OPTIMIZATION OF HEMP YARN GRAFTING DEGREE FOR MEDICAL TEXTILES DURING SIMULTANEOUS WET SPINNING-GRAFTING

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The present paper studies the way the reactive derivative monochlorotriazinyl-β-cyclodextrin can be grafted on bast fibres simultaneously with their wet spinning. Process modelling involved simultaneous mechanical and chemical processing, at different parameters of wet spinning-grafting (concentration of monochlorotriazinyl-β-cyclodextrin and speed of the material in the impregnation stage). The mathematical methods of dispersion analysis and regression were applied to obtain optimum values of the process parameters. To attain physico-mechanical characteristics adequate for a 100% hemp yarn and an optimum grafting degree for subsequent inclusion operations, a soaking time of 23.7 sec and a solution concentration of 55 g/L monochlorotriazinyl-β-cyclodextrin are required for simultaneous wet spinning-grafting.

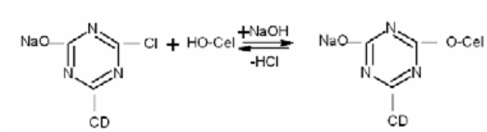
Keywords: grafting degree, optimization, monochlorotriazinyl-β-cyclodextrin, hemp yarns, wet spinning

INTRODUCTION

Due to the impressive progress in the fields of supramolecular chemistry, nanotechnology and polymer technology in recent years, a large number of drug delivery systems have been developed. Cyclodextrins, non-reductive cyclic olygosaccharides, have been successfully immobilized by physical or chemical bonds, on the surface of fabrics.[1-6] Due to their specific molecular structure, with hydrophobic inner side and hydrophilic outer side, cyclodextrins (CDs) are useful compounds for (hydrophobic) drug inclusion.[1,4,6] The inclusion or delivery of substances by the grafted CDs can be used for various medical applications.[5-7]

As part of a more extended research, the present study aims at elaborating a conceptu al model of a nanometrically finished textile material (made of hemp fibres) for medical applications, by grafting the reactive compounds and obtaining cyclodextrin inclusion compounds with bioactive properties. In the first stage, the way the reactive cyclodextrin monochlorotriazinyl-β-cyclodextrin (MCT-β-CD) can be grafted on bast fibre materials simultaneously with wet spinning was analyzed. These original elements are important for this field due to time and labour saving, allowing the extension of bast fibre application in the medical sector.

Grafting of MCT-β-CD on the cellulosic support takes place according to reaction (1). The yarns made of bast fibres have a very low breaking elongation (approximately 3%) and a high unevenness of fineness, causing a great number of breaks during spinning.



(1)

During wet spinning, by passing the roving through the immersion tank, the substances in the lamella that bond the elementary fibres become wet, jellify, behaving like a lubricant, therefore not only technical fibres, but also elementary ones would slide one beside the other during grafting. By increasing the number of fibres in the cross section, unevenness and the number of spinning breaks decrease.[8-10]

This paper presents a modelling study based on experiments designed for wet spinning-grafting of hemp fibres. It was decided to use a D-optimal design, as an effective method for obtaining maximum information with minimum experiments, and to determine which factors significantly influence the measured variables. The independent variables, soaking time of roving in the tank of the wet spinning machine (X_1) and MCT-β-CD (X_2) concentration were considered, while the grafting degree of the yarn was selected as a response (dependent) variable. A composed central rotable program for two independent variables was realized, tested and validated.

The resulting models were used for optimum process management.

EXPERIMENTAL

Materials

Monochlorotriazinyl-β-CD (MCT-β-CD or CAVATEX W7 MCT, Wacker Chemie, Germany) was grafted on the cellulosic support (high grade hemp tow, with a fineness range of 7.7-40 tex)

Methods

Due to the high length of the bundle, the high grade hemp tow, used to obtain yarns, before being introduced into the process, was cut at a length of approximately 80 cm, a value determined by the constructive characteristics of the vertical hackling machine. The tow underwent a manual precombing process, combing with a vertical hackling machine C-302-L and then recombing to straighten the ends of the tow bundle. Further on, the fibres were processed on a Mackie preparation technological flow characteristic of the spinning mills with combing technological process. To assure high quality, the sliver obtained by the sliver-making machine was evened twice with a doubling machine. Four drafting frames followed by the roving frame were used, spinning being realized in wet state on a PM-88-L5 spinning machine.

It is possible to change the time period for the roving to be soaked in the water tank of the wet spinning machine in two ways: by changing the feeding speed, or by modifying the length of the path the roving takes in the tank. The second option was selected.

To observe the influence of the selected parameters, a large variation interval was chosen, that is a length of the roving path of minimum 70 and maximum 770 mm. The modification of the roving path length through the water tank was achieved by the guide pulleys within the tank and by the amount of liquor introduced in the tank.

The physico-mechanical characteristics of the processed yarn spun under different conditions (different soaking times and different concentrations of MCT-β-CD) were measured according to the standardized methodology, being discussed in another paper.[11]

Grafting of MCT-β-CD on hemp fibers occurs in several stages. A solution of MCT (30÷80 g/L) and Na_2CO_3 (10÷100 g/L) of different concentrations was prepared, according to the experimental plan. The solution was then introduced in the tank of the spinning machine, to impregnate hemp roving for 5÷45 s. After squeezing, spinning and air-drying, the yarns were oven-cured for different periods (5÷15 min), at different temperatures (90÷150 °C), to finalize grafting. The excess reactives were removed by repeated warm and cold washings with distilled water, until reaching a pH value of 6.5-7. Finally, the samples were dried at room temperature and conditioned in a conditioning room (20 ºC, 65% humidity).

An electronic analytical balance was used. The mathematical methods of dispersion analysis and regression[12,13] were applied to find the optimum values of the process parameters. The resulting models were used for optimization by the simplex Nelder-Mead method. The maximum values for the response function and the conditions under which these can be achieved were established as necessary data for an optimum process management.

RESULTS AND DISCUSSION

The experiment aimed at establishing a multiple, simultaneous correlation between the soaking time of roving in the tank of the wet spinning machine (X_1), the concentration of MCT-β-CD (X_2) and the resultant characteristic, the grafting degree of MCT-β-CD on the hemp yarn (Y_1).

To calculate the grafting degree, the following relation was used:

$$g\% = \frac{(m_f - m_i) \times 100}{m_i} \quad (1)$$

where m_i and m_f are the weights of the conditioned samples, before and after grafting.[6,14] The regression equation obtained after testing the correctness of the cofficients and the adequacy of the model is the following:

$$Y_1 = 26.9899 + 1.43573\, X_1 + 1.7656\, X_2 - 4.97715\, X_1^2 - 1.69866\, X_2^2 + 2.0525\, X_1 X_2 \qquad (2)$$

For the model expressed by equation (2), the linear and square components were successively analyzed. For the linear component of equation (2), one can conclude that, between the two parameters observed, the concentration of MCT-β-CD (X_2) has a greater influence on the resultant characteristic, because the coefficient of independent variable X_2 is 23% higher than that of variable X_1.

The signs of the coefficients of the linear component are positive for both parameters; taking into account that the resultant is the grafting degree, we have obviously aimed at maximizing this function, therefore the behaviour of the model for the simultaneous variation towards the limits of the experimental region of the two considered parameters, X_1 and X_2, was observed.

The value of the multiple correlation coefficient, R = 0.98742, proves the influence of the two parameters X_1 (soaking time of roving), X_2 (concentration of MCT-β-CD) and Y_1 (grafting degree), the correlation being important (R is very close to 1).

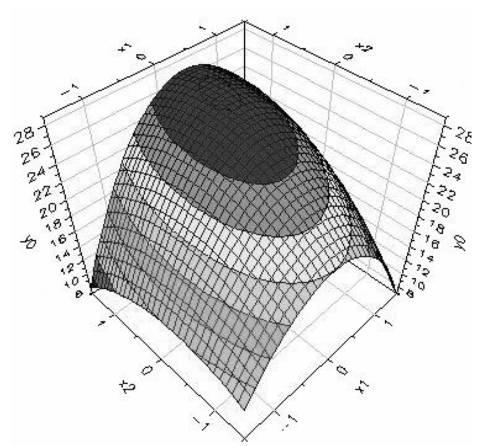


Figure 1: The 3-D response surface for grafting degree

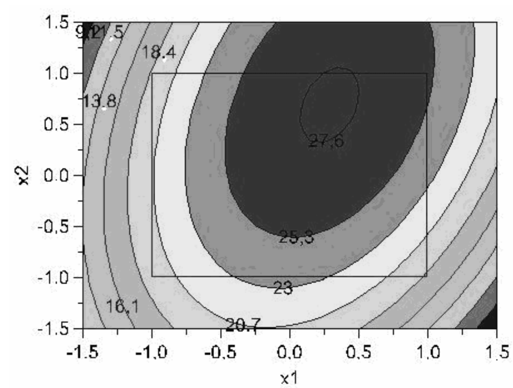


Figure 2: Constant contour lines of resultant variable, grafting degree

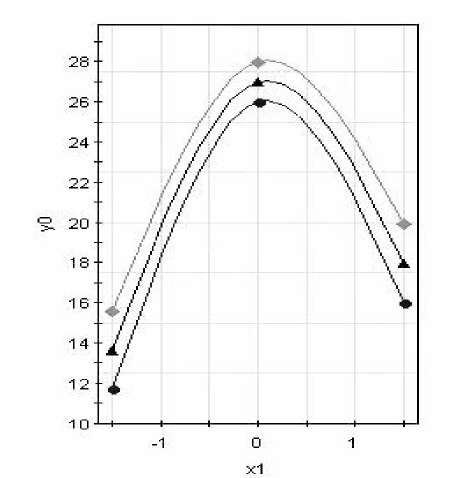


Figure 3: Modification of grafting degree (independent variable Y_1) depending on soaking time of roving (independent variable X_1), for $X_2 = X_{2c}$

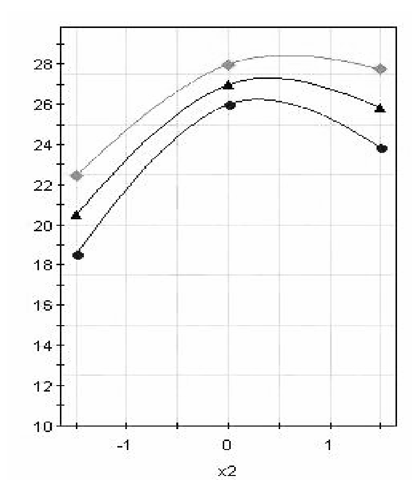


Figure 4: Modification of grafting degree (independent variable Y_1) depending on concentration of MCT-β-CD (X_2), for $X_1 = X_{1c}$

Regarding the square component of the equation, it is worth noting that the presence of the square terms for both X_1 and X_2 confirms the existence of a well-outlined surface (Figs. 1 and 2), a rotation ellipsoid with a maximum point, corresponding to the previous claim of maximization; in this case, a difference may be observed between the absolute values of the square term coefficients, X_1^2 having a coefficient 3 times higher than the one corresponding to X_2^2; the negative sign of these coefficients proves that the square term influences the objective function when decreasing, regardless of the way the considered parameters change.

Analyzing the regression equation (2), it is noticeable that the coefficient of interaction corresponding to the term X_1X_2 is present, meaning that the effect of the simultaneous variation of the two parameters is cumulative.

Figure 3 presents graphically the modification of dependent variable Y_1 as a function of the modification of independent variable X_1 for $X_2 = X_{2c}$ (values in the centre of the domain), while Figure 4 plots the variation of Y_1 as a function of independent variable X_2 for $X_1 = X_{1c}$ (values in the centre of the domain).

A maximum grafting degree of 27.80% was obtained for a soaking time of 27.79 s and a 67.27 g/L concentration of MCT-β-CD.

CONCLUSIONS

The experiments performed to observe the influence of soaking time and concentration of MCT-β-CD on the grafting degree have proved that a concentration of 67.27 g/L MCT-β-CD and values of the soaking time close to the centre of the experimental program are required.

To obtain an optimum grafting degree for subsequent inclusion operations, it is necessary to establish a soaking time of 23.7 s and a solution concentration of 55 g/L monochlorotriazinyl-β-cyclodextrin for the simultaneous wet spinning-grafting process.

The reactive derivative monochlorotriazinyl-β-cyclodextrin (MCT-β-CD) can be grafted on bast fibre materials simultaneously with wet spinning, for obtaining a nanometrically finished hemp material for medical applications (after grafting, bioactive compounds will be included into the nanocavities of the reactive product). Therefore, the application fields for bast fibres may be extended to the medical sector for obtaining ecological products with new functional properties.

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REFERENCES

[1] A.-M. Grigoriu, *PhD thesis*, “Gheorghe Asachi” Technical University of Iaşi, 2009, p. 23.

[2] N. Kistamah, C. M. Carr and S. Rosunee, *J. Mater. Sci.*, **41**, 379 (2006).

[3] A.-M. Grigoriu, C. Luca and A. Grigoriu, *Cellulose Chem. Technol.*, **42**, 103 (2008)..

[4] A. Grigoriu, A.-M. Grigoriu and C. Luca, *14th International Cyclodextrins Symposium*, *Final Program & Abstracts*, May 8-11, 2008, Kyoto, Japan, (poster P2-11), p. 133.

[5] M. R. Ten Breteler, V. A. Nierstrasz and M. M. C. G. Warmoeskerken, *AUTEX Res. J.*, **2**, 175 (2002).

[6] C. Luca and A.-M. Grigoriu, *Cellulose Chem. Technol.*, **41**, 13 (2007).

[7] J. Szejtli, *Pure Appl. Chem.*, **76**, 1824 (2004).

[8] C. Cuzic-Zvonaru, *PhD thesis*, “Gheorghe Asachi” Technical University of Iaşi, 1992, p. 35.

[9] C. Racu, C. Cuzic-Zvonaru and C. Preda, *Ind. Text.*, **50**, 149 (1999).

[10] C. Racu, *Bull. Inst. Pol. Iaşi*, *Textiles – Leathering Section*, LIV (LVIII), **3-4**, 9 (2008).

[11] A. Grigoriu, C. Racu, R. Diaconescu and A.-M. Grigoriu, *Ind. Text.*, (2010) (in press).

[12] R. Mihail, in “Introducere în strategia experimentării cu aplicații din tehnologia chimică” (in Romanian), Scientific and Encyclopaedic Publishing House, Bucureşti, Romania, 1976, pp. 99.

[13] D. Taloi, in “Optimizarea proceselor metalurgice” (in Romanian), Didactic and Pedagogical Publishing House, București, Romania, 1983, p. 76.

[14] C. Luca, A.-M. Grigoriu, R. Diaconescu, V. Slabu and A. Grigoriu, *Bull. Inst. Pol. Iasi*, *Textiles – Leathering Section*, LIII (LVII), **5**, 473 (2007).