*Macedonian pharmaceutical bulletin, 69 (Suppl 1) 185 - 186 (2023)*
*Online ISSN 1857 - 8969*
*DOI: 10.33320/maced.pharm.bull.2023.69.03.091*
*Short communication*

# Functional characterization of microcrystalline cellulose obtained from the crop residues

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## Introduction

Crop residues (CR) are usually burned in the field, which causes air pollution, contributes to global warming, hinders nutrient recycling, and negatively affects soil microbes through overheating and carbon loss. There is growing interest in developing processes for isolation of value added components with application in different industries from CR as feedstock. In this study, different procedures for isolation of microcrystalline cellulose (MCC) from wheat, corn and sunflower CR were evaluated, with further testing of functional characteristics of MCC, which are relevant for tablets production.

## Materials and methods

CR of wheat, corn and sunflower collected in the wider surroundings of Belgrade and Novi Sad (Serbia) served as feedstock for MCC. A multistep procedure was used to obtain MCC, including treatment with sodium hydroxide-NaOH (1% in autoclave at 121°C and 1 bar or 2% and 4% with heating to boiling in reflux system) and bleaching with 10% hydrogen peroxide-$H_2O_2$ to remove hemicellulose and lignin, and finally acid hydrolysis with different concentrations (0.5 M-2 M) of hydrochloric acid-HCl and rebleaching with 5% $H_2O_2$. Composition analysis of the material before and after the treatment was performed according to the: Van Soest method for determination of hemicellulose (Van Soest et al., 1991), Klason method for determination of lignin and cellulose (Kirk & Obst, 1988) and gravimetric method for determination of ash content. FT-IR spectroscopy was used for identification of MCC, while functional characterization of MCC was performed by testing powder flow properties, tablet mechanical properties and compression behaviour by Gamlen D series dynamic powder compaction analyser. Analyses were performed in parallel on commercially available MCC - CMCC (CEOLUS$^{TM}$ PH101, Asahi Kasei, Japan).

## Results and discussion

All treatment procedures resulted in a significant decrease in hemicellulose and lignin content (Table 1). Treatment in autoclave was not more efficient compared to other methods. Due to the highest cellulose content, the sample of wheat CR treated with 4% NaOH and 10% $H_2O_2$ was selected for further hydrolysis to MCC. A broad absorption band between 3200 and 3400 $cm^{-1}$, and absorption bands at 2898, 1644, 1429, 1367, 1335, 1315, 1201, and 1160 $cm^{-1}$ were observed in the FT-IR spectra of all tested MCC samples (Fig. 1), which is consistent with the previously reported MCC spectrum (Wu et al., 2014). All MCC samples showed poor flow properties, with slightly better flowability of commercial sample (Table 2). High values for compression work were obtained for all tested samples, since MCC is deformed by energy demanding plastic deformation. Tensile strength values between 1.7 and 11.5 MPa confirmed good powder compactibility, with the MCC obtained from CR showing equivalent or better compactibility compared to the CMCC (Table 3). For all samples, detachment stresses below 5 MPa and ejection stresses below 3 MPa were determined (Fig. 2) as indicators of the adhesion of the material to the punch and die, meeting the usual criteria for tablet compression (Pitt et al., 2015). The

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samples obtained by hydrolysis with 1 M and 2 M HCl showed very similar adhesion properties to CMCC, while the samples hydrolysed with 0.5 M HCl showed greater adhesion at higher compression pressures.

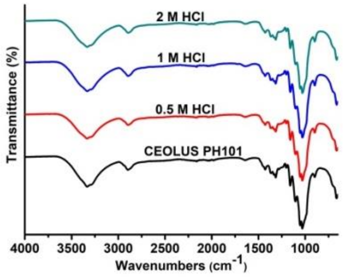

Fig 1. FT-IR spectra of commercial MCC sample and samples obtained by hydrolysis with 0.5-2 M HCl

Table 1. Composition analysis of material before and after treatment with 1-4% NaOH and 10% $H_2O_2$ (Wheat CR-W, Corn CR-C, Sunflower CR-S, cellulose-CELL, hemicellulose-HEM, lignin-LIG)

| Sample | CELL (%) | HEM (%) | LIG (%) | ASH (%) |
|---|---|---|---|---|
| W | 54.09 | 23.26 | 14.11 | 6.51 |
| C | 41.01 | 37.43 | 13.17 | 7.87 |
| S | 26.89 | 26.99 | 21.84 | 11.32 |
| W-1%-10% | 78.52 | 10.21 | 4.62 | 2.34 |
| W-2%-10% | 79.07 | 11.27 | 3.14 | 2.16 |
| W-4%-10% | 81.14 | 8.11 | 4.2 | 2.62 |
| C-1%-10% | 75.67 | 13.60 | 4.12 | 5.29 |
| C-2%-10% | 75.81 | 12.41 | 3.71 | 8.06 |
| C-4%-10% | 73.26 | 15.14 | 2.62 | 5.90 |
| S-1%-10% | 66.49 | 16.14 | 9.76 | 12.27 |
| S-2%-10% | 72.47 | 11.57 | 9.14 | 8.63 |
| S-4%-10% | 68.89 | 12.85 | 10.11 | 8.62 |

Table 2. Compressibility index (CI), Hausner ratio (HR) and flowability for MCC samples

| MCC sample | CI (%) | HR | Flowability (Ph. Eur (2.9.36) |
|---|---|---|---|
| CMCC | 28.33±0.88 | 1.395±0.017 | Poor |
| 0.5 M HCl | 33.19±0.90 | 1.497±0.020 | Very poor |
| 1 M HCl | 31.28±2.01 | 1.456±0.042 | Very poor |
| 2 M HCl | 34.16±3.03 | 1.520±0.069 | Very poor |

Table 3. Net work of compression (NWJ) and tensile strength for MCC samples

| Sample | NWC (J) | Tensile strength (MPa) |
|---|---|---|
| CMCC | 0.85±0.01-2.57±0.08 | 1.72±0.04-10.45±0.28 |
| 0.5 M HCl | 0.67±0.01-2.25±0.03 | 2.31±0.15-11.50±0.81 |
| 1 M HCl | 0.67±0.01-2.22±0.04 | 1.88±0.07-10.18±0.31 |
| 2 M HCl | 0.57±0.01-2.14±0.02 | 1.89±0.05-11.24±0.56 |

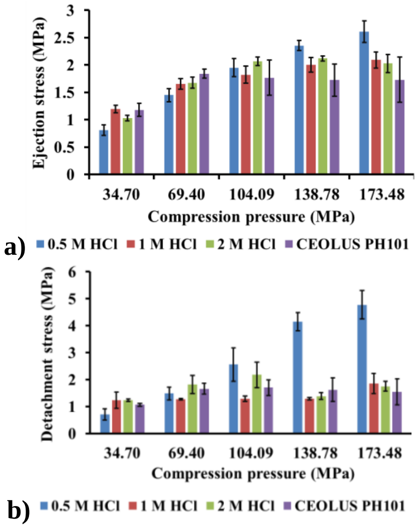

Fig. 2. a) Ejection stress and b) detachment stress at different compression pressures for MCC samples

## Conclusion

MCC with comparable functional properties to commercial MCC was obtained by chemical treatment of wheat CR. An optimal isolation method includes treatment with 4% NaOH, bleaching with 10%$H_2O_2$, hydrolysis with 1 M or 2 M HCl and further bleaching with 5% $H_2O_2$.

**Acknowledgments:** This research was supported by the Science Fund of the Republic Serbia, Grant No 7752847, Value-Added Products from Maize, Wheat and Sunflower Waste as Raw Materials for Pharmaceutical and Food Industry – PhAgroWaste.

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