

DEFIBRILLATION PHENOMENON AND PARAMETERS TO CONTROL MFC PRODUCTION

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ABSTRACT

To evaluate the defibrillation process during the production of MFC, equipment control and key parameters such as speed and number of passes in the defibrillator mill were tested. In addition, conditions inherent to samples such as pulp type and consistency of initial suspension were evaluated. It should be noted that for all morphological indices evaluated, it was noted that the speeds of 1,500 and 2,200 rpm were the ones that resulted in a more efficient defibrillation process, in contrast to the 2,900-rpm speed. Very high speeds (e.g., 2,900 rpm) do not allow for efficient shear and defibrillation times for MFC production. Factors such as viscosities and number of passes in the defibrillator have an exponential correlation, while parameters for controlling preparation of the initial fiber suspension such as consistency (2% and 4% in this study) did not show any correlation and influence on the production of MFC using this grinder equipment in laboratory. Consistencies above 6% should be investigated. The increase in the number of passes in the laboratory grinder promotes a reduction in the size of fibers in order to produce a suspension with a very homogeneous character, so the formation of films from these suspensions confirms the reduction of roughness and formation of more homogeneous surface that influences the transparency values for these films. Therefore, the production of MFC can be controlled for morphological parameters and other indirect measures such as viscosity of MFC suspensions.

Keywords: MFC, defibrillation, grinder, nanocellulose

INTRODUCTION

Many methods are used to extract cellulose nanofibrils, including mechanical, enzymatic and chemical (Abraham et

al. 2011; Tonoli et al., 2016) techniques. Fibrillation requires a large amount of effort to break hydrogen bonds between the micro/nanofibrils. Obtaining Microfibrillated cellulose (MFC) by mechanical processes, many parameters such as speed and number of passes in the grinder can promote different effects in the quality of the final microfibrillated cellulose (MFC) material.

The objective of this study was to investigate the influence of applying different conditions of mechanical defibrillation in a Masuko grinder using unbleached long fibers to produce MFC in laboratory.

METHODS

Figure 1 shows the work plan defined to evaluate the defibrillation behavior of fibers during the MFC production process. The fibers were mechanically fibrillated using a

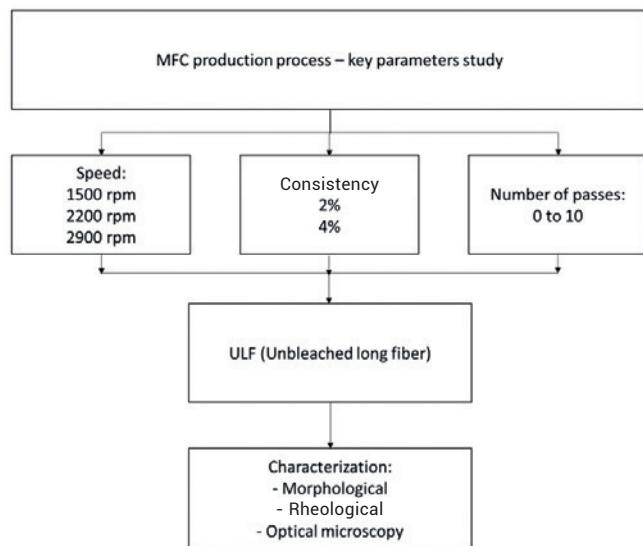


Figure 1. Work plan

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SuperMassColloider grinder (Masuko Sangyo MKCA6-2). During the fibrillation process, some processing parameters like speed, consistency, number of passes and gap were tested.

MFC production

Unbleached long fibers were immersed in distilled water at 2% and 4% cst to guarantee fiber swelling. They were then nanofibrillated by passing the pulp through an ultra-fine grinder Supermasscolloider (model MKCA6-2, disk model MKGA6-80, Masuko Sangyo Co., Ltd., Japan). Disk speeds were set at 1,500 / 2,200 and 2,900 rpm (Dias et al., 2019; Scatolino et al., 2019; Souza et al., 2019).

Morphological characterization

The morphological evaluations were measured using a fiber image analyzer (Valmet FS5, Finland). Important indices like fines, length, fiber curl and fibrillation were used in this study to evaluate the fibrillation process.

Rheological characterization

A Brookfield rotational viscometer was used, which relates viscosity to the resistance that the fluid affects the rotational movement of the spindle sensor at different speeds or shear rates and temperature. In this case, the combination of room temperature and speed at 100 rpm was used with all suspensions at 2% cst to measure viscosity.

Optical microscopy

An Olympus BX51 light microscope was used to evaluate the level of fibrillation during the deconstruction process. Fiber control and fibrillated fibers after different cycles through the mechanical defibrillator were evaluated (0 to 10 passes/cycles of fibrillation).

RESULTS AND DISCUSSION

Figure 2 shows the behavior of important morphological parameters that can be evaluated during the MFC production process, which reflect the defibrillation process.

Fines content is an indirect indicator of the reduction in particle size during MFC production. The 3rd pass at 1,500 rpm speed fines content shows 48% growth and at 2,200 rpm speed 65% comparing with the fiber fines reference at the beginning of the fibrillation. Higher speeds such as 2,900 rpm considering the same number of passes showed a lower fines index.

Pulps without mechanical treatment had a fibrillation index of around 1%. In the beginning of the fibrillation processes, at 1,500 rpm, 1.79% of the material is fibrillated, at 2,200 rpm, 2.53% of the fibers are fibrillated, this being the highest percentage of fibrillation. At 2,900 rpm, 1.02% of the material is fibrillated, this value being very close to that found for the original pulps, without mechanical treatment.

It was noted that the 1,500 and 2,200 rpm speeds were more efficient in the defibrillation process, in comparison to

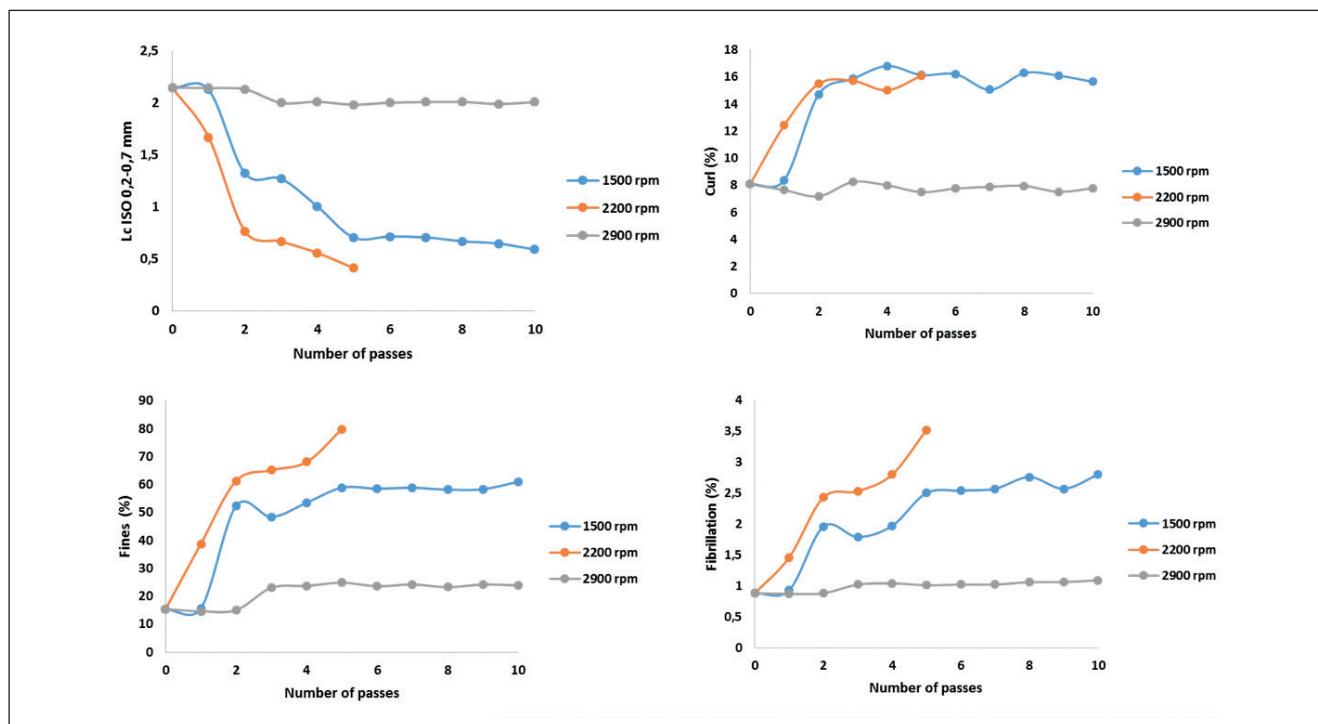


Figure 2. Morphological ULF behavior during MFC production with varying speeds (rpm) and number of passes in the defibrillator, at 4% consistency

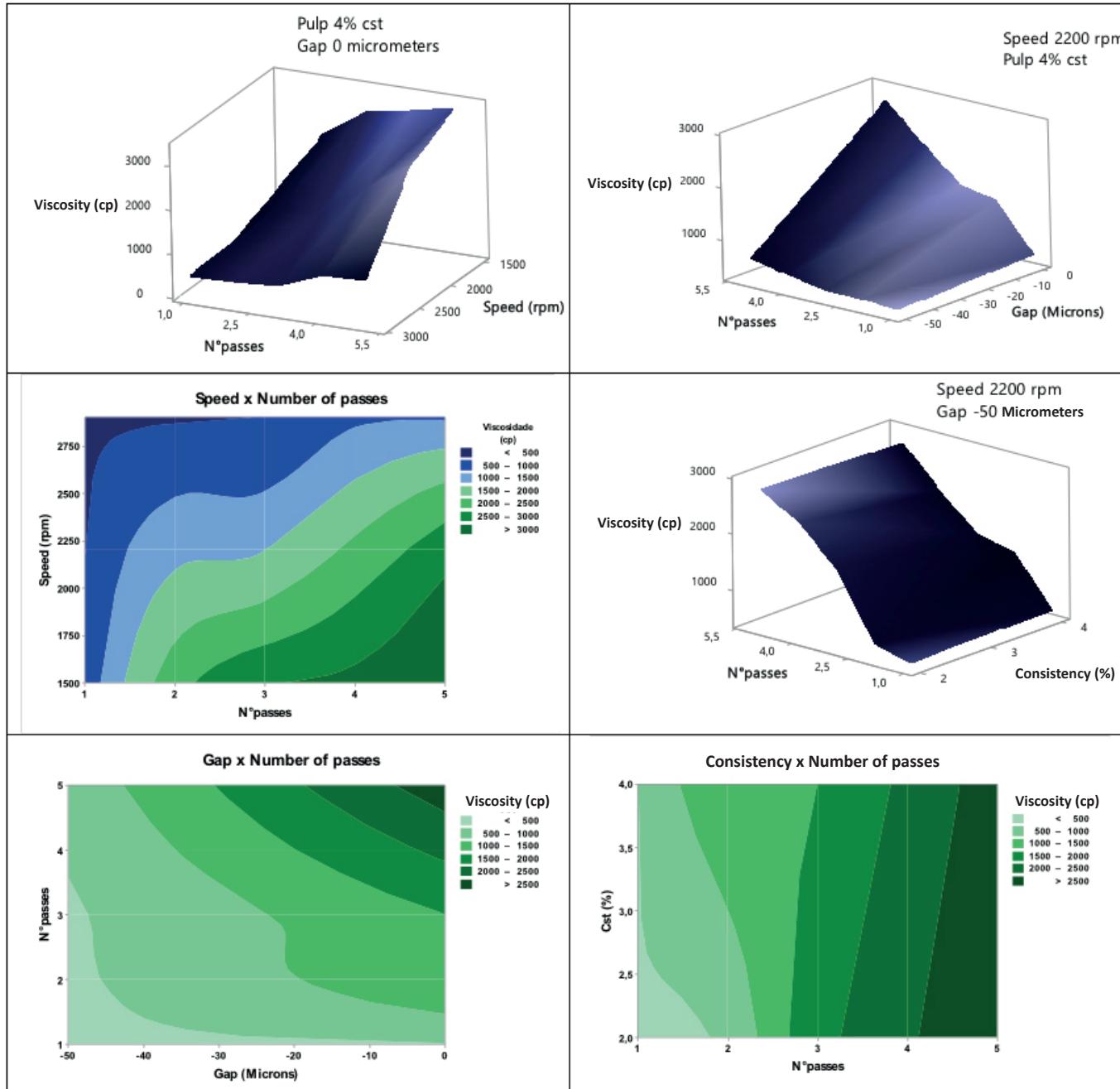


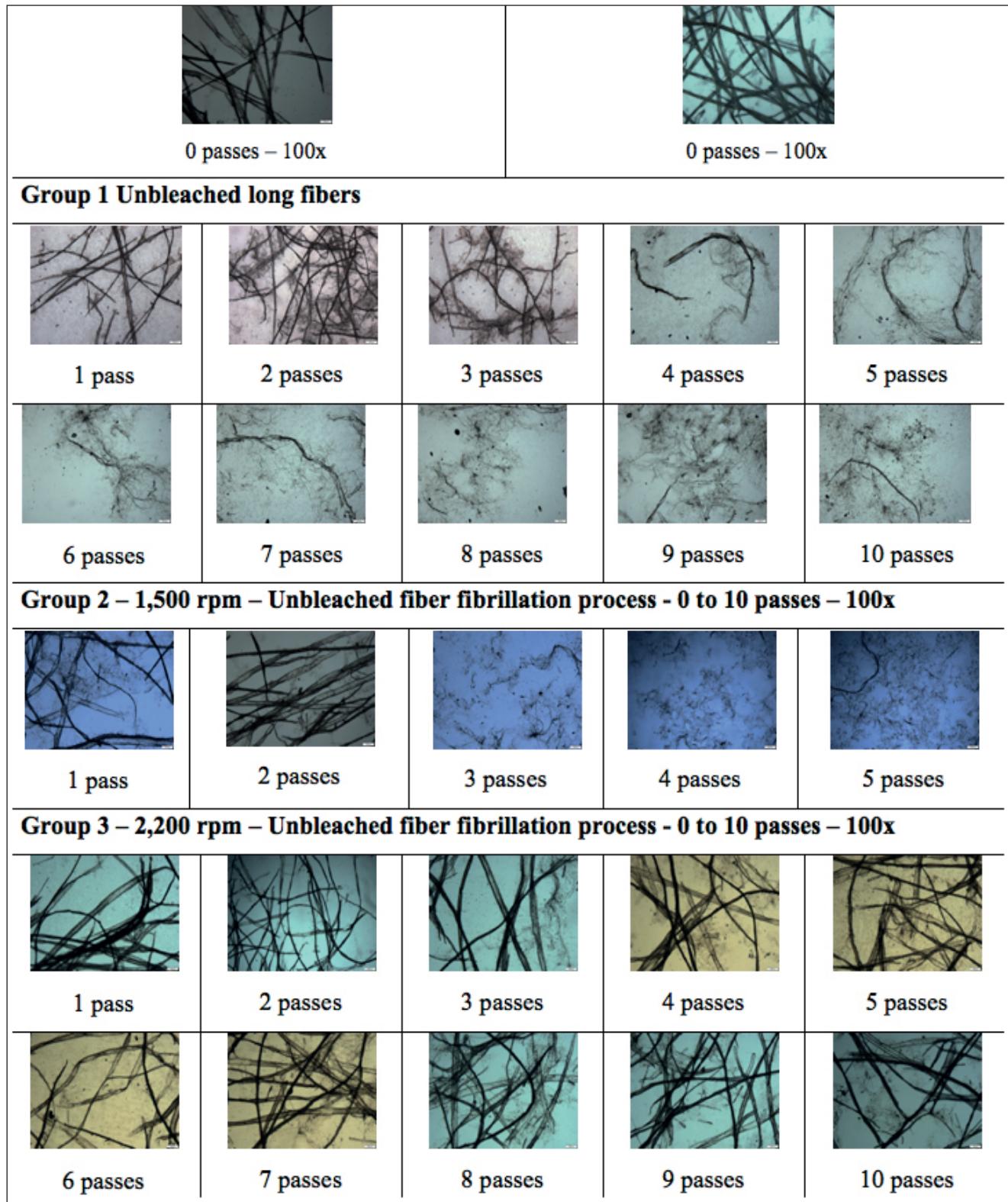
Figure 3. Interaction between operational parameters to produce microfibrillated cellulose (MFC)

the 2,900-rpm speed. Very high speeds (e.g.: 2,900 rpm) do not allow for efficient shear and defibrillation time for MFC production, as shown in Figure 4 (Group 4 – passes 1 to 10).

Figure 3 shows the interaction between process parameters in the control of MFC production, such as viscosity, number of passes in the defibrillator, rotation speed of the defibrillator stone and gap. It is observed that viscosity is directly related to the MFC production process. An increase in the number of passes in the grinder causes

an exponential increase in viscosity as the microfibrils are exposed from the cell wall of the fibers and the final suspension aspect shows higher viscosity in gel phase. Viscosity of the initial pulp is less than 500 cp, whereas after the first 5 passes this value may be greater than 2,000 cp as shown in the graph in Figure 3.

Factors such as viscosity and number of passes in the grinder or other mechanical production methods have an exponential correlation. The 2% or 4% consistency to produce MFC did not

**Figure 4.** Fibrillation process evaluation of unbleached fibers

influence the production process. Consistencies greater than 6% should be investigated, but it can be said that there is no difference between 2% and 4%, as shown in Figure 3.

The gap between rotating discs of the grinder must be considered as it has a direct effect on viscosity and defibrillation efficiency, as shown in Figure 3. Thus, the ideal is to maintain a

working distance between the non-rotating discs of point-zero microns or very close to zero microns.

CONCLUSIONS

- 2,200 rpm was the best speed for MFC production;
- Parameters such as fines content, curl, and fibrillation are easy and quick methods for MFC evaluation;
- Consistencies of 2% and 4% have no difference in the production of MFC;
- The presence of lignin does not affect or influence the MFC production process and, during the process, the

combination between temperature and residual lignin could help the fibrillation process;

- There is an exponential increase in the viscosity of the suspension with an increase in the number of passes in the defibrillator;
- Optical microscopy, morphological indices and viscosity are accurate and practical methods to evaluate MFC production.

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REFERENCES

1. Abraham, E; Deepa, B.; Pothan, L. A.; Jacob, M.; Thomas, S.; Cvellar, A.; Anandjiwala R. (2011). Extraction of nanocellulose fibrils from lignocellulosic fibres: A novel approach. *Carbohydrate Polymers* 86: 1468-1475.
2. Dias, M. C., Mendonça, M. C., & Damásio, R. A. P. (2019). Influence of hemicellulose content of Eucalyptus and Pinus fibers on the grinding process for obtaining cellulose micro / nanofibrils. *Holzforschung*, 73(11), 1035–1046.
3. Scatolino, M. V., Bufalino, L., Mendes, L. M., Guimarães Júnior, M., & Tonoli, G. H. D. (2017). Impact of nanofibrillation degree of eucalyptus and Amazonian hardwood sawdust on physical properties of cellulose nanofibril films. *Wood Science and Technology*, 51(5), 1095–1115. <https://doi.org/10.1007/s00226-017-0927-4>
4. Souza, L. O., Lessa, O. A., Dias, M. C., Tonoli, G. H. D., Rezende, D. V. B., Martins, M. A., ... Franco, M. (2019). Study of morphological properties and rheological parameters of cellulose nanofibrils of cocoa shell (*Theobroma cacao* L.). *Carbohydrate Polymers*, 214. <https://doi.org/10.1016/j.carbpol.2019.03.037>
5. Tonoli, G. H. D., Holtman, K. M., Glenn, G., Fonseca, A. S., Wood, D., Williams, T., ... Orts, W. J. (2016). Properties of cellulose micro/nanofibers obtained from eucalyptus pulp fiber treated with anaerobic digestate and high shear mixing. *Cellulose*, 23(2), 1239–1256. <https://doi.org/10.1007/s10570-016-0890-5>.



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