instrument panel side. Some pertinent data describing this equipment are listed below.

Heat exchanger: 2-pass, 24 tubes 1 inch × 6 feet 16 BWG, triangular spacing, total heating surface 32.8 square feet inside tube area.

Condenser: 2-pass surface type, 24 tubes 0.75 inch × 6 feet 16 BWG, total cooling surface 28.3 square feet outside tube area. Water through tubes.

Circulating pump: centrifugal, forward vane impeller driven by directly connected 25 h.p. 1750 r.p.m. electric motor. Capacity change is obtained by use of different impellers. A 9.5-inch diameter impeller circulates 150 to 200 gallons of water per minute through the heat exchanger when operating at atmospheric pressure.

Vacuum ejector: carbon-lined single-stage ejector, rated capacity 27 pounds of air per hour when operating on 280 pounds per hour of 100 pounds per square inch gage pressure steam.

Feed and discharge pump: cycloidal positive displacement type 0.75 h.p., 1800 r.p.m. electric motor connected through a varidrive speed controller. This pump is used as a feed or discharge pump depending on the type of operation pressure or vacuum. In vacuum operation, feed is supplied by vacuum suction.

The spray dryer, the final principal item of equipment, consists of a 7 × 7 foot vertical cylindrical chamber containing a truncated cone inverted to serve as an atomizer housing. A centrifugal toothed basket-type atomizer is used. This is shaft-mounted in a vertical position on three bearings and is bottom-driven by a 3 h.p. 3600 r.p.m. electric motor, using a small section V-belt. Drying air, mixed with city gas combustion products obtained from a premix type of burner, enters the chamber tangentially near the top and leaves tangentially at the bottom at a point located 180° from the entrance. The air-flow rate is

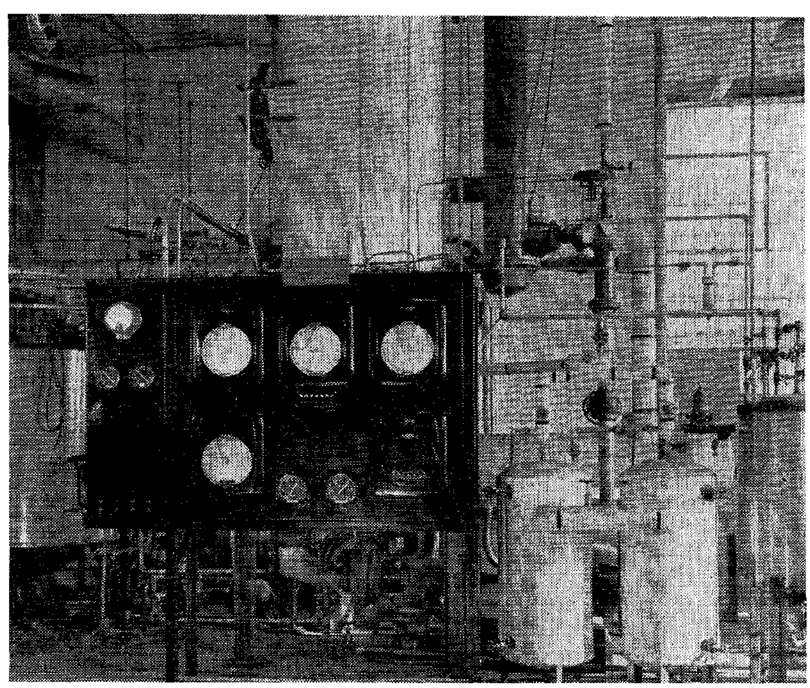


Figure 5. Swenson Experimental Evaporator

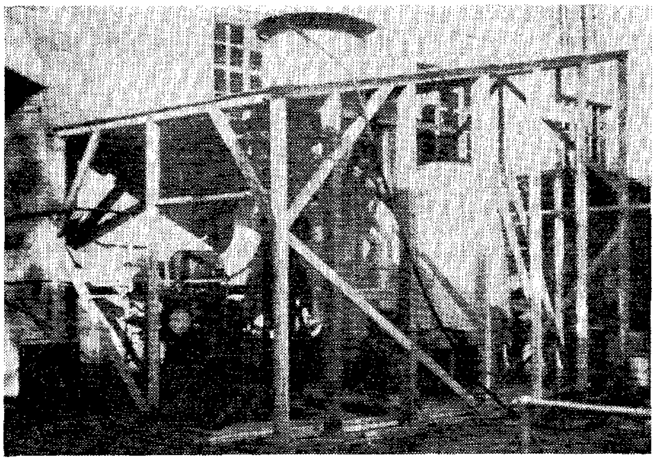


Figure 6. Spray Dryer during Erection

louvre regulated at the burner and controlled secondarily by bleeding atmospheric air into the exhaust duct through a damper located just ahead of the exhauster intake. The atomizer is top fed and the resulting powdered product is exhausted, together with the discharge gases, through a 3 h.p. electric motor-driven, plate-type exhauster. The dry powdered product, in air-stream suspension, is separated in a cyclone separator and collected in the bagging hopper below. Atomizer feed travels by gravity flow through a 2-inch pipe line terminated by a drilled orifice plug. Interchangeable plugs with different size orifices are used to obtain feed streams of various sizes. This piping enters the chamber top through a mounting which is rigid but adjustable, so that the liquor feed stream can be centered over the atomizer plate. An agitated steel feed tank, situated to provide gravity flow, is connected into the line along with a hot water tap. Valving is installed to permit use of either concentrated liquor or water feed and to permit use of water line pressure for flushing the feed line system. The chamber proper is fabricated from angle iron and galvanized sheet iron. To observe the atomizer action, two small windows are located in the top of the drying chamber—one for observation and the other as an entrance for the beam from a spotlight. Pipe tap connections formed by welding on half pipe couplings, are made in both inlet and outlet ducts to permit insertion and installation of instruments. Access to the chamber is through a hatch in the cylinder wall which has the added function of pressure relief in the event of a dust explosion. The only instrumentation is a temperature recorder which furnishes a record of the operation. Dryer equipment without the feed tank and piping are pictured during the process of erection in Figure 6.

As has been previously mentioned, the pilot plant is constituted, in part, of general-purpose experimental equipment which is used frequently for other purposes. Because of this, the plant physical layout is a somewhat scattered one. The equipment is located within an area 65 feet in diameter but in three buildings. This is not an especially desirable arrangement for ease of operation and efficiency of manpower utilization on the particular operation. The use of control instrumentation, however, has been an effective means of combating this condition which arises frequently when operations are intermittent in nature and some of the equipment is employed for other more general purposes. Such an arrangement, therefore, is not particularly objectionable from an over-all institutional viewpoint.

OPERATING PROCEDURES

The pilot plant operation, because of the different equipment capacities, is not a balanced one. Stated in another way, operations are conducted on an 8-hour day basis; during this time the operations (cooking, evaporation, and spray drying) are carried on for periods of approximately 7, 7.5, and 3.5 hours, respectively. The spray dryer is not operated each day as evaporator storage permits accumulation of the concentrated liquor.

In the first step of the process, cooking, the bark dust is weighed into the loading hopper, moved into position over the digester opening, and dumped. The cooking chemical (caustic soda dissolved in the entire water addition) is then run in on top of this dust. The cover is bolted on, the tumbling mechanism is started, and steam is admitted into the digester heating jacket. The charge temperature schedule is 0.5 hour from approximately 25° C. to 90° to 95° C. with a uniform increase, and 1 hour at 90° to 95° C. When necessary, relief is employed to maintain the charge pressure at atmospheric. Prior to expiration of the period at maximum temperature, the tumbling action is stopped to permit connection of the discharge line to the blow-pit. Internal steam is admitted to raise the charge pressure to 50 to 60 pounds per square inch gage; the blow valve then is opened to empty the digester. After blowing, the digester cover is removed and the charging and cooking cycles are repeated. Cover-to-cover time for cooking is approximately 2.25 hours; this gives ample time for a three-cook schedule in an 8-hour day. When the third cook has been blown, the pulp mass is leveled through a hatch in the blow-pit wall and the wash-water distributing pan is positioned as shown in Figure 3 before starting the hot water addition. The use of pipe fittings permits adjustment of overflow height to suit variations in pulp mass depth. This washing setup maintains a constant head of 2 to 4 inches of hot water above the pulp mass throughout the washing cycle. During the cooking and blowing operations, the wash-water distributing pan is raised to the blow-pit top and the overflow pipe is capped for reasons which are obvious. Liquor accumulations in the sump tank are pumped to the evaporator feed storage tank; at the point of discharge the small amount of fibrous materials present are removed by use of the screening device illustrated in Figure 7. In this device, centrifugal force carries the liquor through the screen (40-mesh) and leaves the fibers in a froth with a small amount of liquor to be discharged from the cone bottom into a stationary screen box below. Sloping bottom screen boxes were tried instead of this equipment, but were unsuccessful because rapid plugging of the meshes was encountered. The discharge rate into the screen equipment is approximately 7 gallons per minute. The washing and liquor drainage collection continue unattended until the washing cycle is completed; at that time the insoluble residue remaining in the blow pit is slurried with water and pumped to the sewer. This material represents a potential by-product of the process.

Feed liquor accumulations in the evaporator storage tank vary in concentration, having an average cumulative concentration

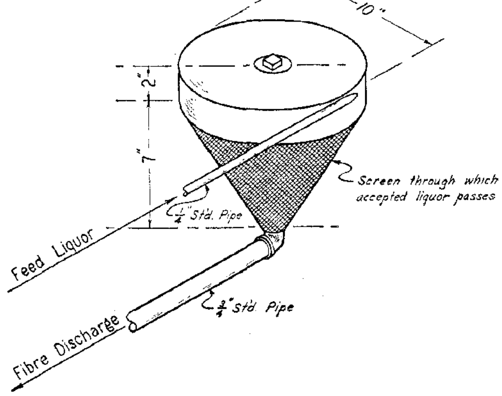


Figure 7. Fiber Screening Device

of 3 to 5%. This material is fed to the evaporator by a flow-regulated pump at the rate necessary to maintain a constant level in the evaporator body, where an operating pressure 2 to 3 pounds per square inch above atmospheric pressure is maintained. This slight positive pressure is utilized to discharge concentrated liquor of 24 to 26% soluble solids into one of the storage tanks where it is agitated to prevent skinning over and flake formation. The evaporator condensate is collected and measured in calibrated tanks; a check on steam consumption may be made by the same method. When steam and evaporator condensate measurements are not made, operator surveillance requirements are slight, except during start-up and shutdown periods.

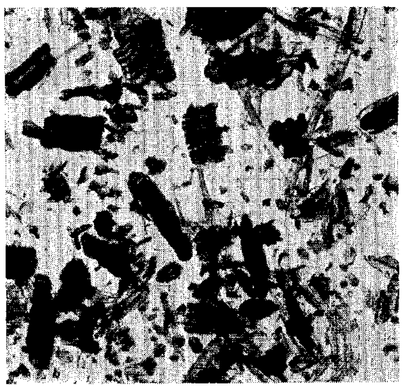


Figure 8. Photomicrograph of Redwood Bark Dust (200×)

An auxiliary pump is used to transport evaporator concentrate to the spray dryer feed tank for final water removal and the preparation of the powdered product. This liquor, often still hot, must be agitated continuously for reasons previously mentioned. Dryer start-up is made on a tap water feed which permits preliminary adjustment to operating conditions. After equilibrium is reached, the feed is switched to concentrated liquor. Slight adjustments are usually necessary after this change is made. In the drying process, the temperature of the outlet air stream serves as the primary control indication. Ordinary glass-stemmed thermometers or some other type of low lag temperature indicator should be used. The inlet air temperature is adjusted by a damper and louvre previously mentioned and by changing the heating gas flow rate. Since the heating unit is somewhat undersize for the drying chamber, the practice has been to operate with gas at maximum flow. Typical conditions for the dryer operation are presented later in this paper. The shutdown cycle involves a reversal of the start-up cycle, with the final operation on water to flush and clean the feed-line piping.

OBSERVATIONS AND TYPICAL OPERATING DATA

Some mention of the physical structure of raw bark dust has already been made. The actual ratio of bits of wood structure, amorphous material, and separated fiber varies somewhat, depending on the point of collection in the lumbering and related by-product operations. This material is pictured in Figure 8 which is a photomicrograph typical of samples taken from raw material during the pilot plant runs. Table I shows wet classification results of a bark dust sample run in a Bauer-McNett fiber length classifier.

Table I. Bauer-McNett Wet Classification of Raw Redwood Bark Dust

Mesh	Retention, %
On 28	23.5
Through 28, on 48	17.8
Through 48, on 100	27.2
Through 100, on 150	7.5
Through 150, and loss	24.0

Bark dust for the pilot plant runs is received in multiwall paper bags; these make it easier to handle the material, which is a potential dust hazard.

The insoluble residue remaining after the extraction operation shows an apparent increase in the coarse fractions and a decrease in the quantity of fines, as compared with the unextracted material. Reduction in the length of the fibrous materials does not occur because cooking conditions are not sufficiently severe to produce physical degradation of the cellulose. Thus, the cooked material (when washed free from the caustic extract) resembles the raw redwood bark dust in physical appearance. However, it has suffered some change in particle size distribution (primarily by a decrease in the quantity of fines) and a loss of the characteristic red color. The fibrous materials have been rendered more flexible through the removal of caustic extractives which, in reality, constitutes a mild pulping operation. Experience with small scale cooks made in 6-liter laboratory autoclaves pointed to the feasibility of using hot wash water to obtain increased flow rates through the pulp mass bed. In this work, a pulp mass of depth comparable with that predicted for pilot plant use was washed in a cylinder fitted near the bottom with a perforated plate identical with those employed in the blow pits. These data were used as a guide for preliminary pilot plant cooking and washing operations. Data taken during the washing cycle of a preliminary operation have been compiled in Table II. In this test, made before installation of the automatic washing equipment, the average temperature of the pulp surface in the blow pit was 50° C. and the average drainage effluent temperature was 40° C. Figure 9 is a plot showing the extracted soluble solids against washing time. Figure 10 shows the accompanying cumulative water and cumulative liquor concentrations, also as a function of time. Spot checks and collection of data during subsequent

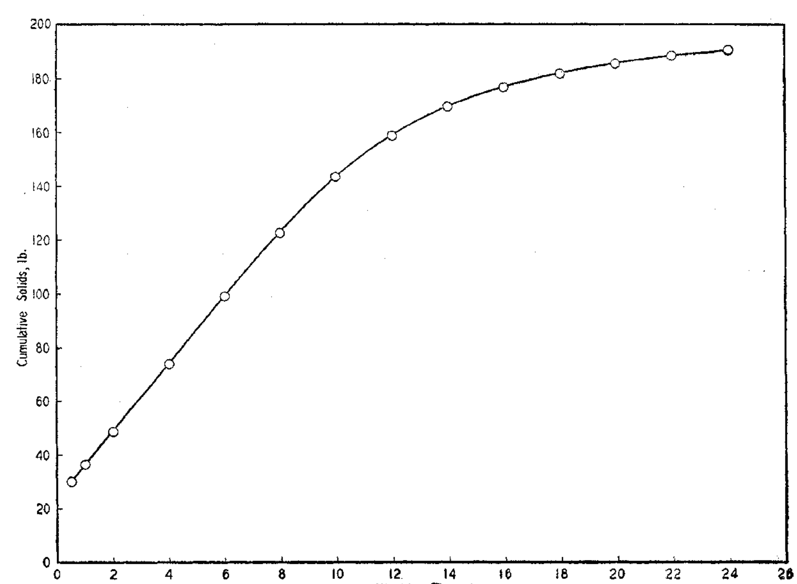


Figure 9. Preliminary Washing Rate Study

Cumulative solids against washing time

Table II. Preliminary Washing Rate Study on Cooked Redwood Bark Dust

Washing Interval, Hr.	Liquor Collected, Lb.		Liquor Concentration (Solids by Weight), %		Solids Collected, Lb.		Water Collected, Lb.	
	Interval	Cumulative	Interval	Cumulative	Interval	Cumulative	Interval	Cumulative
0.0–0.5	319.5	319.5	9.39	9.39	30.0	30.0	289.5	289.5
0.5–1.0	49.0	368.5	13.00	9.88	6.4	36.4	42.6	332.1
1.0–2.0	85.5	454.0	14.03	10.66	12.0	48.4	73.5	405.6
2.0–4.0	172.0	626.0	14.92	11.84	25.7	74.1	146.3	551.9
4.0–6.0	166.0	792.0	15.15	12.53	25.1	99.2	140.9	692.8
6.0–8.0	163.5	955.5	14.52	12.86	23.7	122.9	139.8	832.9
8.0–10.0	172.5	1128.0	12.03	12.74	20.8	143.7	151.7	984.3
10.0–12.0	173.5	1301.5	8.73	12.20	15.1	158.8	158.4	1142.7
12.0–14.0	172.5	1474.0	6.25	11.51	10.8	169.6	161.7	1304.4
14.0–16.0	162.5	1636.5	4.47	10.81	7.3	176.9	155.2	1459.6
16.0–18.0	163.0	1799.5	3.15	10.11	5.1	182.0	157.9	1617.5
18.0–20.0	158.5	1958.0	2.28	9.48	3.6	185.6	154.9	1772.4
20.0–22.0	156.0	2114.0	1.78	8.91	2.8	188.4	153.2	1925.6
22.0–24.0	144.0	2258.0	1.44	8.44	2.1	190.5	141.9	2067.5

Table III. Particle Size Distribution of Through 270-Mesh Fraction of Spray-Dried Sodium Palconate

Photomicrograph Size Count				Distribution Calculation			Percentages of Total Weight[b]	
Diam.[a], μ	Diam.[a], in. × 10^3	Number, N	N, %	μ^3	N (%) × μ^3	Weight, %	Interval, weight % × 59.25 × 0.01[b]	Cumulative
70	2.770	1	0.1	343,000	34,300	1.83	1.08	41.83
60	2.375	4	0.3	216,000	64,760	3.47	2.06	43.89
55	2.180	12	1.0	166,380	166,380	8.91	5.28	49.17
50	1.980	17	1.4	125,000	175,000	9.37	5.55	54.72
45	1.785	30	2.4	91,120	218,500	11.70	6.93	61.65
40	1.585	43	3.5	64,000	224,000	12.00	7.11	68.76
35	1.385	84	6.7	42,880	287,400	15.40	9.12	77.88
30	1.190	105	8.4	27,000	226,700	12.14	7.19	85.07
25	0.990	204	16.4	15,620	256,200	13.72	8.13	93.20
20	0.790	191	15.3	8,000	122,300	6.55	3.88	97.08
15	0.595	279	22.2	3,375	74,900	4.01	2.38	99.46
10	0.395	194	15.6	1,000	15,600	0.84	0.50	99.96
5	0.200	83	6.7	125	840	0.04	0.02	99.98
Total	...	1247	100.0	...	1,867,020	99.98	59.23	...

[a] $\pm 2.5 \mu$.
[b] 100 grams, 59.25 grams through 270 mesh.

pilot plant runs, using the automatic washing equipment, have indicated blow pit and drainage effluent temperatures close to 75° and 60° C., respectively, and a cumulative liquor concentration against time curve as indicated in the dashed line curve of Figure 10. These differences in wash liquor effluent rate and concentration arise as a result of the difference in wash water temperature.

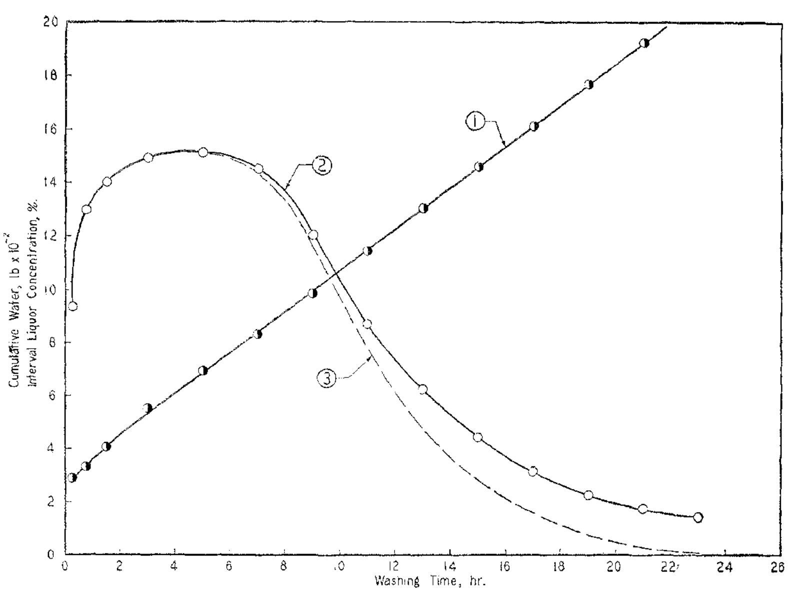


Figure 10. Washing Rate Study

(1) Cumulative water against washing time, preliminary study; (2) interval liquor concentration against washing time, preliminary study; (3) interval liquor concentration against washing time, pilot operations

The increase in solids concentration in the first part of the washing cycle is a point worthy of comment. This is probably the result of a small amount of water on the blow-pit floor when washing is started and the drainage of more concentrated material formed as a result of continued reaction in the mass of the first two cooks in the blow pit during the time in which the third cook is being made. The first effluent, therefore, is representative of the free drainage of the first two cooks diluted, perhaps with a slight amount of water remaining on the blow-pit floor. After the third cook is blown and the head of wash water is added, the subsequent drainage is, as already mentioned, of higher concentration formed by continued reaction in the blow pits. When the maximum effluent concentration is reached, it is followed by a decrease in concentration resulting from the addition of liquor from cook three, followed by penetration of wash water through the pulp mass.

The collected wash liquor effluent is stored in the evaporator feed tank from which it is withdrawn as required. As mentioned, the wash liquor concentration fluctuates considerably; the average cumulative concentration is generally in the range of 3 to 5% soluble solids. Evaporator feed and discharge rates must be adjusted to meet the demands of operating load and fluctuating feed liquor concentration. The steam and evaporator condensates are collected and periodically measured in calibrated tanks before discharging from the system. This and the start-up and shutdown periods require considerable operator attention. The usual practice has been to run the unit at the average operating load of 500 pounds of water removal per hour. On a 26% solution of sodium palconate, this gives a steam economy of 0.962 pound of water evaporated at atmospheric pressure per pound of steam (30 pounds per square inch gage) condensed. Operation on water has given a comparable result of 1.046 pound per pound.

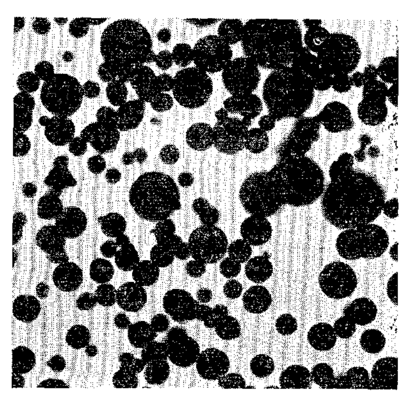


Figure 11. Photomicrograph of Through 270-Mesh Fraction of Spray-Dried Sodium Palconate (200×)

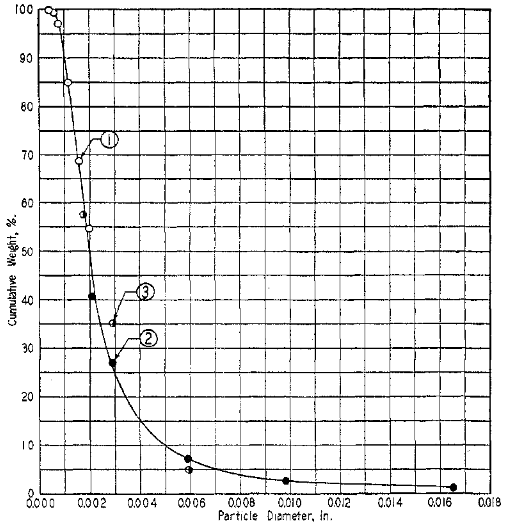


Figure 12. Particle Size Analysis of Spray-Dried Sodium Palconate

Cumulative weight percentage against particle diameter: (1) microscopic determination; (2) sieve analysis; (3) commercial spray-dried sulfite waste liquor

The use of water as a feed in the start-up and shutdown cycles of the spray-dryer operation is excellent insurance against excessive, unnecessary build-up on the dryer walls and against plugging of the feed-line piping. Dryer operating data taken from a typical run are shown below.

Feed liquor (ovendry solids), %	24.6
Inlet air (dry bulb temperature), ° F.	450
Outlet air	
Dry bulb temperature, ° F.	213
Wet bulb temperature, ° F.	111
Air mass velocity, lb./min.	78.3
Pressure drop across dryer, in. of water	1.70
Inlet air duct (vacuum), in. of water	2.07
Outlet air duct (vacuum), in. of water	3.77
Heat economy, %	60
Atomizer speed (rotation in counter direction to air flow), r.p.m.	5400
Atomizer power load, h.p.	
Without feed	1.50
With feed	2.25
Operating capacity, lb. water evaporated/hr.	145
Spray-dried product moisture, %	9

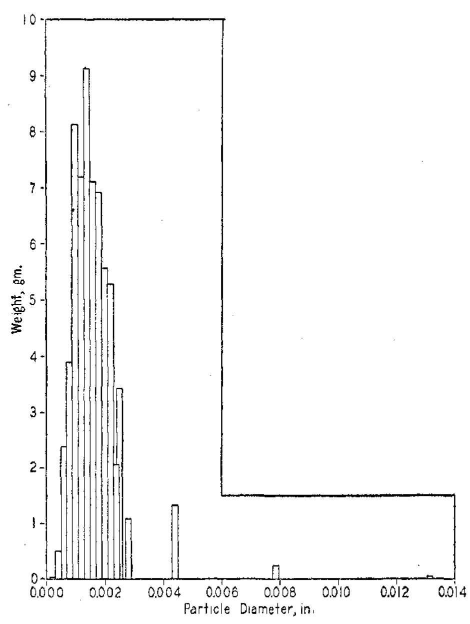


Figure 13. Particle Size Weight Distribution in a 100-Gram Sample of Spray-Dried Sodium Palconate

TABLE IV. SIEVE ANALYSIS OF SPRAY-DRIED POWDERED PRODUCTS

Mesh	Particle Diam., In.	Cumulative Weight, % of Total
PILOT PLANT SPRAY-DRIED SODIUM PALCONATE		
40	0.0165	1.25
60	0.0098	2.65
100	0.0059	7.25
200	0.0029	27.0
270	0.0021	40.75
COMMERCIAL SPRAY-DRIED SULFITE WASTE LIQUOR		
40	0.0165	Trace
100	0.0059	5.0
200	0.0029	35.2
325	0.0017	57.6

Examination of the spray-dried product is of interest from the viewpoint of providing information on the general subject of spray drying and atomization. Microscopic observation has shown that the dried material consists almost entirely of spherical shaped particles. Samples have been subjected to particle size determination by combined techniques of sieve analysis and microscopic observation. Specifically, a sieve analysis was extended into the subsieve range by microscopic observations on the through 270-mesh material. A photomicrograph of this fraction is shown in Figure 11. This picture attests to the predominance of spherical-shaped particles. The presence of connected spheres, a result of contact before dry surfaces are achieved is indicated also.

In Tables III and IV the cumulative percentages of the different particle sizes are shown for a representative quantity of the powdered product starting at the large diameter and moving down the particle size (diameter) scale. A set of unpublished data obtained from the commercial spray drying of sulfite waste liquor has been included for the purpose of comparison (Figure 12).

More than 50% of the pilot plant sodium palconate material (on a weight basis) passes through a 270-mesh screen. The high percentage of small particle sizes noted is more readily seen in Figure 13, in which weight distribution in a 100-gram sample is shown for particle sizes taken at diameter intervals of 5 microns. This distribution represents the data from Table V compiled from the microscopic data in Table III and calculated sieve analysis results. Particles of 35 ± 2.5 microns (0.001285–0.001485 inch) are present in larger quantity (on a weight basis) than for any other 5-micron interval. Particle size figures of further interest are obtained by examination of the dryer cyclone stack loss

TABLE V. WEIGHT PERCENTAGE OF PARTICLE SIZES IN SPRAY-DRIED SODIUM PALCONATE

(Diameter intervals of 5 microns)

μ	Diameter, In. × 10^3	Weight, % of Total
PHOTOMICROGRAPH DETERMINATION		
5	0.200	0.02
10	0.395	0.50
15	0.595	2.38
20	0.790	3.88
25	0.990	8.13
30	1.190	7.19
35	1.385	9.12
40	1.585	7.11
45	1.785	6.93
50	1.980	5.55
55	2.180	5.28
60	2.375	2.06
70	2.770	1.08
CALCULATED FROM SIEVE ANALYSIS		
63	2.500	3.44
110	4.400	1.32
198	7.850	0.24
332	13.150	0.04

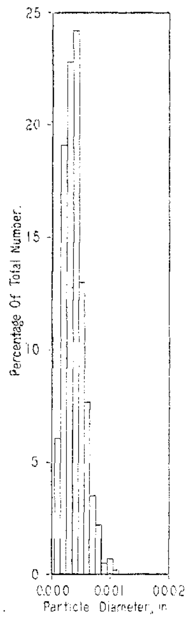


Figure 14. Particle Size Distribution of Spray Dryer Cyclone Stack Loss

material. Distribution of this material, taken by microscopic size count, is shown in Figure 14 and Table VI. A photomicrograph of the material shows some particles which are welded together and indicates that at least a certain portion of the particles are hollow spheres—a point worthy of special note. This was established by microscopic observations of a ground sample of the same material. The subject of hollow sphere particles has been covered in some detail (*1*).

Specific gravity determinations, made on a representative sample of the powdered product, indicate an average particle density of 1.23 grams per ml. This same material has a bulking density, which is a rather vague empirical measurement, of approximately 45 pounds per cubic foot. When water solutions of the powder are prepared, the solution viscosity increases rapidly beyond concentrations of 15% solids. A curve of viscosity at 25° C. against solids concentration is shown in Figure 15. Gels occur in the range of 25 to 30% solids concentration. Weight volume relations show that solutions of the material exhibit an almost linear increase in specific gravity (25°/4°) from 0.997 at 0.09% to 1.176 at 26%.

SOME OPERATING PROBLEMS

A step from laboratory to pilot scale operations is seldom made without encountering certain difficulties and problems which arise, notwithstanding a backlog of laboratory data and experience. Problems encountered in the construction and operation of the described sodium palconate pilot plant will be discussed briefly.

Before many cooks were made, the handling of bark dust in the weighing and charging operation was recognized as hazardous to operators. The high percentage of fine material of low specific gravity made installation of an exhaust system necessary at points where the charges were weighed and dumped into the digester. Operators were forced to wear dust masks even with use of the ventilating equipment.

The slow washing rate encountered in pilot scale work had been predicted and the problem of handling three cooks daily was solved easily by alternate use of two blow pits; these permitted a 22 to 24 hour washing cycle. Some exploratory work in cooking and washing was carried out in the pilot plant—namely, a study was made of the cooking of 50% redwood bark dust and 50% headrig sawdust. This material, because of the coarse sawdust fraction, washed much faster and produced a product somewhat different from the straight bark dust cooks.

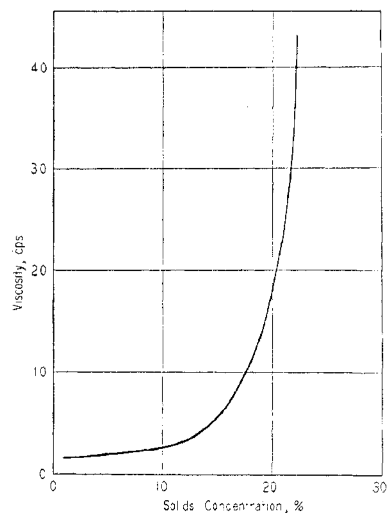


Figure 15. Viscosity of Sodium Palconate Solutions

The first evaporation tests immediately presented a problem of no great practical importance but of interest. In evaporator operation, hot liquor circulated through the heat exchanger is discharged through a nozzle which is centrally located and enters the evaporator body from the bottom. This stream of liquor impinges against a baffle to prevent liquor carry over into the condenser. With this type of equipment, it is not difficult to visualize the formation of foam if the material being handled possesses certain characteristics. Low concentration sodium palconate solutions foam rather badly under the conditions mentioned. When foaming occurs, an entire evaporator charge is likely to be lost in a short time. Because of this, it has been necessary to maintain a minimum liquor solids concentration of 17% in the evaporator. When operating below this figure, the circulating pump is not used and pumping action in the heat exchanger tubes is relied on for circulation. Experience has shown that somewhere in the range of concentration between 13 and 17% solids, the solutions stop foaming. On the viscosity against solids concentration curve, this may be noted as the range in which viscosity starts increasing rapidly.

In the spray-dryer operation, the subject of build-up (the collection of inadequately dried particles on the chamber walls) has been the predominant problem. This, however, is mainly a matter of operator education. When the dryer operation is allowed to become unbalanced, the operator has a real clean-out job on his hands, and soon becomes convinced of the necessity for proper operation. Some mention should be made of critical vibration periods which are encountered when starting and stopping the centrifugal atomizer. Unless the atomizer assembly has been balanced dynamically, these vibration periods probably will be encountered but the effect is largely one of demoralizing uninitiated operators. The important thing is to have a smooth running atomizer at operating speed. This unit, with a 10-inch outside diameter basket has been run successfully at speeds of 4500, 5400, and 8000 r.p.m. The effect of the different speeds on the product particle size distribution for this installation has not been determined.

Table VI. Particle Size Distribution of Cyclone Stack Discharge Loss Encountered in Pilot Plant Spray Drying of Sodium Palconate

	Photomicrograph Size Count		
Diam.[a], μ	Diam., In. × 10³	Number, N	N, %
27.5	1.090	2	0.2
25.0	0.990	9	0.7
22.5	0.890	6	0.5
20.0	0.790	27	2.2
17.5	0.695	44	3.5
15.0	0.595	96	7.7
12.5	0.495	163	13.0
10.0	0.395	303	24.2
7.5	0.300	285	22.8
5.0	0.200	238	19.1
2.5	0.100	76	6.1

[a] ±1.25μ.

ACKNOWLEDGMENT

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LITERATURE CITED

(1) Lamont, Dallas R., U. S. Patent 1,734,260 (Nov. 5, 1929).

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