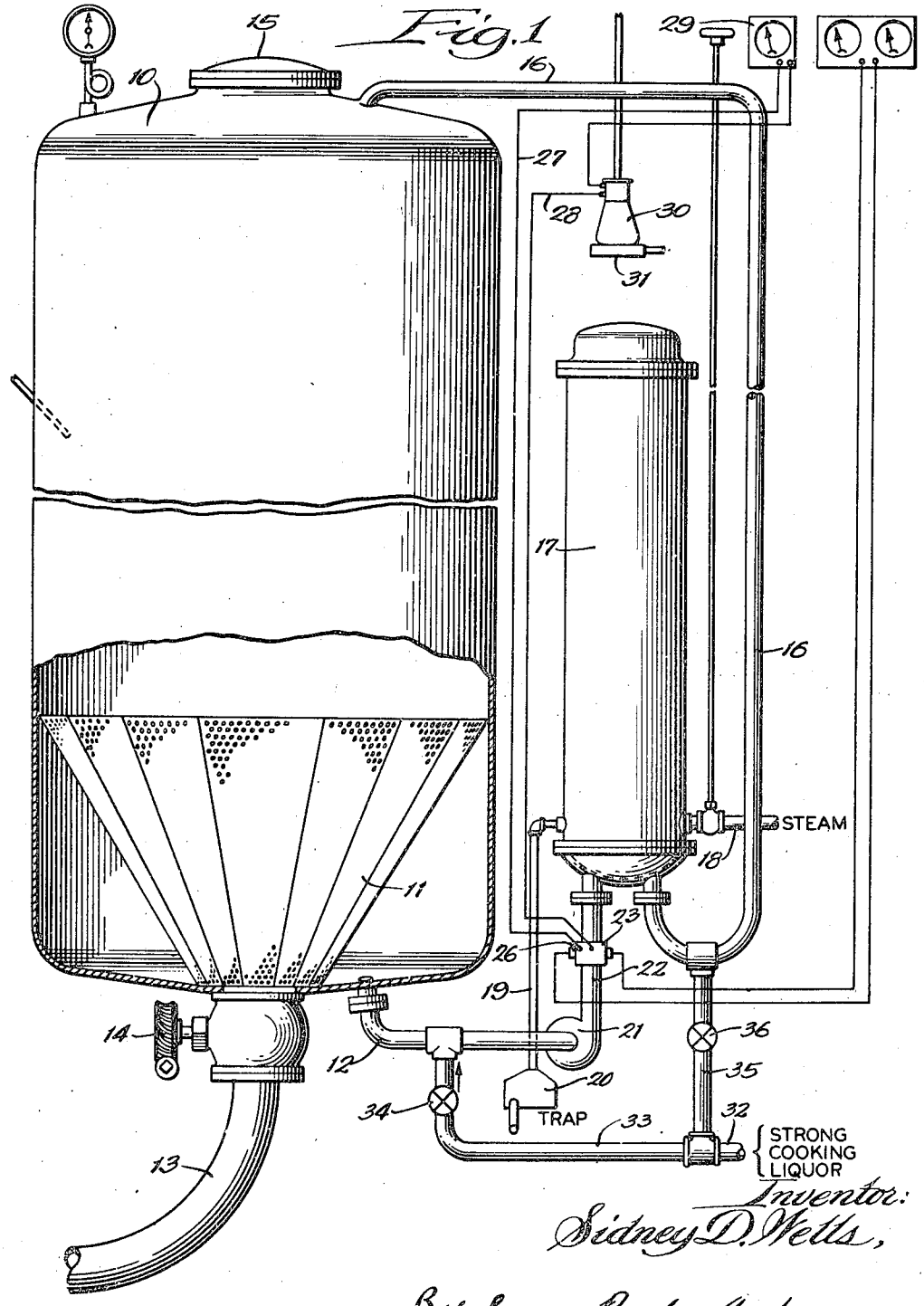
April 5, 1949. S. D. WELLS 2,466,290

METHOD OF DIGESTING PULP BY THE ALKALINE PROCESS

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Fig. 1



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Fig. 2

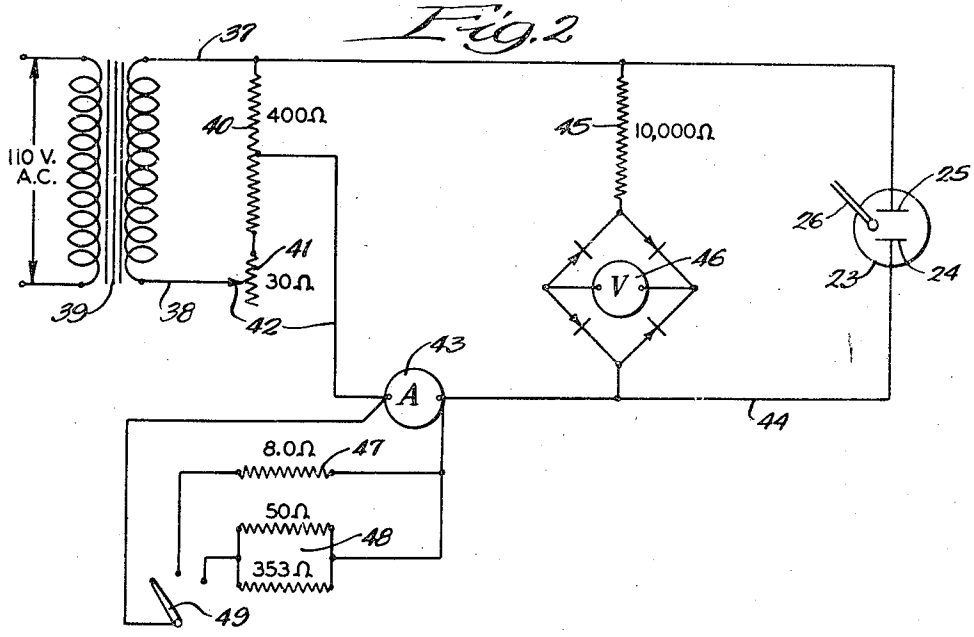
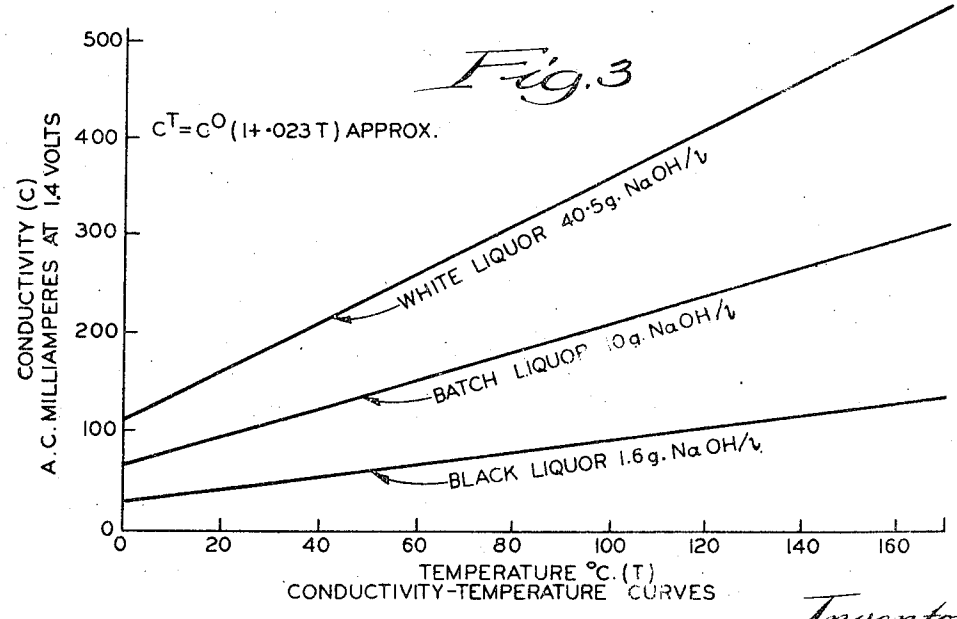


Fig. 3



CONDUCTIVITY-TEMPERATURE CURVES

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Fig. 4

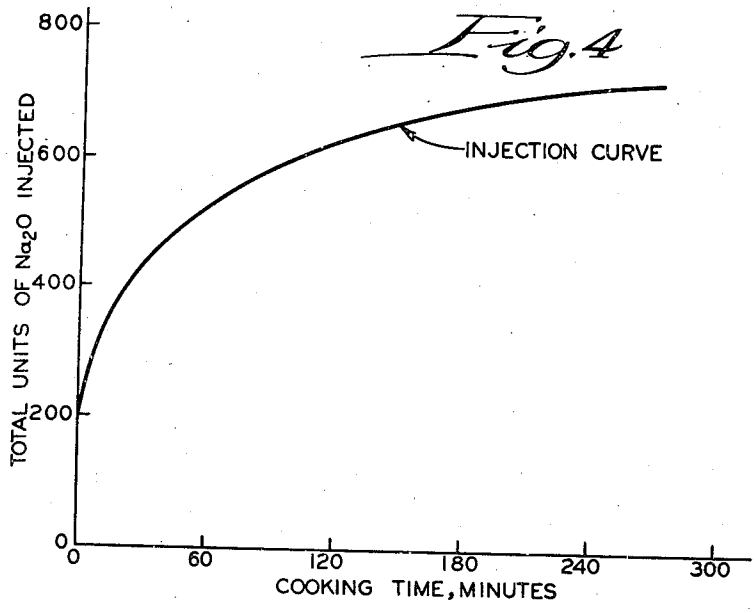


Fig. 5

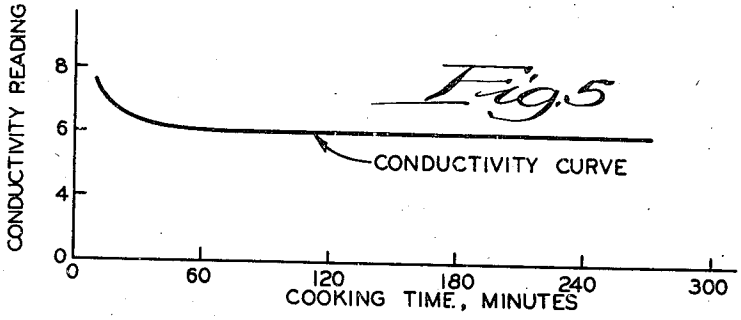
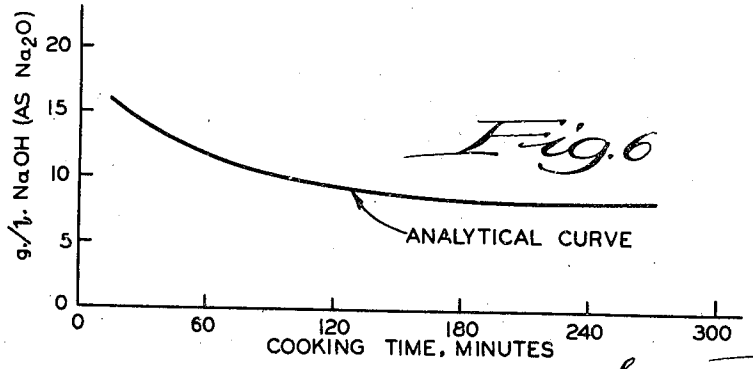


Fig. 6



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METHOD OF DIGESTING PULP BY THE ALKALINE PROCESS

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Fig. 7

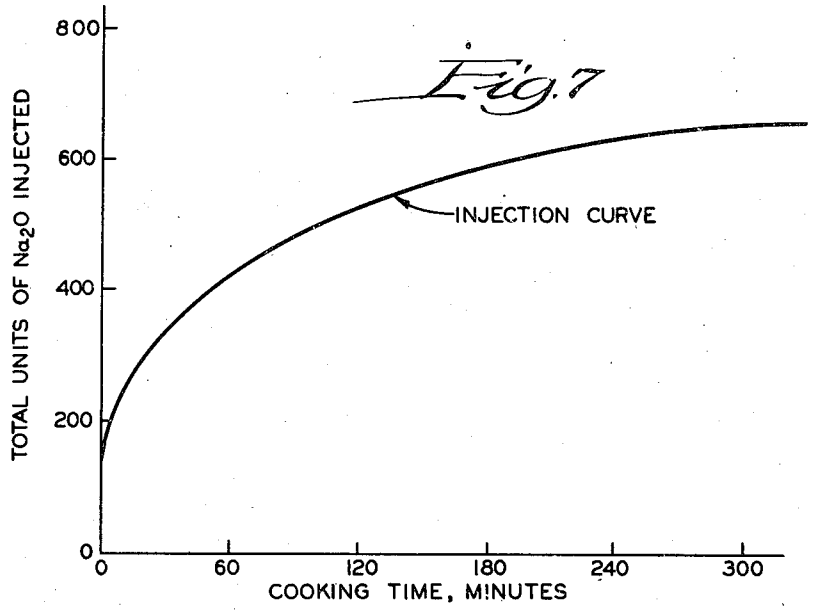


Fig. 8

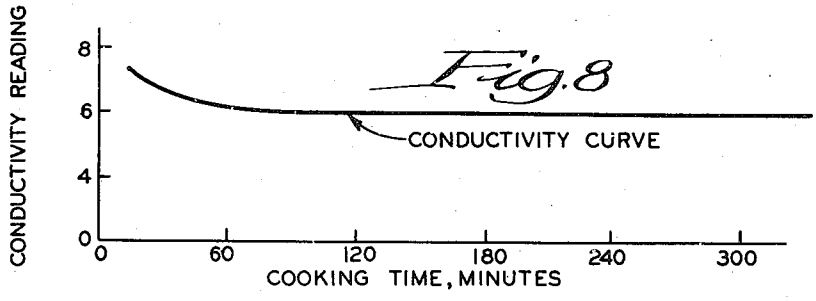
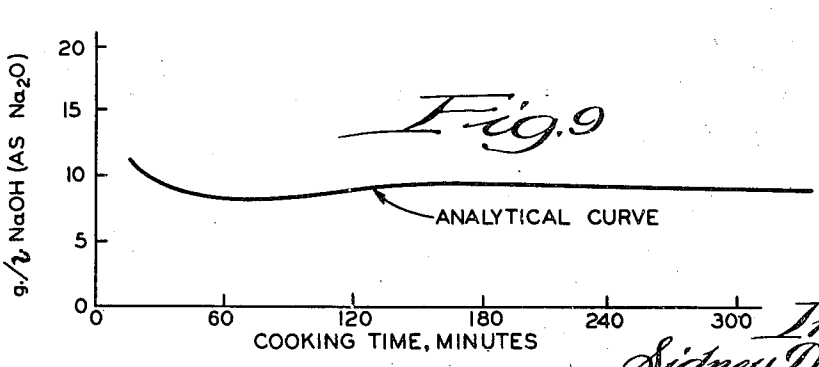


Fig. 9



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UNITED STATES PATENT OFFICE

2,466,290

METHOD OF DIGESTING PULP BY THE ALKALINE PROCESS

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Application March 15, 1944, Serial No. 526,537

1 Claim. (Cl. 92—13)

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This invention relates to improvements in a process for making pulp and has particular reference to the manufacture of chemical pulp used for paper-making and other purposes. The invention has been found to be of particular utility in connection with pulp made by the soda pulp or kraft process, although it may be employed in connection with other pulp processes.

In my previous U. S. Patent No. 1,949,669, dated March 6, 1934, I have described a process and apparatus for making kraft pulp, which differs from prior processes in that the cooking liquor, instead of being introduced into the digester in one batch at the start of the process, is fed more or less continuously or at intervals throughout the digesting cycle. The purpose of adding the chemical during the course of the treatment in place of adding all of it at the beginning of the cook is to prevent the development of undue alkalinity which may have a weakening effect upon the fibers.

However, due allowance must be made for the fact that, if, during the cook, the alkalinity drops to too low a figure, the fibers will become incrusted with deposits of undesirable materials, and the quality of the pulp will be seriously affected thereby. Therefore, it will be understood that, when a cooking operation is being carried on, the operator should, if he can, not only limit the concentration of alkali so as to prevent over-alkalinity, but, on the other hand, he should supply at all times throughout the cook enough alkali to prevent under-alkalinity from developing. This reconciliation of these two opposing requirements is difficult, first because of the different rates at which different batches of chips will consume chemical at various stages of the cook, and second because a greater total amount of chemical may be required in one cook than in another cook in order to arrive at an optimum final result when the cooking schedule is completed.

Said prior Patent No. 1,949,669 sets forth a schedule of liquor additions which contemplates the supply of more liquor during the early part of the cook than during later stages of the cook, in the effort to supply chemical in accordance with the need for it. Since there is more need for chemical at the beginning of the cook than at the later period when the cook is almost completed, more chemical is added during the early stages of the cooking cycle.

Although the process as set forth in my said previous patent works well when intelligently operated by experienced persons, and where the

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conditions as to character of raw material can be maintained substantially uniform, I have found that, in practice, there is considerable variation in the results obtained, especially under conditions encountered at the present time when it is difficult to obtain a constant and reliable supply of raw material, i. e. wood, of uniform quality and analysis. Also, under present day conditions, it is necessary to operate the digesting equipment with relatively inexperienced or unskilled help. Hence, it has become increasingly important to obtain a more simple and effective control of the processes employed in the digesting operation.

Other difficult problems are encountered in the digesting process. For example, in the kraft pulp process, as pointed out in said previous patent, it is an advantage to use the black liquor for a preliminary treatment of the chips. This in itself introduces a problem, because the black liquor resulting from a previous cook is always to some extent different from the black liquor which results from any other cook, and it is not feasible to standardize the black liquor. Hence there is often considerable uncertainty as to the effect of the preliminary black liquor treatment.

So far as I am informed the only way in which the efficiency of a cooking schedule has been evaluated heretofore has been by observing or testing the properties of the pulp after completion of the cook. Hence, the schedules adopted for admission of chemical, for example as set forth in my previous patent, have been largely the result of long experimentation, experience, or good guess-work, on the part of experienced operators, and the result has been a predetermined rigid program which is designed to fit the conditions which it is hoped will exist in the batch during the cooking period.

If such a standard schedule or program does not fit the requirements of any particular batch, the operator heretofore has been unable to discover the lack of fit, either before the cook or at any time during the progress of the cook, and not until after the end of the cook. The quality of the paper made from the pulp discloses whether any particular cook has been imperfectly, inefficiently or wastefully performed, but then it is too late to correct the error.

The principal object of this invention is to provide a way of regulating the admission of active chemical into the batch or circulating liquor in accordance with the condition or chemical activity of the circulating liquor at the time or about the time when the chemical is added, as distinguished from prior art systems in which

the precise schedule of chemical addition or injection has been determined prior to the commencement of the cooking cycle. Further objects of the invention and the advantages flowing therefrom will appear as the description proceeds.

One of the principal obstacles encountered in the development of a practical method of working out the program of chemical addition by self-determination, (i. e. in view of conditions developed and existing in the cycle itself) was the difficulty of making a more or less instantaneous determination of the chemical activity from time to time throughout the cooking cycle. This was due to the fact that even the simplest chemical analysis to determine the concentration of alkali or acid, as the case may be, requires an appreciable amount of time, and consequently, because of the time element involved, ordinary methods of making chemical analyses could not be employed. Hence, it was necessary to develop an instantaneous method of determining the chemical activity by some method differing from the usual chemical analysis procedure.

Hence an important feature of the invention consists in providing a method by which the chemical activity of the liquor can be instantaneously determined, computed, or compared, without resorting to time-consuming chemical analytical methods. This result has been accomplished through the discovery that the chemical activity of the liquor in a digester, so far as its usefulness in the cooking cycle is concerned, is a function of its electrical conductivity. Hence, in view of experience obtained through prior cooks, and the results thereof, it has been found possible to develop a schedule of optimum conductivity values throughout the cooking cycle, which conductivity values are readily translatable into values of chemical activity, i. e. active chemical concentration.

Having established a program of optimum conductivity values for the cycle, the program of admission of active chemical throughout the cycle is regulated or adjusted from time to time throughout the cooking cycle so as to produce the same program of conductivity values as have been found to represent optimum results in a previous cycle.

In the drawings, which illustrate a selected embodiment of my invention,

Fig. 1 is a vertical elevation showing one type of apparatus which may be usefully employed in carrying out the invention;

Fig. 2 is a diagram of the conductometric circuit;

Fig. 3 comprises a curve showing the relation between conductivity and temperature in the case of black liquor, and a similar curve for white liquor;

Fig. 4 is a curve showing the amounts of alkali injected during a cooking cycle in a typical example.

Fig. 5 is a curve showing the conductivity values as observed from time to time during said cooking cycle represented in Fig. 4;

Fig. 6 is a curve showing the results of analyses of actual samples of the circulating liquor during said cooking cycle represented in Fig. 4;

Fig. 7 is the alkali injection curve for another cook;

Fig. 8 is the conductivity curve for the cook shown in Fig. 7; and

Fig. 9 is the analytical curve for the cook shown in Fig. 7.

In general, it may be said that the apparatus, so far as the digester and its associated equipment for circulating and heating the liquor and admission of chemical are concerned, is about the same as disclosed in my previous Patent No. 1,949,669.

The digester 10 is of the usual construction, except that, at the bottom end, there is located a perforated cone 11, which serves as a screen for preventing chips from flowing out of the pipe 12 through which the liquor is withdrawn for circulating purposes. The blow-off or discharge pipe 13 is controlled by the usual valve 14, the chips are charged into the top of the digester through the manhole arrangement 15, and the circulating liquor returns to the digester through pipe 16.

The liquor circulates between the bottom digester outlet 12 and the upper inlet 16 through a heat exchanger 17 where said liquor is heated to any desired temperature by means of steam coils supplied through steam supply line 18. The condensate from the heat exchanger is discharged through a condensate discharge 19 and steam trap 20.

The circulating liquor is caused to travel through the heat exchanger 17 by means of a suitable pump 21, and between the pump 21 and the heat exchanger there is a pipe 22 in which is located an insulating sleeve 23 in which there are inserted a pair of opposed electrodes which, in Fig. 2, are represented by plates 24 and 25. Also, in said insulating sleeve 23, there is installed a thermocouple 26 connected by leads 27 and 28 to a micro-volt meter 29 and a connection or couple 30.

It will be understood that the couple 30 may be kept at a constant temperature, either 32° F. by means of ice, or at boiling temperature by means of a hot plate indicated at 31, so that the micro-volt meter 29 will be influenced only by changes in the temperature of the thermocouple 26 located in insulating sleeve 23.

The purpose of the thermocouple 26 is to enable the temperature of the circulating liquor at the electrode shown to be conveniently determined at a remote point where the conductivity is more readily observed than at a point adjacent the bottom of the digester. It will be understood that suitable piping injection arrangements are employed to facilitate an initial charging of the digester with black liquor and white liquor at the commencement of the cooking cycle. These are conventional and need not be illustrated since they form no part of the present invention.

The strong cooking liquor which is injected from time to time, or continuously in regulated amounts, throughout the cooking cycle, may be introduced at either of two points. It is supplied through the pipe 32 and may be injected into the pipe 12 between the digester and pump 21 through pipe 33 controlled by valve 34, or valve 34 may be closed and strong liquor may be injected directly into the digester inlet pipe 16 through pipe 35 and valve 36.

In the former case, the injected liquor will be heated up to the same temperature as the remainder of the liquor when it passes through the heat exchanger 17, but a slight allowance must be made for the increased chemical content when reading the conductivity, whereas, if the liquor is injected through pipe 36 directly into pipe 16, such injection will have no immediate effect upon the conductivity of the liquor passing between the electrodes.

Describing now the conductometric circuit, 24 and 25, as previously stated, are the electrodes which are located in the insulated pipe section 23. Low voltage current is supplied to the circuit through leads 37 and 38 attached to the terminals of the secondary coil of a conventional bell-ringing transformer 39 the primary of which is connected to an alternating current 110 volt supply line. Across the leads 37 and 38, there is connected a variable resistance consisting of a constant 400 ohm resistance coil 40 and a resistance variable up to 30 ohms represented by the coil 41 and arrow 42. Lead 42 is connected to the approximate center of the 400 ohm coil 42, so that between 37 and 42 there is a potential difference equal to half the voltage at the output terminals of the bell-ringing transformer 39.

The conductor 42 is connected to the electrode 24 through ammeter 43 and a conductor 44, so that in effect the current passing through the circulating liquid between the electrodes 24 and 25 is due to one-half of the voltage of the transformer, and is indicated by the reading of ammeter 43.

For the purpose of determining whether the electrical conditions are constant, a voltage measuring device is bridged across the lines 37 and 44. This may take the form of a high resistance element 45, for example of the order of 10,000 ohms, and a D. C. microammeter 46 (used as a volt meter) connected between conductor 44 and the end of high resistance 45 through a four-way instrument rectifier, as indicated in Fig. 2.

In order to obtain an indication of the proper amplitude for convenient reading on milliammeter 43, it may be advisable to use suitable shunt resistances, for example 47 and 48, which may be bridged across the terminals of the ammeter 43 by means of a suitable three-way switch 49.

It will be understood that the resistance 41 is adjusted to supply a given voltage across the electrodes, as indicated by the reading on the instrument 46, the milliammeter 43 having first been shunted to give a reading of satisfactory magnitude. The reading on ammeter 43 will indicate quite accurately the current which is flowing between electrodes 24 and 25 through the circulating liquid and is thus a satisfactory indication of the conductivity of the circulating liquor. Inasmuch as the conductivity of a liquor of constant concentration is a function of the temperature, and since the temperature of the liquor is changed considerably during the cooking cycle, it is advisable to make a proper allowance for such change in temperature, in order that the conductivity at any given temperature may be a reasonably accurate indication of the composition or concentration of the liquor. When making a conductivity observation, the proper correction is made by also observing the temperature reading on the thermocouple volt meter 29, whereupon, on reference to a scale of previously determined values, the true conductivity reduced to a given standard temperature can be readily computed.

The relation between temperature and conductivity, i. e. the milliamperes passing between electrodes at a voltage of 1.4 volts in the particular equipment referred to, is indicated in Fig. 3, the lower curve representing the change in conductivity and current for black liquor having a concentration of 1.6 grams of NaOH per liter, and the upper curve representing the differences in conductivity at different temperatures for white liquor having a concentration of 40.5 grams of NaOH per liter.

In view of the fact that both of these curves appear to be substantially in straight lines, it is obvious that by interpolation, the corresponding curves for any desired concentration of liquor between 1.6 grams per liter and 40.5 grams per liter may be constructed.

Figs. 4, 5 and 6 represent actual conditions in a typical cook selected for purposes of illustration. It will be seen, by an examination of said curves, that, during the cooking cycle of about 270 minutes, a total of about 700 units of Na_2O were injected. About 400 of such units were injected during the first 30 minutes, after which the rate of injection for maintaining optimum conditions was gradually reduced until at the end of the cycle the rate of injection of active chemical was reduced to almost zero.

Fig. 5 shows that the conductivity at the beginning of the cycle, no doubt due to the large amount of alkali admitted at the beginning of the cycle, was represented by a value of approximately 8 units on the conductivity meter, this figure dropping to a value of about 6 at the end of the first 30 minutes, and thereafter remaining substantially constant until the end of the cycle. The analysis curve representing the number of grams per liter of NaOH (as Na_2O) was found to be substantially constant, between 10 and 15 grams per liter, the latter figure being slightly exceeded during the very first part of the cooking cycle. These concentrations of alkali, at the times during which they existed, are found to be well within the range of values for optimum alkali concentration at those particular points in the cooking cycle.

It was found that an optimum program or schedule of alkali concentration throughout the cycle could be readily ascertained by adjusting the injection of liquor so as to maintain the conductivity reading at a substantially constant point (after correction for temperature) during the cycle. The results obtained by cooking various batches with the conductivity adjusted (by regulation of chemical injection) so as to give different conductivity readings for different batches made it possible to determine the optimum results and hence the optimum value at which to maintain the conductivity throughout the cooking cycle.

In Figs. 7, 8 and 9, it will be seen that the rate of injection was somewhat slower at the commencement of the cycle, and the total number of units injected was less than in the case of the preceding example, and in this second case the concentration of alkali (as determined by withdrawing samples from time to time) was lower than in the first case represented by Fig. 6. However, the conductivity curve shows that the reading was approximately 6 units throughout the cooking cycle, as shown in Fig. 8, being substantially the same as the first example shown in Fig. 5.

Although, in the second example, the period of cook was about 330 minutes as compared with 270 minutes in the first cook, and although the alkalinity in the first cook averaged substantially above 10 grams per liter, whereas in the second case the alkalinity averaged slightly less than 10 grams per liter, the results were equally satisfactory. This shows that, if the injection of liquor is so regulated that the conductivity reading is maintained as nearly as possible at about 6 units during the entire cooking cycle, good results

are obtained, regardless of minor variations in the alkali concentration, as determined by drawing off samples which were subsequently analyzed.

The scope of the invention should be determined by reference to the appended claim.

I claim:

The improved process of digesting cellulosiferous material in the production of chemical pulp suitable for paper making and other purposes, which consists in: (1) circulating alkaline cooking liquor continuously in a closed circuit through a batch of pulp in a digester; (2) adding supplemental alkali to the circulating stream of cooking liquor substantially continuously; (3) continuously reheating the circulating stream of cooking liquor and returning it to the digester; (4) continuously and automatically measuring the electrical conductivity of the stream of cooking liquor coming from the digester; and (5) controlling the additional supplemental alkali supply as a function of the measured conductivity of the liquor in the circulating stream; (6) said control being exerted to maintain the conductivity of said liquor substantially constant at a predetermined value after about the first quarter of the cook.

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