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Investigations of Lignocellulosic Materials from Rape for the Purpose of Producing Composites with Thermoplastic Polymers

Abstract
So far we know of no complex investigations defining lignocellulosic materials obtained from the stem of rape for the purpose of application in composites with thermoplastic polymers. This study is concerned with investigating the morphology of the rape stem, and the cellulose content in the wooden parts of the rape stem. Investigations with a microscope of the structure of the rape stems and the thickness of the wooden layers for various winter seed oil varieties were carried out. On the basis of the analytical results, it has been found that the average cellulose content in the wooden parts of the winter rape stem ranges from 35% to 40%. The amount of the cellulose in rape in comparison with the amount of cellulose in various species of wood is comparable. The structural investigations confirmed the presence of cellulose I. The X-ray patterns in the internal and external areas of the wooden parts of the stem are similar.

Key words: *rape, composites, polypropylene, structure, PLM, SEM, WAXS.*

Introduction

On the basis of preliminary investigations, we concluded that the lignocellulosic material from rape is suitable for developing composites with isotactic polypropylene [3]. During investigations into new kinds of composites, it is important to characterise the components used. World crops of rape are estimated at over 36 million tons, which means that this fibre may become a significant source of lignocellulosic materials.

Since cellulose plays an important role as a skeletal component in wood fibres, and has an impact on their mechanical properties [1], an analysis was carried out to determine the content of cellulose in rape stems.

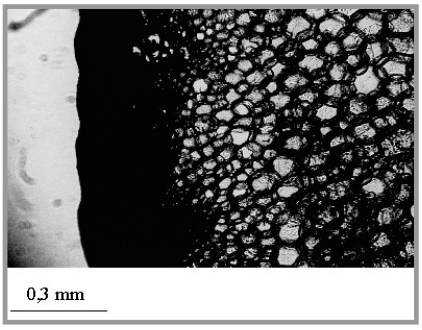

Figure 1. *Cross-section of the rape stem (Lisek varieties).*

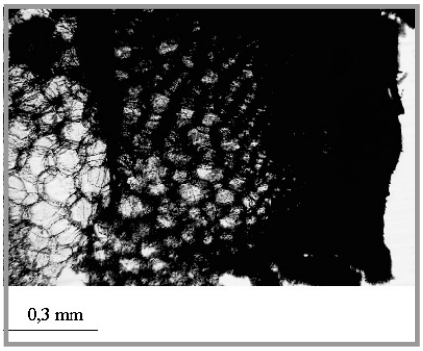

Figure 2. *Cross-section of the rape stem (Kaszub varieties).*

Up to now, only one study [2], published in the 1960s, has described the morphology of the rape stem. Therefore, it would be advisable to characterise this structure in the context of using the wooden parts of the rape stem as a source of lignocellulosic material to be used in producing composites with thermoplastic polymers.

Our previous investigations on composite rape stem/isotactic polypropylene confirm the good mechanical properties of this new composite material [3].

This study is concerned with investigating the morphology of the rape stem and the content of cellulose in the wooden parts of the rape stem.

Experimental

In order to characterise the morphology of the rape stem and the cellulose content in the wooden parts of the rape stem, we analysed three winter oilseed rape varieties: the German Lisek, the French Casek and the Polish Kaszub. The Polish rape used came from the Plant Breeding and Acclimatisation Institute (IHAR) fields in Poznań and grew in 2003. For our studies we used dried rape stem that had been stored for 6 months.

The percentage content of the cellulose from the wooden parts of the rape stems was analysed by means of the analytical methods described earlier [2,4].

The anatomical structure of the rape stems was examined by means of optical mi-

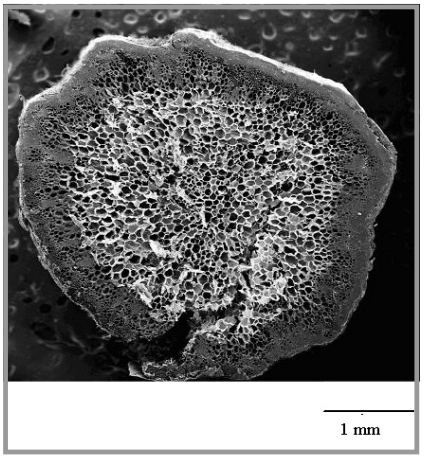

Figure 3. *Cross-section of the rape stem (Casek varieties).*

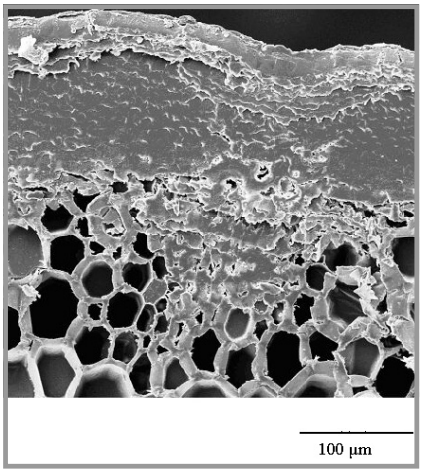

Figure 4. *Cross-section of the rape stem (Kaszub varieties).*

croscopy (a Biolar PI polarising-interferencing microscope connected to a Minolta 300X camera) and electron microscopy (Philips SEM 515) methods, using a voltage of 15 kV.

The structural investigations of rape stems were carried out by means of the wide-angle X-ray scattering method (WAXS), using CuKα radiation. The samples were prepared in the following way: the stem was cut lengthwise, the soft tissue (parenchyma) was then removed, and the flat surfaces obtained in this way were examined on both sides.

Results

The microscope investigations characterised the structure of the rape stems and thickness of the wooden layers in each of the mentioned varieties. The images (Figures 1-4) show the stems after they matured that is this sort of sample which is used as raw material for production of the composites.

As we can see from optical microscope images, the inside of the stem is parenchyma. As regards the outside part of the stem, this represents the wooden part of the stem. This is also shown on the images obtained by the electronic microscope method (Figures 3,4). Figure 1 shows a microscope picture of the Lisek varieties, and Figure 2 shows the Kaszub varieties. A cross-section of the whole rape stem is shown in Figure 3 (for the Casek varieties). All the samples display a similar stem structure.

On the basis of the photographs taken of the samples, we can establish that the thickness of the wooden layers falls within a wide range of 0.2 to 0.7 mm (Figures 1-4). Before the samples were tested for cellulose content, we decided to establish the percentage of the wooden part of the stem. This showed that the amount of the wooden substance in the rape stem exceeds 80% for all varieties (except for parenchyma).

We used a method for estimating the cellulose in rape stems which was based on separating the cellulose of other compounds (mainly lignin) in an acid mixture of acethyloacetone and 1,4-dioxane [1,4]. On the basis of the analytical results, it has been found that the average cellulose content in wooden parts of winter rape stem are as follows: for Lisek: 35.1%; for Kaszub 36.8% and for Casek 37.1% (Table 1) [5].

Table 1. *Cellulose content in the wooden parts of winter rape stem (varieties: Lisek, Kaszub, Casek); m – mass of the crucible with precipitate after drying [g], m1 – mass of the crucible without precipitate after drying [g], m2 – mass of the sample [g], Δm, Δm_1, Δm_2 – error in weighing (0.0001 [g]), C – amount of the cellulose [%], ΔC – error of the measurement.*

AMOUNT OF THE CELLULOSE									
Variety	Lisek			Kaszub			Casek		
Test	1	2	3	1	2	3	1	2	3
m, g Δm, g	49.4029 0.0001	38.0568	37.9597	19.2902	18.2177	18.5215	18.3625	18.1965	19.2775
M.1, g Δm1, g	49.0543 0.0001	37.7091	37.6095	18.9438	17.8727	18.1745	18.0205	17.8562	18.9367
M.2, g Δm2, g	1.0250 0,0001	1.0220	1.0281	1.0252	1.0254	1.0286	1.0259	1.0227	1.0218
C, % ΔC, %	35.114 0.025	35.139 0.025	35.170 0.025	36.839 0.027	36.808 0.027	36.868 0.027	37.057 0.027	37.118 0.027	37.078 0.027

Table 2. *The amount of the cellulose in various species of the wood [1].*

Species of the wood	Amount of the cellulose [%]
pine	41.9 – 54.2
spruce	38.1 – 58.0
oak	46.6
white willow	37.2 – 50.0

Table 2 shows the average content of cellulose in various species of woods: pine, oak, spruce and white willow [1].

As is known, wood is a valuable lignocellulosic material used for manufacturing composites [6,7,8]. As shown in the comparison in both tables, the amount of the cellulose in wood is comparable to that in the rape stem.

As shown in Figures 1-4, the structural element which is the source of lignocellulosic materialsis the wooden part of the stem.

The lignification process of the stem's inside layer progresses along with the growth of the plant. The size of the layer increases because of the lignification of the soft tissue of the core. For that reason, the structure of both the interior and exterior of the stem was checked.

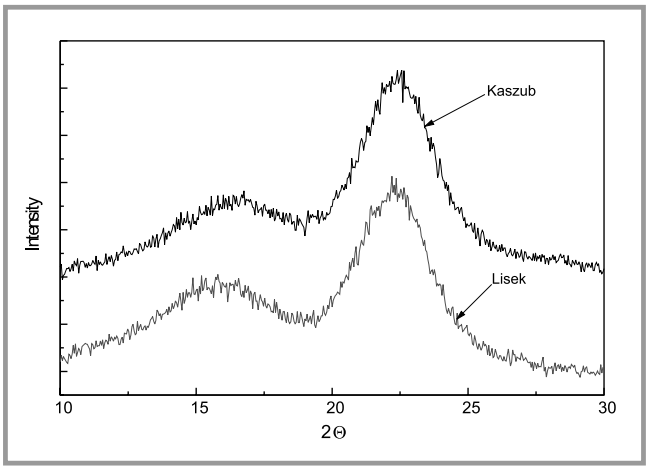

Figure5. *X-ray patterns of outside layers of rape stem (Lisek and Kaszub varieties).*

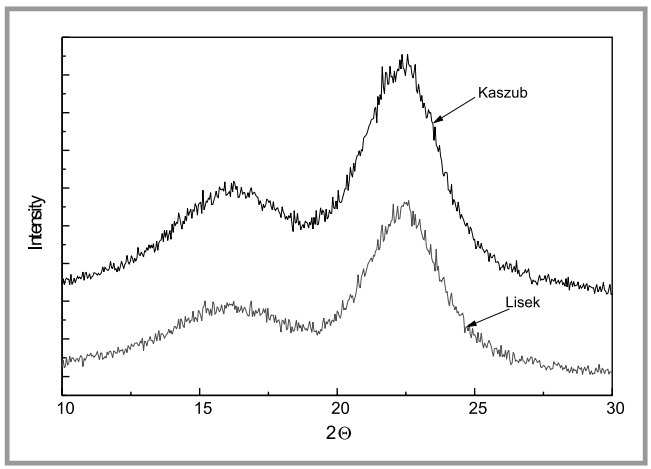

Figure 6. *X-ray patterns of inside layers of rape stem (Lisek and Kaszub varieties).*

Figure 5 shows the X-ray patterns of the outside part of the Lisek (lower graph) and Kaszub varieties (upper graph). On Figure 6 the X-ray patterns from the other side are presented.

As we could expect, the X-ray picture represents a typical image of cellulose I [8,9]. As shown in the comparison of both X-ray patterns, there were no significant differences between the varieties. The comparison of X-ray patterns stems from both the inside and outside shows that the crystalline structures are similar.

Conclusions

- The thickness of the wooden layers in rape stems determined by microscopic methods is in the range of 0.2 – 0.7 mm.
- The content of the cellulose in rape is comparable with the content of the cellulose in other lignocellulosic materials used for manufacturing composites.
- The X-ray patterns of wooden rape stems from both the inside and outside are similar, and confirmed the presence of cellulose I.

Acknowledgments

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