

INFLUENCE OF XYLANASE PRETREATMENT ON REFINING ENERGY AND BRIGHTNESS OF P-RC APMP PULP OF ITALIAN BLACK POPLAR BRANCHES

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The study evaluates the influence of commercial xylanase (AU-PE89 and 51024) pretreatment applied in different refining stages of pulping on refining energy and properties of P-RC APMP pulp of Italian black poplar branches. Compared to the non-pretreated P-RC APMP pulp, the refining energy consumption of the pulps pretreated by xylanase before the second and third refining stages, performed during P-RC APMP pulping, decreased by 8-17% and 9-21%, respectively, while the brightness value of the pretreated pulp improved by 1.0-2.3 and 0.9-1.2% ISO, respectively, with minor changes in pulp strength properties. The pretreated pulp had longer fiber length, a higher fiber torsion index, smaller fiber width and a higher degree of cellulose crystallinity. The effect of xylanase pretreatment before the third refining stage was better, compared to the one applied before the second stage, as to refining energy saving, while the effect of xylanase AU-PE89 pretreatment was better than that of xylanase 51024.

Keywords: Italian black poplar branches, xylanase, pretreatment, P-RC APMP pulp, refining energy

INTRODUCTION

The fast-growing poplar species, widely planted in northern China,¹⁻³ are good raw materials for pulping and papermaking, due to their good mean fiber length, uniform fiber length distribution and high length/width ratio. Italian black poplar is one of the fast-growing poplar species cultivated and modified by researchers from Shandong Forestry Institute for growing in saline areas. Compared to other hardwood species, Italian black poplar possesses some advantages, such as better adaptability, easier breeding, good quality and high economic efficiency.

With the improvement of effective utilization of wood resources, high yield pulping technolo-

gies, such as preconditioning refiner chemical alkaline peroxide mechanical pulping (P-RC APMP), have been rapidly developed in the past ten years all over the world. The P-RC APMP pulping technology has many advantages,^{1,3} such as high pulp yield, mild pretreatment conditions, utilization of scrap fiber materials, and so on. However, there are some problems, such as high refining energy consumption and poor brightness stability. Energy consumption in the refining stage during high yield pulping is of about 18% of the total electrical energy cost for producing paper from wood. Thereby, energy saving has become necessary in pulping and papermaking industry.⁴⁻⁷

The utilization of enzymes in the paper industry has been popular since mid-1980s.⁸⁻¹⁸ Numerous studies on the application of xylanase in pulping and pulp bleaching of different hardwood, softwood and non-wood materials have been carried out.^{9,19-23} The beneficial effect of xylanase can be attributed to the degradation and elimination of the xylans from the fiber surface, and to the breaking of the existing linkages between cellulose and lignin, lignin being more easily removed during pulping or bleaching processes.²³⁻²⁴ Xylanase pretreatment can enhance fiber swelling and absorption capacity, thereby improving the refining properties of the pulp, and reducing refining energy.²³⁻²⁵ The influence of a xylanase (AU-PE89 and 51024) pretreatment on the refining and properties of the P-RC APMP pulp of Italian black poplar branches was studied, together with the morphology and properties of the fibers pretreated and non-pretreated with xylanase.

EXPERIMENTAL

Materials

Italian black poplar branches were taken from Laiwu, in the Shandong province. After manual debarking, the branches were chipped into sizes of 20-25 mm in length, 15-20 mm in width and 3-4 mm in thickness. The chemical composition (% dry weight, w/w) was the following: 1% NaOH extractives – 19.63%, benzene-alcohol extractives – 2.35%, holocellulose – 78.55%, Klason lignin – 18.09%, acid insoluble lignin – 1.89%, pentosan – 23.80% and ash – 0.56%. AU-PE89 xylanases were purchased from SUKEHAN Co. The characteristics of AU-PE89 xylanase were as follows: solid, activity 85000 IU/g, optimal pH value 6.5-8.0, and optimal temperature 50-55 °C. 51024 xylanases, purchased from Novozymes Biologicals, Inc., had the following characteristics: liquid, activity 17000 IU/mL, optimal pH value 6.5-7.0, and optimal temperature 45-55 °C.

The P-RC APMP pulp was produced in the pulping and papermaking laboratory of Shandong Polytechnic University, according to the following

process: wood chips → washing with 60-65 °C water → first stage extrusion with JS10 extruder (China) → first stage chemical pretreatment (3.3% NaOH (w/w), 3.0% H₂O₂ (w/w), 1.0% Na₂SiO₃ (w/w), 0.2% MgSO₄ (w/w), 0.2% EDTA (w/w), liquid ratio 1:4, 50 °C, 50 min) → second stage extrusion with JS10 extruder → second stage chemical pretreatment (3.0% NaOH (w/w), 3.0% H₂O₂ (w/w), 2.0% Na₂SiO₃ (w/w), 0.3% MgSO₄ (w/w), 0.3% EDTA (w/w), liquid ratio 1:4, 50 °C, 20 min) → first refining with KRK refiner (Japan) (consistency, 20% (w/v), with a refining gap of 0.50 mm) → high consistency retention → second refining with a KRK refiner (consistency, 20% (w/v), with a refining gap of 0.30 mm) → third refining with a KRK refiner (consistency, 20% (w/v), with a refining gap of 0.15 mm) → latency removal with 70-80 °C water for 30 min → refining with PFI mill (Japan) (consistency, 10% (w/v), with a refining gap of 0.25 mm, pressure, 3.33 N/mm) → ending.

Enzyme pretreatment

The xylanase pretreatment conditions were selected according to the previously determined experimental results on fast-growing poplar. Xylanase pretreatment was performed before the second or third refining stages. The pretreatment conditions for AU-PE89 xylanase were as follows: pulp consistency – 10%, xylanase dosage – 10, 20 and 30 IU/g (based on bone dry pulp), pH value 7.0, 50 °C, 90 min, while those for 51024 xylanase were: pulp consistency – 10%, xylanase dosage – 25 IU/g (based on bone dry pulp), pH value 6.5, 53 °C, 90 min.

300 g (bone dry pulp) of original pulp and xylanase were placed into a polyethylene plastic bag according to the xylanase pretreatment conditions. Subsequently, the bags were put into a thermostatic water bath at constant temperature, after mixing. During the reaction, the bags were taken out and heated every 10 min for assuring good pulp mixing, up to the setting time, then put into boiling water for 10 min to make the xylanase inactive and thus terminate the reaction. Finally, the pretreated pulps were washed thoroughly, being ready for further refining.

Handsheets forming and testing methods

Handsheets were formed on a PTI rapid handsheet former (Austria), under the following conditions: grammage – 60 g/m², drying temperature – 95 °C, drying time – 7 min, drying vacuum – 0.6 MPa, and conditioning treatment for 24 h in an atmosphere of 50% relative humidity, at 23 °C. The pulp and paper properties were measured according to the following standard methods: pulp was beaten according to ISO5264-2; handsheets were made according to ISO5269-2; Canadian standard freeness (CSF) was measured according to ISO5267-2; brightness – ISO2470; opacity – ISO2471; tensile index – ISO1924-1; tearing index – ISO1974; bursting index – ISO2578; folding number – ISO5626.

Fiber quality analysis (FQA)

The analysis of pulp fiber characteristics was done according to ISO16065. To prepare the sample, 0.1 g of the non-pretreated and pretreated P-RC APMP pulps were initially dispersed in 1000 mL of water, and 100 mL of fiber suspension was obtained. Fiber characteristics, such as fiber length, fiber width and fines content of pulp, were analyzed on a FQA device (OpTest, Canada), model LDA-02. The fiber length of pulp is expressed as an arithmetic mean (L_n), a length weighted mean (L_w) and a weight weighted mean (L_{ww}), as defined by Eq. (1)-(3), respectively.

$$L_n = \frac{\sum n_i L_i}{\sum n_i} \quad (1)$$

$$L_w = \frac{\sum n_i L_i^2}{\sum n_i L_i} \quad (2)$$

$$L_{ww} = \frac{\sum n_i L_i^3}{\sum n_i L_i^2} \quad (3)$$

where i is 1, 2 ...N categories of fiber length, n_i is fiber count in the “ i th” length category and L_i is contour length in the “ i th” category.

Scanning electronic microscopy (SEM) observations

The morphological characteristics of the fibers of P-RC APMP pulp, both non-pretreated and pretreated by xylanase, were observed by SEM. First, the samples

were dehydrated with 30, 50, 70 and 100% ethanol. Subsequently, the dehydrated pulp samples were frozen and vacuum-dried for 48 h. The specimens were gold-coated with gold-palladium in a Sputtergerät SCD 005 sputter coater (England). Sputter current of 60 mA, sputter time of 90 s, and film thickness of 20-25 nm were selected as coating conditions. The fiber surfaces of the samples were observed on a QUANTA 200 SEM (Holland).

X-ray diffractometer (XRD) measurements

The non-pretreated and treated pulp samples were first dehydrated, then frozen and vacuum-dried for 48 h. Subsequently, the dried samples were placed in a sample carrier, and their degree of crystallinity was analyzed with an X-ray diffractometer (XRD, D8 ADVANCE, Germany). The main scanning parameters were the following: Cu X-ray tube, tube voltage – 40 kv, tube electricity – 40 mA, scan speed – 0.03°/step and 0.1 s/step, scan range – 10°-50°. The degree of crystallinity of the samples was obtained with the relation:

$$X_c = \frac{F_k}{F_k + F_A} \times 100\%$$

where X_c is degree of crystallinity, F_k is crystalline area and F_A is amorphous area.

RESULTS AND DISCUSSION

Compared to APMP pulping, chemical preconditioning in P-RC APMP pulping is more relaxed, and preconditioning temperature (ranging from 40 to 50 °C) is similar to the pretreatment temperature of AU-PE89 and 51024 xylanases. Consequently, xylanase pretreatments were applied in P-RC APMP pulping processes, and the influence of xylanase pretreatment on refining energy, physical and optical properties of P-RC APMP pulp was investigated.

Xylanase AU-PE89 pretreatment before the second refining stage in P-RC APMP pulping

The influence of xylanase pretreatment, applied before the second refining stage of P-RC APMP pulping, on the refining energy of Italian

black poplar branches pulp is shown in Table 1. The results show that the refining energy of pretreated P-RC APMP pulp decreased from 3 to 8%, while refining revolution decreased in the subsequent PFI mill refining process by 2000 and 1000 r, respectively, up to reaching the same freeness for xylanase dosages of 20 and 30 IU/g, which means a decrease of refining energy consumption by 9 and 5%, compared to the non-pretreated P-RC APMP pulp. Xylanase may degrade xylan from the fiber surface and make fiber structure porous and incompact, which favoured pulp beating and refining. However, no energy saving was recorded at a xylanase dosage of 10 IU/g. Consequently, the optimal dosage of AU-PE89 xylanase was of 20 IU/g, which assured an energy saving of 21%.

According to Table 2, compared to the non-pretreated P-RC APMP pulp, the brightness of the P-RC APMP pulp pretreated by xylanase AU-PE89 was significantly improved, while the physical properties were only slightly changed. The brightness of the pretreated P-RC APMP pulp was improved by over 1.0% ISO, reaching the maximum increase, of 2.3 %ISO, at an AU-PE89 xylanase dosage of 30 IU/g. The xylan on the fiber surface was degraded partially by xylanase, while the structure of the lignin-carbohydrate complex (LCC) was destroyed, part of the lignin being dissolved and wiped out from the pulp by washing, which may explain why the xylanase pretreatment improved the brightness of P-RC APMP pulp. The physical strength properties of the pretreated pulp were improved at xylanase AU-PE89 dosages of

20 and 30 IU/g. Therefore, the optimal dosages of xylanase were of 20 and 30 IU/g. In short, considering the slight difference between the effects of xylanase-assisted refining on xylanase dosages between 20 and 30 IU/g, and taking into account the xylanase pretreatment cost, the optimal dosage of xylanase AU-PE89 pretreatment before the second refining stage was selected to be 20 IU/g.

Xylanase pretreatment before the third refining stage in P-RC APMP pulping

Table 3 shows the influence of xylanase pretreatment, applied before the third refining stage of P-RC APMP pulping, on refining energy. As seen, the pretreated P-RC APMP pulp showed a decrease of refining energy between 4-9%, for different xylanase dosages, a decrease of refining revolution by 1000 and 3000 r, on reaching the same freeness during subsequent PFI mill refining, which means a decrease of refining energy consumption of 5-13%, compared to non-pretreated P-RC APMP pulp. The positive effect of xylanase can be attributed to the degradation and elimination of the xylans from the fiber surface, and to breaking of the existing linkages between cellulose and lignin, which favoured pulp refining, and resulted in reduced refining energy. The effect of xylanase-assisted refining at a xylanase AU-PE89 dosage of 20 IU/g was the best, and the optimal dosage of xylanase AU-PE89 was of 20 IU/g, with an energy saving of 22%.

Table 1
Comparison of refining energy of non-pretreated and pretreated P-RC APMP pulps by xylanase AU-PE89^a

Characteristics	Non-pretreated pulp	Pulp pretreated by xylanase AU-PE89		
		10 IU/g	20 IU/g	30 IU/g
Refining energy, kw/h·t	2562	2485	2362	2450
Freeness (CSF), mL	737	763	737	737
PFI refining revolutions, r	22000	22000	20000	21000
Freeness(CSF), mL	280	285	282	280

^a Beating gap, 0.25 mm

Table 2
Comparison of physical and optical properties of non-pretreated and pretreated P-RC APMP pulps by xylanase AU-PE89^a

Pulp properties	Non-pretreated pulp	Pulp pretreated by xylanase AU-PE89		
		10 IU/g	20 IU/g	30 IU/g
Brightness, % ISO	74.1	75.6	76.1	76.4
Opacity, %	80.9	79.5	79.9	80.3
Tensile index, kN/m	38.91	38.87	39.12	38.93
Tearing index, mN·m ² /g	4.61	4.59	4.63	4.71
Bursting index, KPa·m ² /g	1.82	1.79	1.84	1.87
Folding number, (135°)/time	14	12	15	17

^aPFI mill refining g gap, 0.25 mm; freeness (CSF), 284 mL

Table 3
Comparison of refining energy of non-pretreated and pretreated P-RC APMP pulps by xylanase AU-PE89 and 51024^a

Characteristics	Untreated pulp	Pulp pretreated by xylanase AU-PE89		Pulp pretreated by xylanase 51024
		20 IU/g	30 IU/g	25 IU/g
Refining energy, kw/h·t	2486	2295	2369	2398
Freeness(CSF), mL	725	725	763	737
PFI beating revolutions, r	23000	20000	22000	21000
Freeness(CSF), mL	255	255	261	249

^aPFI mill refining gap, 0.25 mm

The effect of the xylanase AU-PE89 pretreatment on energy saving was higher than that of the xylanase 51024 pretreatment.

Table 4 shows the influence of xylanase pretreatment on the physical and optical properties of P-RC APMP pulp. Compared to the non-pretreated P-RC APMP pulp, both brightness and opacity of the pretreated P-RC APMP pulp were improved, while the physical properties were only slightly changed. The brightness of the pretreated P-RC APMP pulp was improved in the 0.9-1.2% ISO range. The xylan from the fiber surface was partially degraded by xylanase, while the LCC structure was destroyed, and part of lignin was dissolved and wiped out from the pulp by washing, which explains why the xylanase pretreatment improved the brightness of the P-RC APMP pulp. The maximum increase in the brightness and opacity of the pretreated pulp was obtained by using xylanase AU-PE89, at a dosage of 30 IU/g. The physical strength properties of the pretreated pulp were similar to those of the untreated pulp, at xylanase AU-PE89 dosages between 20 and 30 IU/g. The effect of xylanase

AU-PE89 pretreatment on improving pulp properties was higher than that of xylanase 51024 pretreatment. Therefore, considering the small difference in the effects of xylanase-assisted refining, at different xylanase dosages and xylanase types, and taking into account the xylanase pretreatment cost, the optimal dosage of xylanase AU-PE89 pretreatment before the third refining stage was considered to be 20 IU/g.

Analysis of fiber characteristics

Table 5 lists the FQA analysis results for non-pretreated and pretreated pulp fibers. Compared to the untreated pulp fiber, the fiber length (L_n , L_w and L_{ww}) of the pretreated pulp increased slightly, while fiber width and fines decreased, and fiber torsion increased. Compared to chemical pulps (such as kraft pulp and soda-AQ pulp), high-yield pulps (such as APMP and BCTMP) have a higher fines content resulting from the refining process. Due to the relatively larger surface area of the fines fiber, xylanase can easily find and degrade these fines, hence the fiber length of the pretreated pulp was longer than that

of the non-pretreated pulp. The conclusion is that a xylanase pretreatment can easily split and torque

fibers, avoid cutting during subsequent refining or beating, and improve strength properties.

Table 4
Comparison of physical and optical properties of non-pretreated and pretreated P-RC APMP pulps by xylanase AU-PE89 and 51024^a

Pulp properties	Non-pretreated pulp	Pulp pretreated by xylanase AU-PE89		Pulp pretreated by xylanase 51024
		20 IU/g	30 IU/g	25 IU/g
Brightness, %ISO	73.7	74.7	74.9	74.6
Opacity, %	77.9	77.7	78.1	77.8
Tensile index, kN/m	36.32	36.35	36.55	35.74
Tearing index, mN·m ² /g	1.65	1.66	1.67	1.66
Bursting index, KPa·m ² /g	4.04	4.13	4.40	4.38
Folding number (135°)/time	9	9	10	8

^a PFI mill refining gap, 0.25 mm, pulp freeness (CSF), 255 mL

Table 5
Analysis of fiber characteristics of non-pretreated and pretreated P-RC APMP pulp by xylanase AU-PE89^a

Fiber characteristics	Non-pretreated pulp	Pulp pretreated by xylanase AU-PE89 before the second refining stage	Pulp pretreated by xylanase AU-PE89 before the third refining stage
Arithmetic length, mm	0.530	0.548	0.552
Length weighted length, mm	0.631	0.647	0.656
Weight weighted length, mm	0.726	0.759	0.772
Fiber width, μm	21.3	20.3	20.4
Fibers torsion, mm	0.52	0.56	0.59
Fines, %	37.47	35.89	35.12

^a Xylanase AU-PE89 pretreatment conditions: pH value 7.0, dosage – 20 IU/g, 50 °C, 100 min, consistency – 10%, pulp freeness (CSF) – 255 mL

Morphology of fiber surface

Figure 1 shows the SEM observations on non-pretreated and pretreated pulp fibers. The fibers were taken from the untreated and treated pulp refined at a freeness (CSF) of 284 mL. The dosages of xylanase were of 20 IU/g during the pretreatment, before the second refining stage of P-RC APMP pulping. As shown in Figure 1, the non-pretreated pulp appeared stiffer, and had a lower fibrillation extent. In addition, the xylanase-pretreated fibers were softer and had a higher fibrillation extent. The pretreated fibers had a higher fiber-bonding capability, which increased the pulp strength properties. Compared to the chemical pulp, the fibers of high-yield pulps (HYP) were inflexible and short, which resulted in poor fiber-bonding capability. The xylanase pretreat-

ment may modify the properties of HYP by hydrolyzing micro-fiber on the fiber, by degrading xylan and destroying the LCC structure, it may make the HYP fiber flexible and improve its fiber-bonding capability. The enzymatic pretreatment loosened the fiber cell wall and softened the fiber, making it easier to refine and beat, thus leading to energy saving.

XRD analysis of pulp

Figure 2 shows the XRD analysis results for non-pretreated and pretreated pulp fibers. The fibers were taken from the untreated and treated pulp refined at a freeness (CSF) of 284 mL. The dosage of xylanase was of 20 IU/g in the pretreatment before the second refining stage of P-RC APMP pulping.

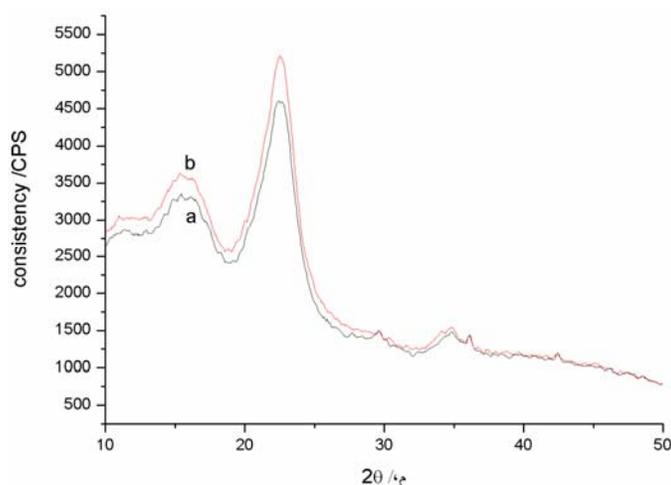
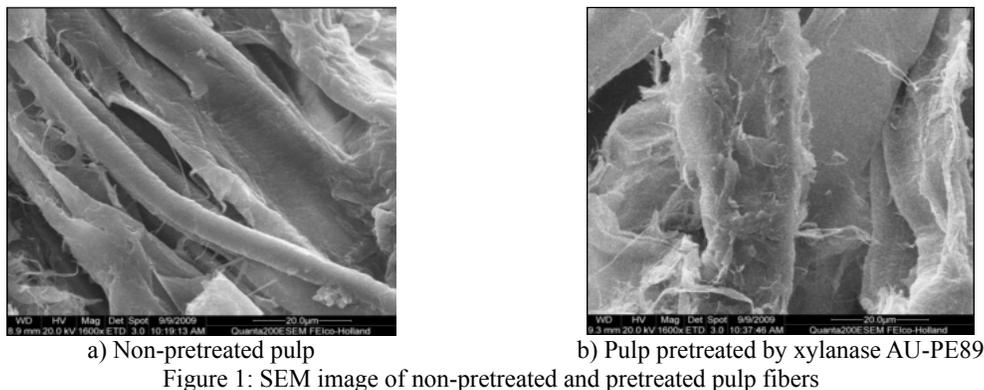


Figure 2: XRD patterns of non-pretreated and pretreated pulp fibers: (a) non-pretreated pulp; (b) pulp pretreated by xylanase AU-PE89

Figure 2 shows that the crystallinity degree of the cellulose from P-RC APMP pulp fibers increased after the xylanase pretreatment. The degree of crystallinity of the non-pretreated pulp was of 61.36%, and that of the P-RC APMP pulp pretreated by xylanase was of 62.75%. Xylanase may reach the amorphous area of pulp fibers to hydrolyze micro-fiber more easily, but the crystalline area of the pulp fibers may be difficult to affect. The crystalline area of the pretreated pulp was wider than that of the non-pretreated pulp, which may explain why the crystallinity degree of pretreated pulp was higher than that of non-pretreated pulp.

XRD analysis indicated that xylanase pretreatment was beneficial for the improvement of the crystallinity degree of cellulose. The increase of the crystallinity degree of the pretreated pulp may contribute to the increase of the pretreated pulp fiber strength, enhance fiber

flexibility and avoid severe cutting of fibers, which is responsible for the improvement of the physical strength properties of the pretreated P-RC APMP pulp and for the decrease of refining energy consumption in P-RC APMP pulping.

CONCLUSIONS

Compared to the non-pretreated P-RC APMP pulp of Italian black poplar branches, the P-RC APMP pulp pretreated by xylanase AU-PE89 before the second refining stage of pulping showed a decrease of refining energy between 3-8%, and a decrease of refining energy consumption in subsequent PFI mill refining by 9-5%, while reaching the same freeness at xylanase dosages of 20 and 30 IU/g, respectively, as well as an increase in the brightness of the pretreated pulp between 1.5-2.3 %ISO. At an optimal dosage of AU-PE89 xylanase (20 IU/g), the energy saving was of 21%, and the physical and optical properties of the

pretreated pulp were improved.

Compared to the non-pretreated P-RC APMP pulp of Italian black poplar branches, the P-RC APMP pulp pretreated by xylanase before the third refining stage of pulping showed a decrease of refining energy between 4-9%, and a decrease of refining energy consumption in the subsequent PFI mill refining process of 5-13%, on reaching the same freeness, and an increase in the brightness of the pretreated pulp of 0.9-1.2 %ISO. The effect of xylanase AU-PE89 pretreatment was more significant than that of xylanase 51024 pretreatment. The optimal dosage of xylanase AU-PE89 was of 20 IU/g, leading to an energy saving of 22%, and improving the physical and optical properties of the pretreated pulp.

The fiber length of xylanase-pretreated pulp increased slightly, while the fiber width and fines content decreased. The fiber torsion increased. Compared to the non-pretreated fiber, the fibers pretreated by xylanase were softer and had good flexibility, which explained the decrease of refining energy consumption in the P-RC APMP pulping. The xylanase pretreatment improved the crystallinity of cellulose, which can enhance the physical strength properties of the P-RC APMP pulp.

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