

Supporting Information

Preparation of Nanocellulose with High-Pressure Homogenization from Pretreated Biomass with Cooking with Active Oxygen and Solid Alkali

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Table of Contents

Sections	Page
Experimental:	S1
Figures:	S2
Tables:	S3-S5

EXPERIMENTAL

The process of cooking with active oxygen and solid alkali (CAOSA) to treat WP or BR

A pre-processing and post-processing sketch of CAOSA showed as Figure S1b. The raw materials WP and BR were milled to the size of 10 - 20 mesh and cooked in ball-shaped digester (independently designed by our group and built by Yantai Keli Chemical Equipment Company) with active oxygen and solid alkali, Na_2CO_3 . 1 kg of WP or BR (dry weight) was cooked with 4 kg of water and 150 g Na_2CO_3 at an initial oxygen pressure of 2.0 MPa and temperature of 160 °C for 1/2/3/5/10 h. The reaction that was allowed to continue for 10 h proceeded to near completion.

Cooling water was injected into the jacket of the reactor to quench the reaction, and the pressure was reduced through a blow-down valve. The muddy product, which was a mixture of pulp and YL, was filtered using filter cloth of 100 mesh to separate the YL and the solid pulp. The solid was further washed several times with clean water and stored as the pulp produced by the CAOSA method. The pulp is referred to as WP-CAOSA or BR-CAOSA.

FIGURES

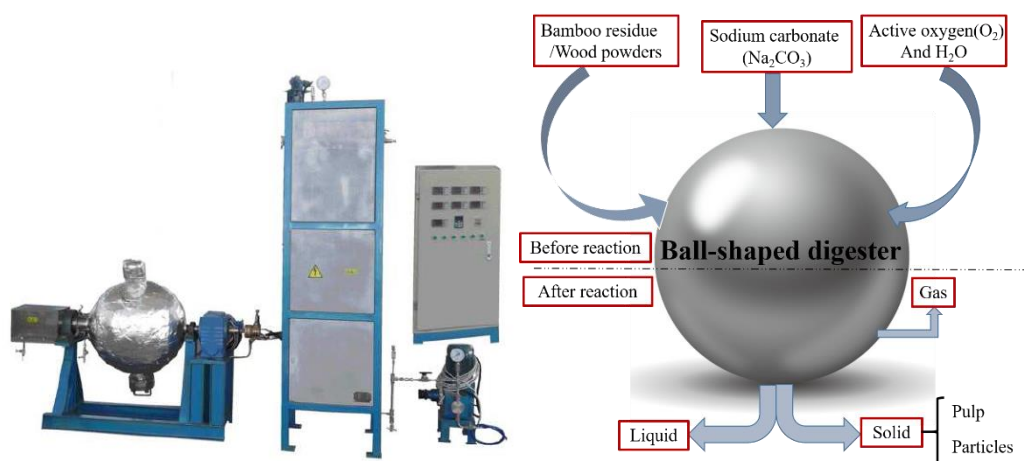


Figure S1. The image of ball-shaped digester and the process of CAOSA

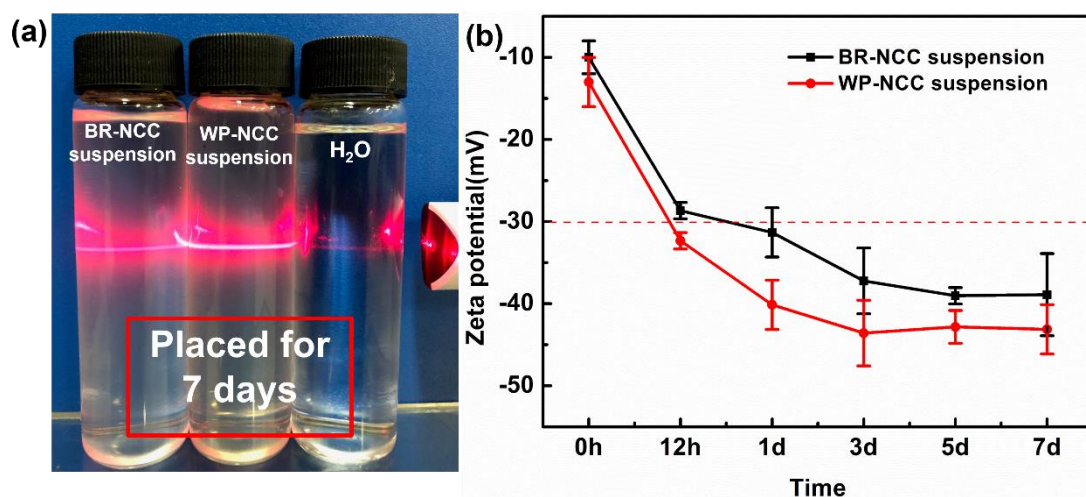


Figure S2. (a) Visual image after storage of BR-NCC and WP-NCC solution for seven days after dispersion and its Tydal phenomenon compared with H₂O; (b)

Zeta-potential of BR-NCC and WP-NCC

TABLES

Table S1 List of the fibers obtained at each stage of the NCC isolation process.

Samples	Condition
WP	Untreated- Spruce wood powder
WP-CAOSA	CAOSA process treat WP raw material
WP-CAOSA-B	CAOSA process treat WP+ Bleaching at 10%(w/w)H ₂ O ₂
WP-NCC	CAOSA process treat WP+ Bleaching at 10%(w/w)H ₂ O ₂ + high-pressure homogenization
BR	Bamboo residue
BR-CAOSA	CAOSA process treat BR raw material
BR-CAOSA-B	CAOSA process treat BR +Bleaching at 10%(w/w)H ₂ O ₂
BR-NCC	CAOSA process treat BR +Bleaching at 10%(w/w)H ₂ O ₂ + high-pressure homogenization

Table S2 Chemical compositions of the raw material and the corresponding bleached and unbleached pulps.

Constituent	WP[% (w/w)]			BR[% (w/w)]		
	Untreated	WP-CAOSA Pulp	Bleached pulp	Untreated	WP-CAOSA Pulp	Bleached pulp
Lignin	21.6	6.2	1.3	27.64	11.2	3.1
Cellulose	48.5	81.6	88.4	50.51	75.6	84.3
Hemicelluloses	24.8	10.1	9.8	21.3	11.6	10.3
Ash	0.59	0.50	0.2	0.4	0.36	0.2

Table S3 Infrared transmittance peaks (cm⁻¹) of the fibres in different stages.

Samples	-OH stretching	C-H vibration	Absorbed water	C-H stretching	Aromatic ring vibration of lignin	C-C stretching
BR	3468	2923	1637	1386	1250	1120
BR-CAOSA	3405	2900	1596	1375	1242	1112
BR-CAOSA-B	3465	2940	1636	1382	-	1112
WP	3450	2921	1637	1390	1256	1121
WP-CAOSA	3242	2902	1597	1375	1220	1124
WP-CAOSA-B	3412	2900	1636	1374	-	1131

Table S4 Thermal properties of WP and BR fibers after various stages of treatment

Samples	T _{on} (°C)	T _{max} (°C)	Residue at 600 °C (%)
WP	302.34	348.78	29.05
WP-CAOSA	243.42	297.23	32.58
WP-CAOSA-B	306.98	345.27	19.40
WP-NCC	280.72	328.12	18.58
BR	294.62	352.09	30.10
BR-CAOSA	238.51	288.95	32.55
BR-CAOSA-B	314.35	345.45	25.25
BR-NCC	300.95	343.87	22.74

Table S5 Nanocellulose concentration with varying time of placement

Time	0h	12h	1day	3days	5days	7days
Concentration	1.0wt%	0.86wt%	0.69wt%	0.62wt%	0.63wt%	0.60wt%

Table S6. Effect of reaction time on the properties of nanocellulose

Condition (temperature, concentration, pressure, time)	PDI	AS/nm
100°C,10%,100Mpa,10min	0.44	264
100°C,10%,100Mpa,20min	0.35	262
100°C,10%,100Mpa,30min	0.29	232
100°C,10%,100Mpa,60min	0.28	210