

# Isolation of Suberinic Acids from Extracted Outer Birch Bark Depending on the Application Purposes

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## Abstract

The research applies to the woodworking industry and the utilisation of bark for obtaining of saturated higher fatty acids, containing reactive hydroxyl and epoxy groups, which are promising raw materials for the synthesis of high added value derivatives for cosmetics, emulsion production, the polymer industry, etc. Therefore, the application of the results of our investigation has synergy with material science. The aim of the investigation was to increase the utilization efficiency of birch bark and to inform the specialists of the forestry and wood industry about the wide scope of high added value products, which could be produced from this valuable raw material. The fractionation of suberinic acids by sedimentation in certain ranges of the alkaline-alcohol solution pH 4.7-6.0 made it possible to obtain suberinic acids with the content of 9,10-epoxy-18-hydroxy octadecanoic acids up to 58% oven dry suberinic acids fraction.

The mixture of suberinic acids obtained by alkaline-water depolymerisation acts as an excellent binding agent to produce medium and high density formaldehyde free particle boards with low thickness swelling and high bending strength. The biobased rigid polyurethane foams obtained from suberinic acids polyols also demonstrate good mechanical properties.

**Key words:** Birch bark, depolymerisation, suberinic acids, particle boards, polyurethane foams

## Introduction

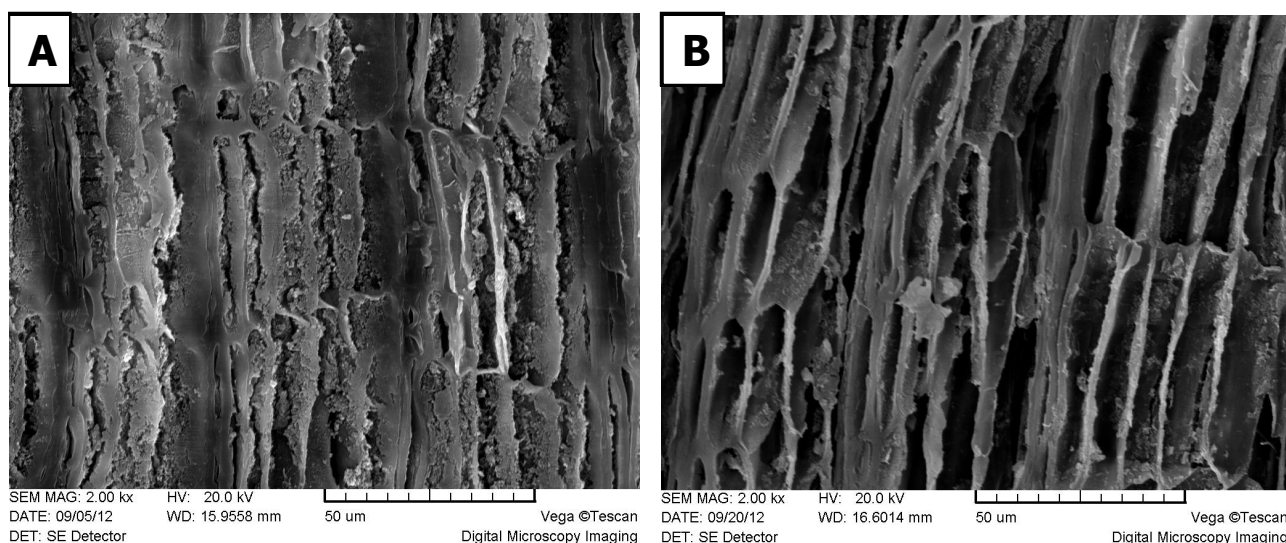
In Latvia's plywood factories, along with veneer shorts, birch bark is burned up in boiler-houses for steam and heat production. Silver birch (*Betula pendula* Roth.) outer bark comprises up to 3.4% of the veneer blocks' mass as reported by Jensen (1948). In the factories for producing 1 m<sup>3</sup> of plywood, on the average, 2.7 m<sup>3</sup> of veneer blocks are necessary, of which 28.6 kg is oven dry (o.d.) outer birch bark. If the plywood export was 246.1 thousand m<sup>3</sup> in 2012, the total annual potential of such a valuable readily accessible raw material, concentrated in one place, can reach 7,000 t in Latvia. Besides triterpenes, about 40-50% of the outer bark mass is polyester – suberin, formed by higher saturated oxyfatty acids with a unique and rarely naturally found location of functional groups in the molecule (Pinto et al. 2009). Simultaneously with producing 1 m<sup>3</sup> of plywood, it is possible to obtain also about 12.8 kg of suberinic acids from outer birch bark.

It is known from the literature that, in the plant material, suberin is aromatic-aliphatic cross-linked polyester, in which suberinic acids are cross-linked with aromatic polyphenols (Gandini et al. 2006, Ferreira et al. 2013). It is widespread in the plant kingdom, where it plays a key role as a hydrophobic barrier that restricts the paths available for the movement of water and sol-

utes in roots and which is an important in pathogen resistance (Pollard et al. 2008). It is found mainly in the cell walls of normal and wounded external tissues of aerial and/or subterranean parts of many plants, mainly in the outer bark of higher plants and in tuber periderms (Pinto et al. 2009). Figure 1 demonstrates that there are two types of cork cells in outer birch bark: narrow radial dimension cells, dark in colour due to the polyphenols in their lumena and very broad radial dimension cells, appearing light in colour due to the deposition of white coloured triterpenes.

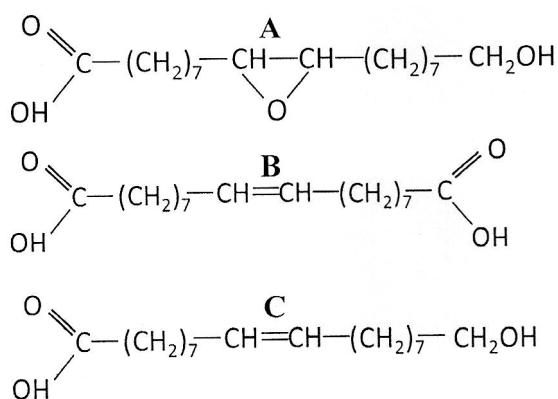
After extraction of triterpenes, mostly betulin and lupeol, which comprise about 30-35% of the o.d. outer birch bark and about 40-50% of the suberin-containing cork cell residue, are left over (Figure 1). The rest 15-20% of outer birch bark components are sugars, polyphenols, tannins, cellulose and lignin (Kisilitsyn 1994, Pinto et al. 2009).

To obtain suberinic acids in the free form, hydrolytic depolymerisation of suberin in a basic medium should be carried out, obtaining the corresponding salts. After acidifying, free acids can be isolated. After depolymerisation, the monomer composition of suberin mostly consists of fatty acids,  $\omega$ -hydroxyfatty acids,  $\alpha$ -,  $\omega$ -dicarboxylic acids, aliphatic alcohols and aromatic acids depending on the plant material.  $\omega$ -hydroxyfatty acids are generally the most abundant



**Figure 1.** Cross section of outer birch bark before (A) and after (B) extraction by SEM

group of suberin monomers in the outer birch bark of *Betula pendula* (Gandini et al. 2006), and the main compounds in the suberinic acid mixture are epoxy and hydroxyl groups containing derivatives. If more drastic alkaline depolymerisation conditions (methanolysis) are used, alkanedioic and hydroxyalkanoic acids (Pinto et al. 2009) are formed (Figure 2).



**Figure 2.** Main saturated fatty acids derivatives obtained by birch bark alkaline depolymerisation: epoxy derivatives (A), alkanedioic acids (B), hydroxyalkanoic acids (C) (Pinto et al. 2009)

18-hydroxy-9,10-epoxy-octadecanoic acid (HEPA), which comprises 11.9-19.3% of the total suberinic acids' mass, can be obtained by alkaline-alcohol depolymerisation of outer birch bark (Ekman 1983, Ekman and Eckerman 1985, Holloway 1983). HEPA is an interesting raw material for organic synthesis, because it is encountered seldom in nature, and it is very difficult to obtain it synthetically. These and related monomers are useful to design and synthesise unique

functional polyesters, which could be also biodegradable (Yang et al. 2010).

In the Soviet Union patent SU382657 (Федорищев и Калайков 1973), a method for chemical processing of outer birch bark is shown, with simultaneous obtaining of betulin and suberinic acid salts, according to which outer birch bark is treated with an alkali-alcohol water solution, filtered and treated with direct steam for distillation of alcohol. As a result, a mixture is obtained, which contains betulin, sodium suberinate and lignin, which are then separated from each other. This method is laborious and does not allow obtaining high-quality betulin and sodium suberinate due to the complicated isolation from the obtained mixture. For this reason, the isolation of betulin and suberinic acid salts is carried out gradually, firstly isolating betulin, and then suberinic acid salts.

Basic depolymerisation is commonly carried out in an organic polar solvents' medium, most often using aliphatic alcohols such as methanol, ethanol, isopropanol, etc. (Cordeiro et al. 1997, Gandini et al. 2006, Pinto et al. 2009, Krasutsky et al. 2004). The main reason is that, in the organic solvents' medium, the suberin depolymerisation is more complete, and more individual acid salts are formed, acidifying which till pH 5-6, a great amount of suberinic acids, containing epoxy acids (mainly 9,10-epoxy-18-hydroxy octadecanoic acid), are obtained.

A method of similar trend for hydrolytic depolymerisation in basic water medium is recommended by the World Intellectual Property Organization (WIPO) in the patent application WO/2010/093220 (Iversen et al. 2010). The advantage of the method is the water medium, but the non-indicated yield makes to doubt whether it would be high.

There are several ways to use suberinic acids for obtaining of high added value products. Some publications deal with the potential of the suberinic acids in the polyurethane foam synthesis from suberinic acids obtained from the cork of *Quercus suber* (Cordeiro et al. 1997, Cordeiro et al. 1999). Also promising could be the use of isolated suberinic acids as a binder for the production of wood board materials to improve the water stability, wherein the effect of the pressing temperature on the physico-mechanical characteristics of particle board material samples was tested (Sudakova et al. 2008).

During the present investigation, two main goals were advanced. Firstly, we compared and evaluated from the technological point of view two methods for isolation of suberinic acids – depolymerisation in alkaline-water and alkaline-alcohol media. Secondly, we tried to find another practical solution to the use of suberinic acids depending on the isolation method, and to make this isolation procedure as simple as possible and still obtain high added value products from the raw materials, which are currently burned in boiler houses to produce energy and technological steam. Therefore, the isolated mixture of suberinic acids was tested in the biobased polyurethane foam synthesis and as a binder in particle board preparation. The obtained products were characterised and their validity was evaluated.

## Materials and Methods

### Feedstock

Freshly isolated birch bark of *Betula pendula* and *Betula pubescens*, left over at a plywood factory, was chosen as a representative of industrial waste. Birch bark samples, dried at room temperature (moisture content 2-4 wt.%), were milled in a cutting mill SM 100 (Retsch GmbH & Co) to pass the sieve with holes of diameter 2.00 mm. Milled birch bark was soaked by mixing from time to time in deionised water for 24 h. Outer birch bark, floating on the surface, was collected and dried at room temperature and in a drying cabinet at 50°C to a moisture content of 2-4 wt.%. Extraction was carried out in a Soxhlet apparatus by 95% ethanol (aqueous solution, v/v) for 11 h to be sure that all the extractives are removed.

### Calorific value and elemental analysis

Calorific value was determined on a “Parr” Oxygen Bomb Calorimeter, in which about 1.0 g of an o. d. birch bark sample was completely combusted under a pressurised (3000 kPa) oxygen atmosphere. The rise in temperature of the cylinder allows the calculation of the calorific value when the exact weight of the sample is known.

Birch bark samples were ground to fine sawdust and passed through different sieves to obtain a powder having a granulometry of 0.2 to 0.5 mm. Elemental analysis was performed on an Elementar Analysensysteme GmbH – vario MACRO CHNS Element Analyser – an analyser for the determination of C, H, N and S in solid and liquid samples, using a thermal conductivity detector.

### Depolymerisation

#### *Depolymerisation in alkaline-alcohol solution*

According to the Krasutsky et al. 2004, the extracted outer birch bark was saponified with sodium hydroxide isopropanol solution for 1 h (outer bark - isopropanol - sodium hydroxide mass ratio 1:9:0.33), the solution was discharged, and the outer birch bark was boiled with isopropanol for another 20 min. The solutions were evaporated till a dry state, diluted with water, and the insoluble 9,10-epoxy-18-hydroxy-octadecanoic acid salts were separated. By acidifying the filtrate with 6% HCl till pH 6.0, 4.7 and 2.1, after washing, centrifugation and drying in vacuum, three suberinic acid fractions were obtained.

#### *Depolymerisation in alkaline-water solution*

For hydrolytic depolymerisation in basic water medium, the method described by Iversen et al. (2010) was chosen. Improved contacting of suberin with alkali was reached, using outer birch bark vacuum treatment prior to the alkali solution's pouring to the outer birch bark. Boiling under reflux for 1 h and filtering were carried out. The only difference was that the salts of suberinic acids in water solution without evaporation were acidified with nitric acid till pH 2, then left in the cold overnight, separated by centrifugation and dried in a vacuum.

Three parallel depolymerisation experiments were made for each method and two parallel aliquots of each sample were analysed by triple procedures. The results represented the average values of each experimental method, which did not exceed a 5% variation for both the indices between the parallel aliquots and those between the parallel aliquots of the same sample.

### Synthesis of polyols and foam preparation

Polyols from suberinic acids were synthesised by esterification with triethanolamine in an inert atmosphere at a temperature of 170-175°C. The synthesis was accomplished, when the acid value was below 5 mg KOH/g. Polyurethane foams were prepared from the average sample of suberinic acid esters with the OH number 440 mg KOH/g and 432 mg KOH/g. The polyol system was prepared by mixing of suberinic acid esters with the polyether polyol “Lupranol” with the

OH number 400 mg KOH/g and the additives – flame retardant, blowing agent, catalyst and stabiliser (Table 1). Polyurethane samples were obtained by mechanical mixing of appropriate amounts of polyisocyanate (isocyanate index – 120) and the polyol system for 10-15 s. The unreacted mixture was poured into a plastic mould (20 × 30 × 10 cm) for free foaming. The polymerisation reaction occurred at room temperature for all obtained samples and was completed in about 3-5 min; the samples were conditioned before the tests for 24 h. The content of different components in the composites was calculated on the basis of the initial weights.

**Table 1.** Polyurethane foam components based on suberinic polyols

Component, g	Polyol OH value 432 mg KOH/g	Polyol OH value 440 mg KOH/g
Polyol Suberin + triethanolamine	70	70
Polyether polyol "Luprano" 3300 (OH value 400 mg KOH/g)	30	30
Flame retardant – tris (chloropropyl) phosphate	35	25
Water	2.5	2.0
Blowing agent "Solkane 365/227"	30	25
Foam stabiliser "L6915"	2.0	2.0
Catalyst "Policat 5"	1.0	1.0
Polyisocyanate (NCO group content 31.5%)	153	146
Isocyanate index	120	120

#### *Methods for characterising polyurethane foams*

The physical and mechanical properties of the foams were measured in accordance with the following standards: foam density – ISO 845:2009; comprehensive strength – ISO 844:2007; closed cell content – ISO 4590. The comprehensive strength of polyurethane samples was tested parallel and perpendicular to foam rise. Mechanical testing of polyurethane samples was performed on testing machines Zwick/Roell 500N and Zwick/Roell Z100 (Ulm, Germany).

#### *Particle board's preparation*

For the production of wood plate materials, grey alder (*Alnus incana*) sawdust with a fraction of 0.125-2.00 mm and a moisture content of 9% was used. The mixture of suberinic acids (moisture content 80%) prepared by alkaline-water hydrolytic depolymerisation was mixed with a wood filler at the ratio of 30:70. Then the produced raw material was dried in a drying chamber up to a moisture content of 3% and then pressed

on a laboratory press LAP-40 (Gotfried Joos Maschinefabrik GmbH & Co) for 10 min under 10 MPa at temperatures of 140-180°C. The quality of the obtained particle boards was characterised by density (EN 323), thickness (EN 324-1), thickness swelling (EN 317) and bending strength (EN 310), which was determined on a testing machine Zwick/Roell 500N (Ulm, Germany).

#### *Determination of acid value*

0.12-0.15 g of the suberinic acid sample was dissolved in 25 mL dimethylsulfoxide and then potentiometrically titrated with 0.1 M KOH aqueous solution. The result was expressed as mg KOH/g suberinic acid.

#### *Determination of epoxy groups*

0.12-0.15 g of the suberinic acid sample was treated with 5 mL of non-aqueous HCl solution in dimethylsulfoxide for 20 h. Then the excess of HCl was titrated with 0.1 M KOH aqueous solution. The blank sample, i.e. 5 mL of non-aqueous HCl solution in dimethylsulfoxide was also titrated. The amount of epoxy groups was equivalent to the loss of hydrochloric acid estimated in the titration of the analysis and blank samples. The amount of 9,10-epoxy-18-hydroxyoctadecanoic acid was calculated using a molecular weight of 313 g/mol.

## Results

The elemental analysis (N – 0.5%, C – 70.4%, H – 6.3% and O – calculated by the difference 22.8%), ash content 0.41% and calorific value (31-32 MJ/kg) of outer birch bark show that it is an excellent fuel. Since our intention was obtaining of a valuable raw material for preparing products with a high added value, depolymerisation in alkaline (NaOH or KOH) solutions in water or an alcohol-water mixture was used to prepare suberinic acids from suberin. To be acceptable for practical use, a method should be chosen, depending on the application purpose. The obtained suberinic acids were characterised and used as a hydroxyl groups containing component in the biobased polyurethane foam synthesis and also as a binding agent in particle or fibreboard production.

Figure 3 shows that obtaining of suberinic acids from extracted outer birch bark by alkaline-alcohol depolymerisation would be entangled even on a pilot scale. To carry out such procedure, the certain pH and temperature ranges are required if 9,10-epoxy-18-hydroxy octadecanoic acids are to be obtained in high yields. The method includes many centrifugation and drying operations, resulting in large amounts of wastewater (1:20). This makes the process time consuming and laborious.

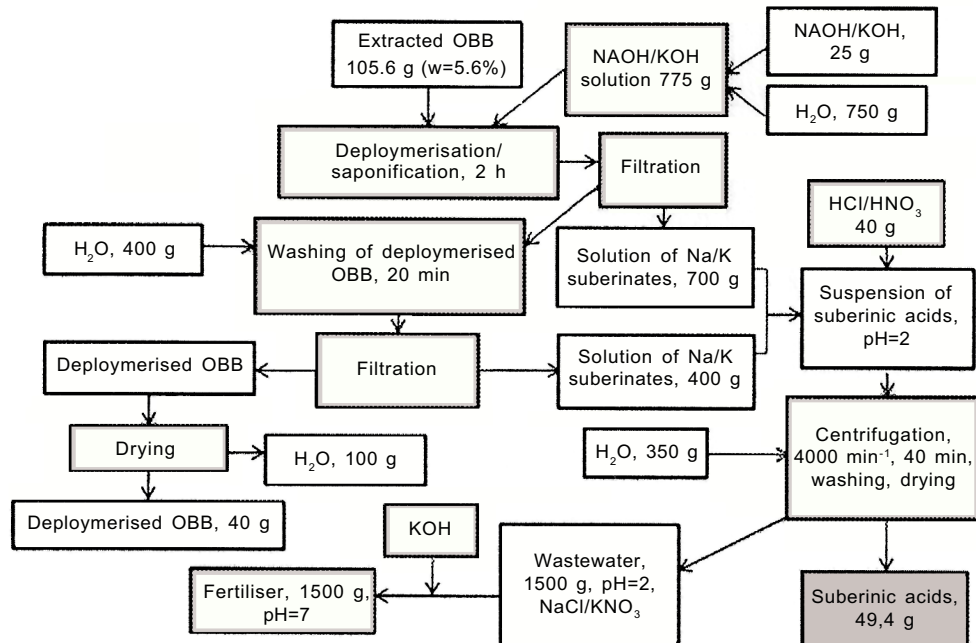


**Table 2.** Properties of suberinic acids obtained by depolymerisation in alkaline-alcohol solution

pH of the solution	Yield, % o.d. extracted outer birch bark	Acid value, mg KOH/g	HEPA content, % o.d. acids fraction
6.0	19.3 ± 0.6	138.1 ± 3.4	58.0 ± 2.4
4.7	11.3 ± 0.3	75.3 ± 1.2	57.3 ± 2.2
2.1	4.9 ± 0.2	165.9 ± 3.2	3.3 ± 0.2
Total average:	35.5	-	17.8

density rigid spray polyurethane foam even if the bio-based content reached 17.6%.

The suberinic acids mixture obtained from extracted outer birch bark by alkaline-water depolymerisation was also used as a binding agent in particle board preparation. The obtained data shown in Table 4 demonstrated that the increase of the pressing temperature from 140 to 180°C at a pressure of 10 MPa resulted in a minor growth of the boards' density and bending strength. Thickness swelling of particle boards



**Figure 4.** Obtaining of the suberinic acids mixture from extracted outer birch bark by alkaline-water depolymerisation

tained mixture of suberinic acids was successfully used as a hydroxyl groups containing component in polyurethane synthesis. By increasing the polyisocyanate and flame retardant content (Table 1) in the polyol system, mechanical properties were slightly improved, whereas the biobased content (16.6%) and closed cell content (90.0%) showed decreased values for the obtained foam samples (Table 3). The preliminarily obtained rigid polyurethane foams had good mechanical properties and high closed cell structure, which complied with the ISO/WD8873-1 standard for medium

**Table 3.** Properties of suberinic acid polyol polyurethane foams

Indices	Acid value of suberinic acid esters polyols, mgKOH/g		ISO/WD8873-1 requirements
	440	432	
Apparent core density, kg/m <sup>3</sup>	49.0 ± 0.0	36.7 ± 1.2	28
Comprehensive strength, MPa	0.28 ± 0.01	0.27 ± 0.00	0.17
Modulus of compression, MPa	6.04 ± 0.36	5.05 ± 0.15	...
Comprehensive strength, MPa	0.19 ± 0.00	0.12 ± 0.01	...
Modulus of compression, MPa	3.97 ± 0.21	2.28 ± 0.17	...
Closed cell content, %	90.0 ± 1.0	94.8 ± 1.2	92.0
Biobased content, %	16.6	17.6	...

moulded at a temperature of 160°C was rather noteworthy, but the particle boards' samples obtained at 180 °C showed excellent water resistance when there was no thickness swelling at all. A good contact between particles help to reduce the penetration of moisture into panels and particles (Wong et al. 1999), but none of the specimens met the lowest density requirement, i.e. 850 kg/m<sup>3</sup> as specified in EN 312 for medium density non load-bearing boards for use in humid conditions (P3). It means that the obtained suberinic

**Table 4.** Properties of particle boards prepared from the mixture of suberinic acids obtained by alkaline-water depolymerisation

Indices	Pressing temperature, °C			EN 312 requirements*
	140	160	180	
Thickness, mm	7.7	7.1	6.6	6-13
Density, kg/m <sup>3</sup>	1160	1220	1270	850
Thickness swelling, %	38.3 ± 5.3	8.5 ± 1.1	0.0	14
Bending strength, MPa	23.9 ± 0.1	27.9 ± 1.0	29.2 ± 1.1	15

\* Non load-bearing boards for use in humid conditions (P3) – Requirements for specified mechanical and swelling properties



acids act as an excellent hydrophobic binding agent to produce high density particle boards with high water resistance and rather high bending strength without the potential emission of formaldehyde.

When pressing at high temperature and pressure, obviously, there occurs the formation of grid-type structures due to the generation of new chemical bonds between suberin and the wood filler; hence, the physico-mechanical characteristics of wood board materials are improved (Sudakova et al. 2008).

Our further experiments related to obtaining of medium density particle boards with the density below 850 kg/m<sup>3</sup> and retaining of high enough bending strength and thickness swelling by reducing the pressure and/or pressing time. Certainly, some problems could arise, because in boards of lower density, the more porous structure allows easy penetration and uptake of water, resulting in higher water absorption, which causes the board to swell, giving rise to a relatively high thickness swelling. Of course, in our case, the positive role will be probably demonstrated by the hydrophobic character of suberinic acids.

Our investigation has shown that it is possible to use one of the plywood production residues – outer birch bark as a raw material for obtaining products of high added value, i.e. polyurethane foam and particle boards, which have good mechanical properties. The used methods are also environmentally friendly, realisable in practice and have a potential to be cost-effective.

## Conclusions

1. If it is necessary to retain epoxy groups or positioned hydroxyl groups in a suberinic acid molecule, or isolated fractions of individual suberinic acids such as HEPA, certain pH and temperature ranges are required, and alcohol-alkaline hydrolytic depolymerisation must be applied, which certainly makes the process more complex as well as time and water consuming.

2. If it is planned to use suberinic acids as a binding agent in particle board compositions, or as a component in an acidic form for polyurethane synthesis, there is no need to retain all functional groups during the preparation process.

3. The obtained biobased polyurethane foams from suberinic acids show very good mechanical properties, which can be developed into novel “green” composite materials.

4. The mixture of suberinic acids obtained by alkaline-water depolymerisation acts as an excellent binding agent to produce high density particle boards with low thickness swelling and high bending strength.

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## ВЫДЕЛЕНИЕ СУБЕРИННЫХ КИСЛОТ ИЗ ЭКСТРАГИРОВАННОЙ БЕРЕСТЫ В ЗАВИСИМОСТИ ОТ ЦЕЛИ ПРИМЕНЕНИЯ

Я. Рыжиков, Я. Зандерсонс, А. Паже, А. Тарденака и Б. Спинце

Резюме

Исследование относится к деревообработке и использованию коры для получения насыщенных высших жирных кислот, содержащих реакционноспособные гидроксильные и эпоксидные группы, которые используются для синтеза производных с высокой добавленной стоимостью для косметики, эмульсий, полимерной промышленности и т.д. Таким образом, применение результатов нашего исследования имеет взаимодействие с наукой материалов. Цель исследования заключается в том, чтобы повысить эффективность использования бересты и информировать аудиторию о широком спектре продукции с высокой добавленной стоимостью, которая может быть получена из этого ценного сырья. Смесь суберинных кислот, полученных с помощью деполимеризации экстрагированной бересты в щелочной воде, действует как отличное связующее для производства высокоплотных древесностружечных плит с высокой водостойкостью и прочностью на изгиб. Жесткие пенополиуретаны на биологической основе, полученные из полиолов суберинных кислот, также показали очень хорошие механические свойства.

**Ключевые слова:** береста, деполимеризация, суберинные кислоты, древесностружечные плиты, пенополиуретаны