

Supplementary Information

Preparation and Characterization of Cellulose Nanocrystals from Corncob via Ionic Liquid

[Bmim][HSO₄] Hydrolysis: Effects of Major Process Conditions on Dimensions of the

Product

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Table. S1 Process conditions determined by central composite design (CCD) for BmimHSO₄ hydrolysis of corncob

| Std order | Run order | Parameter | | | Size (nm) Measured | Size (nm) Predicted |
|-----------|-----------|------------------|------------------|----------|-----------------------|------------------------|
| | | Mass percent (%) | Temperature (°C) | Time (h) | | |
| 17 | 1 | 8 | 100 | 1.5 | 303 | 317 |
| 10 | 2 | 1 | 80 | 2 | 209 | 272 |
| 4 | 3 | 5 | 46 | 2 | 270 | 285 |
| 11 | 4 | 2 | 60 | 2.5 | 263 | 223 |
| 1 | 5 | 2 | 100 | 2.5 | 166 | 161 |
| 20 | 6 | 2 | 60 | 1.5 | 461 | 443 |
| 18 | 7 | 5 | 80 | 2 | 402 | 379 |
| 19 | 8 | 8 | 100 | 2.5 | 529 | 521 |
| 3 | 9 | 5 | 110 | 2 | 173 | 200 |
| 8 | 10 | 5 | 80 | 2 | 499 | 379 |
| 15 | 11 | 2 | 100 | 1.5 | 211 | 151 |
| 13 | 12 | 5 | 80 | 2.8 | 336 | 372 |
| 2 | 13 | 8 | 60 | 2.5 | 343 | 377 |
| 9 | 14 | 5 | 80 | 2 | 421 | 379 |
| 5 | 15 | 10 | 80 | 2 | 547 | 512 |
| 16 | 16 | 5 | 80 | 2 | 358 | 379 |
| 12 | 17 | 5 | 80 | 1.2 | 307 | 386 |
| 14 | 18 | 5 | 80 | 2 | 333 | 379 |
| 7 | 19 | 5 | 80 | 2 | 346 | 379 |
| 6 | 20 | 8 | 60 | 1.5 | 424 | 403 |

Table. S2 Coded levels of three variables in CCD matrix

| | Variables | Units | Coded levels | | | | |
|---|--------------|-------|--------------|-----|----|-----|------------|
| | | | - α | -1 | 0 | 1 | + α |
| A | Mass percent | % | 1 | 2 | 5 | 8 | 10 |
| B | Temperature | °C | 46 | 60 | 80 | 100 | 110 |
| C | Time | h | 1.2 | 1.5 | 2 | 2.5 | 2.8 |

Table. S3 Analysis of variance (ANOVA) of the regression equation

| Source | Sum of Squares | Degree of freedom | Mean Square | F-value | P-value | |
|----------------------------|----------------|-------------------|-------------|---------|---------|-----------------|
| Model | 199594 | 7 | 28513.4 | 8.23 | 0.0009 | significant |
| A-Mass fraction of corncob | 80324.8 | 1 | 80324.8 | 23.18 | 0.0004 | |
| B-Temperature | 18029.6 | 1 | 18029.6 | 5.2 | 0.0416 | |
| C-Time | 228.89 | 1 | 228.89 | 0.07 | 0.8015 | |
| AB | 21218 | 1 | 21218 | 6.12 | 0.0292 | |
| AC | 18818 | 1 | 18818 | 5.43 | 0.038 | |
| BC | 26450 | 1 | 26450 | 7.63 | 0.0172 | |
| B ² | 37655.7 | 1 | 37655.7 | 10.87 | 0.0064 | |
| Residual | 41577.2 | 12 | 3464.76 | | | |
| Lack of Fit | 22442.3 | 7 | 3206.05 | 0.84 | 0.5998 | not significant |
| Pure Error | 19134.8 | 5 | 3826.97 | | | |
| Cor Total | 241171 | 19 | | | | |

Std. Dev. 58.86 **R²** 0.8276
Mean 344.95 **Adjusted R²** 0.7270
C.V. % 17.06 **Predicted R²** 0.5040
Adeq Precision 9.9301

Table S4 Characteristics of CNC produced by ionic liquid hydrolysis and traditional methods

| Reagents | Condition (Temp, Time) | Raw material | Pretreatment | Diameter (nm) | Length (nm) | CNC yield (%) | CrI (%) | Decomposition Temperature (°C) | References |
|--|------------------------------|----------------------------------|--------------------------------------|------------------|----------------|------------------|------------|--------------------------------------|------------|
| H ₂ SO ₄ | 45 °C, 1 h | Corncob | 3% NaOH, bleaching by NaOCl | 198 | 5.5 | 35 | 55.9 | 313 | 1 |
| H ₂ SO ₄ | 45 °C, 1 h | Corncob | 2% NaOH, bleaching by acetate buffer | 210.8 | 4.15 | 41 | 83.7 | 276 | 2 |
| H ₂ SO ₄ | 50 °C, 20 min | Corn stover | 2% NaOH, bleaching by acetate buffer | 7 | 356.3 | 64 | 55.04 | - | 3 |
| [Bmim][HSO ₄]/H ₂ O | 100 °C, 12 h | Microcrystalline Cellulose (MCC) | N/A | 3.6 | 146 | 48 | 82 | - | 4 |
| | 130 °C, 12 h | Softwood pulp | Bleaching | 7 | 219 | 60 | 78 | - | |
| | 130 °C, 12 h | Hardwood pulp | Bleaching | 6 | 227 | 56 | 77 | - | |
| [Bmim][HSO ₄] | 80 - 120 °C, 2 h | Cotton linter | Defatting | 50-100 | 500-800 | 33 | 69 | 365 | 5 |
| [Bmim][HSO ₄] | 100 °C, 1.5 h | corncob | 2% KOH | 52 | 182 | 40 | 63 | 334 | This study |

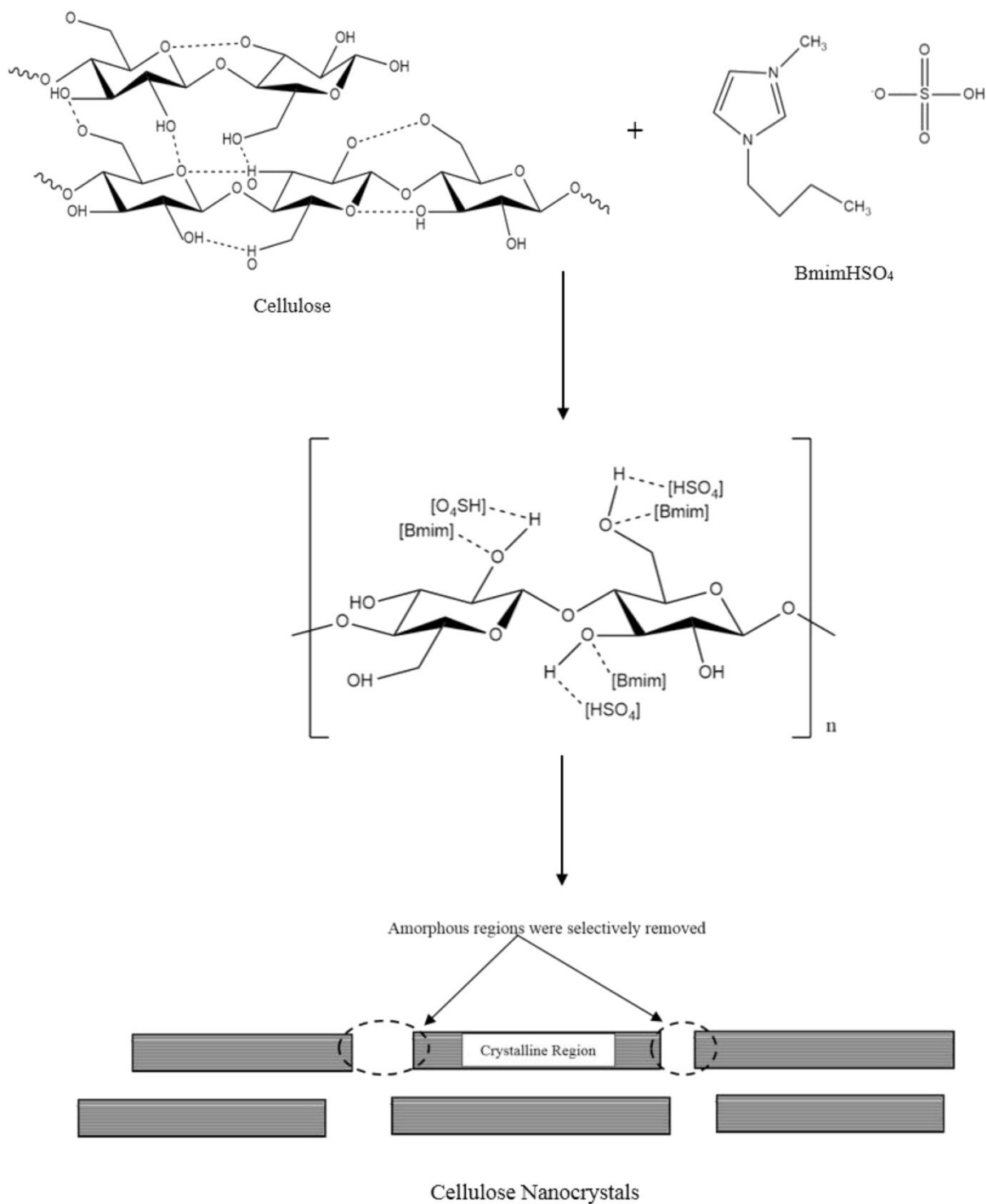


Figure S1. Schematic diagram showing reaction of single cellulose chain repeating unit with [Bmim][HSO₄]. (Tan, X. Y., Abd Hamid, S. B., & Lai, C. W. (2015). Preparation of high crystallinity cellulose nanocrystals (CNCs) by ionic liquid solvolysis. *Biomass and Bioenergy*, 81, 584-591. Copyright 2015 by Elsevier)

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