Process Control for Dissolving Pulp by SuperBatch Cooking

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Abstract: The process of producing dissolving pulp with SuperBatch cooking was introduced. The factors that affected the quality of the dissolving pulp and the corresponding operations were investigated.

Keywords: SuperBatch cooking; dissolving pulp; steam pre-hydrolysis; process control

1 Introduction

Dissolving pulp is a raw material consisting of regenerated cellulose and other cellulose derivatives and is widely used in products including viscose rayon, cellulose, and cellulose ester. The raw material used to produce dissolving pulp is wood and cotton linter\(^1\). Since 2007, because of the shortage of cotton linter, viscose mills have used dissolved wood pulp as the raw material instead of dissolved cotton pulp. Domestic pulp mills have built and rebuilt several dissolving pulp lines. In 2014, the global dissolving pulp capacity was 6.4–6.8 million tons; the device utilization was roughly 80\(^\circ\)\(^2\). Because the price of dissolving pulp remains low and the dissolving pulp capacity continues to increase, the amount of dissolving pulp released is expected to exceed 2.6 million tons in the next five years\(^3\).

Because of the development of the viscose industry, the price of viscose has fallen sharply, and competition has increased in intensity. However, the quality of domestically produced dissolving pulp is not good, and thus, it can only be used to produce viscose; the demand of dissolving pulp in other areas, such as the production of cellulose ether and acetate fiber, must be satisfied by imports. Therefore, improving the quality of the dissolving pulp is urgently needed.

A good dissolving pulp should have a high $\alpha$-cellulose content, low contents of ash, iron, and hemicellulose or other impurities (non-cellulosic components), and high brightness\(^4\). The degree of polymerization should be uniform, and the distribution should be as narrow as possible. The chemical reactivity of the dissolving pulp should be high, and good contact between cellulose and chemicals should be achieved\(^5\).

Received: 26 September 2016; accepted: 24 October 2016.
Many factors affect the quality of dissolving pulp, and among them, the cooking process is one of the most important. The SuperBatch cooking process has been demonstrated to produce uniform quality and high cellulose purity of dissolving pulp\(^6\), and its environmental impact is low. In this system, the conditions alternate between acid and alkali, and thus, the pipe and equipment scaling or plugging caused by hydrolysis can be reduced or even avoided, allowing the system to operate stably for long periods. In addition to advanced technology, strict operation and process control are required to obtain high-quality dissolving pulp.

2 Process control and its effect on dissolving pulp

There are two main stages in the SuperBatch pre-hydrolysis cooking process: the pre-hydrolysis stage and the lignin-removal stage; these stages are performed in the same reactor. The detailed cooking sequence is as follows (Fig.1): chip filling, heating and pre-hydrolysis, neutralization, hot liquor filling, heating and cooking, displacement and discharge\(^7\).

2.1 Chip filling with low-pressure steam

The cooking cycle begins with chip filling via low-pressure steam packing, which is the most important unit sequence for a successful displacement batch cooking process. The chips are conveyed to the digester via screw conveyors and fall into the digester because of gravity. Fresh low-pressure steam is used to facilitate packing the chips to obtain a uniform distribution of the chips in the digester; thus, more chips will be charged into the digester, and no chip mountain will form in the digester. If the chips are not distributed evenly, channeling may occur during the liquor filling in the neutralization, hot liquor filling, and displacement stages. Low-pressure steam also heats the chips and
displaces the air in the chips, allowing the cooking liquor to penetrate the chips easily and thus, react adequately.

As shown in Fig. 2, during chip filling, the pressure in the digester is controlled by the control valve on the steam pipe. The chips distribution depends on the pressure: when the pressure is high, the chips are packed against the digester wall; whereas when the pressure is low, there is no efficient method of distributing the chips evenly. The pressure is set by the operator based on the chip species, dimensions, and moisture. In addition, the chip flow and steam pressure must be stable to ensure that the chips are evenly vertically distributed in the digester. Chip filling takes 25~35 min depending on the capacity of the chip belt and the size of the digester.

![Fig. 2 Chip filling with steam](image)

2.2 Heating and pre-hydrolysis

When chip filling finished, the capping valve is closed. Low-pressure steam enters the digester through the steam pipes on the top and bottom, heating the chips and displacing the air. When the exhaust temperature exceeds the set value (95°C), the exhaust valve is closed, and simultaneously, the pressure in the digester increases as steam is added. When the pressure inside the digester is 100 kPa lower than the pressure of the low-pressure steam, the low-pressure valve is closed, and the middle-pressure steam valve is opened until the temperature reaches the set point. The digester is maintained at this temperature for about 90 min.

The purpose of hydrolysis is to purify the dissolving pulp, and the pre-hydrolysis is controlled by the P-factor, which is set by the operator using the control system. When the P-factor reaches the set point, the pre-hydrolysis is complete. A study on *Eucalyptus urophylla* revealed that xylan content in the range of 3%~4% could be easily achieved by applying P-factors of less than 300[8]. When the P-factor is below 500, the xylan content in pulp decreases quickly as the P-factor increases; when the P-factor is above 500, the xylan content decreases slowly as the P-factor increases. Lowering the xylan content to the value below 2%, as is required for the production of high-purity acetate-grade pulps, is accompanied by high losses in yield and drastic reductions in viscosity. The massive cellulose degradation induced by intensive pre-hydrolysis conditions (with P-factors >600) is also reflected by the alkali resistance of the R18 and R10 contents of the resulting unbleached pulps. As the pre-hydrolysis intensity increases, the R10 content decreases sharply, whereas the R18 content increases slightly, reaching a maximum at a P-factor of approximately 1500; thus, the uniformity of the pulp is reduced. In addition, at higher P-factors, more acid and gas are generated, and the risk of scaling in equipment and pipes increases. For eucalyptus steam pre-hydrolysis, P-factors of 500~600 and pre-hydrolysis temperatures of 168~170°C are appropriate.

2.3 Neutralization with white liquor

During the hydrolysis stage, large amounts of organic acids are generated by the hydrolysis of chips; thus, the hydrolysate is acidic, and the pH value is lower than 3. When the P-factor reaches the set value, the hot white liquor is pumped from the hot white liquor tank to the digester through the pipe at the bottom, forming a neutralizing liquor column, and the hydrolysate from the pre-hydrolysis stage is neutralized from the bottom to the top of the digester. Subsequently, alkali conditions are developed in the system for delignification. Approximately 60%~80% of the total white liquor is used in the neutralization stage.
The temperature and the speed at which the white liquor is added are very important during the neutralization stage. The neutralization process involves the reaction between the white liquor and chips. If the high-concentration alkali cannot penetrate the wood quickly, the fiber on the surface of the chips will degrade. Therefore, in actual production, the neutralization speed must be as fast as possible to reduce the contact time between the white liquor and the chips; otherwise, the pulp quality will not be uniform. Additionally, the waiting time between the pre-hydrolysis stage and the neutralization stage should be as short as possible. Typically, the neutral control period is approximately 15 min.

The neutralization reaction is an exothermic process, and therefore, the temperature of the hot white liquor used for neutralization must not be too high. If this temperature is too high, during the reaction with acid, a large amount of steam will be produced, and more gas will be emitted. High temperatures also accelerate the reaction between the white liquor and chips. The temperature of the hot white liquor in this stage is usually 135°C or even lower.

2.4 Hot liquor filling

After the neutralization stage, hot black liquor filling is performed. Hot black liquor from the hot black liquor accumulator displaces the neutralized liquor into the 2# hot black liquor tank via the pipe on the bottom. The displacement liquor is sent to the white liquor plant.

During dissolving pulp processing, the hot black liquor filling has two functions: the first is to raise the neutralized liquor column sufficiently to neutralize all of the hydrolysate in the digester, which requires the hot liquor filling to be rapid; the second is to adjust the hot black liquor to the desired level for the cooking liquor. The black liquor displaced from the previous cooking cycle is pumped to 1# hot black liquor accumulate tank, and the temperature should be approximately 150°C. Hot liquor filling reduces the steam consumption and shortens the cooking time, and simultaneously, the high concentration of sulfide ions in the hot black liquor accelerates the speed of the delignification reactions and improves the pulp strength and bleachability. To achieve an even distribution of the cooking liquor concentration in the digester, after hot liquid filling, the circulation pump is started. The temperature in the digester is slightly lower than the set cooking temperature.

2.5 Heating and cooking

After the hot liquor filling, the digester temperature is near the cooking temperature. Raising the temperature to the final cooking temperature is achieved by heating the circulating liquor. Some screens are located in the center of the digester through which the circulation liquor is sucked out of the digester by a circulation pump. Heating is performed by introducing medium-pressure steam into the circulation pipe. No heat exchanger is needed because of the small amount of steam required. Generally, 10–20 min of heating is sufficient.

During the cooking phase, the digester is kept at the desired cooking temperature and pressure until the target H-factor is reached. Because the H-factor (thus, the pulp cooking rate) depends strongly on the temperature, precise measurement and control of the temperature of the circulation liquor are vital.

The liquor circulation during the heating and cooking sequence passes through the suction screens in the middle section, and the liquor is returned into the digester through the displacement screens and bottom section. The displacement liquor from this step is routed to the hot black liquor accumulator. The amount of the circulation flow decreases as the cooking progresses because the internal resistance flow increases when the chips are cooked. The cooking time is highly dependent on the temperature, alkalinity, and wood species. Typically, cooking takes 40–60 min. For eucalyptus, the cooking temperature is approximately 168°C, and the H-factor is roughly 400–600. After cooking, the residual alkali in the pulp exceeds 15 g/L (as NaOH), which facilitates bleaching.
2.6 Displacement

The function of displacement is to terminate the cooking reaction and remove the hot black liquor from the digester for use in subsequent cooks.

In the SuperBatch cooking process, displacement starts by pumping liquor from the displacement liquor tank to the bottom of the digester while the H-factor reaches the set value. The displacement liquor is coming from brown stock washing stage and cooled from approximately 85~90°C to 75°C. The displaced hot black liquor is moved to the hot black liquor accumulator for the next cooking or evaporation. The amount of the displacement liquor used corresponds to the total volume of the brown stock washing filtrate. At the end of the displacement step, the pulp temperature is below 100°C.

In actual production, more attention must be paid to the amount of residual alkali in the displacement liquor. Indeed, when the residual alkali content is low, the removed lignin will reprecipitate onto the fiber, and the bleachability of the pulp will be reduced. Thus, a certain amount of white liquor needs to be added. The residual alkali concentration is approximately 5 g/L (as NaOH).

To prevent channeling, at the beginning of the displacement stage, the displacement liquor flow rate must be controlled to reduce its impact on the chip. If channeling occurs, the circulation pump should be turned on to close the channel flow. The displacement liquor volume depends on the dilution factor of the brown pulp washing. All of the displacement liquor must be used for displacement (no-bypass to evaporation) to improve the pulp washing during the displacement process. Displacement requires approximately 40~50 min, depending on the pumping equipment and the size of the digesters.

2.7 Discharge

After displacement, the pulp consistency in the digester is roughly 7%. The digester is gently emptied by pumping the pulp at a low digester pressure to a normal storage tower. During the discharge, the pulp is diluted in the bottom of the digester with liquor from the displacement liquor tank. The pulp temperature should be below 100°C to prevent the fiber from being damaged in the fiberline. This type of discharge method effectively keeps the fibers intact and prevents malodorous total reduced sulfides (TRS) emissions.

The pulp is pumped into a discharge tank at atmospheric pressure, and is under the level of the pulp to reduce foaming in discharge tank.

During the discharge, maintaining a stable pulp concentration facilitates controlling the brown pulp washing process. When the discharge concentration is higher, the fiberline is better controlled, but when the concentration is too high, discharging the pulp and emptying the digester are difficult. In general, the discharge concentration is approximately 5.5%.

When discharge is completed, the digester should be checked to confirm that it is empty. If some pulp remains, it will directly affect the hydrolysis of the next cooking. The weighing elements of the digester must be calibrated periodically. In addition, to prevent lignin deposition on the fibers, the residual alkali content must be controlled at roughly 6 g/L (as NaOH).

In general, the aim is to empty the digester as fast as possible. On average, emptying usually takes 20~30 min, depending on the sizes of the digester, pumps, and pipelines.

2.8 Tank farm

In the SuperBatch cooking process, the tank farm consists of three pressurized tanks and two atmospheric pressure liquor tanks. During the operation, attention should be paid to the following issues.

During the neutralization and hot liquor filling stages, hydrolysis gas is sent to the 2nd hot black liquor tank through the pipeline, and the pressure inside the tank increases. When the pressure reaches the set value, the pressure control valve opens, and acid gas moves into the cooler. To prevent cooler scaling and plugging, one digester should be in the displacement stage because the displacement liquor, which has a lower temperature, can condense large amount of the acid...
hydrolysis gas.

The warm black liquor in the 2nd hot black liquor tank is cooled in the heat exchanger and then sent to the black liquid filter to separate the fiber from the black liquor. The separated black liquor is then sent to the evaporation plant. A large amount of lignin is present in the black liquor. Thus, if the residual alkali content in the black liquor is low, the dissolved lignin will reprecipitate, causing scaling or plugging of the equipment and/or pipeline. Typically, the recycle pipeline around the 2nd hot black liquor tank is equipped with an online residual alkali analyzer. Therefore, when the residual alkali content is low, white liquor can be added in a timely fashion.

3 Conclusions

Good processes and equipment are required to produce high-quality dissolving pulp. However, the most important factor is that the operator knows how to control the process and has comprehensive and integrated knowledge of the cooking system.

References