

**University of West Hungary  
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Doctoral Thesis

**Grafting of Industrial Cellulose Pulp with Vinyl-acetate Monomer**

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## 1. INTRODUCTION

The behaviour of paper made on a paper machine by planking the fibres – despite the development of ingredients used in paper industry – depends first of all on the physical and chemical properties of cellulose fibres. With the advance of paper-synthetics combinations, however, several disadvantageous properties of paper due to the behaviour of fibres can be excluded.

**There are two basic methods for the completion of paper-synthetics systems:**

- the paper or cellulose is associated with finished synthetics or semi-finished synthetic products. Such products are: plywood paper, paper covered with plastics, paper coated with warmish, and paper treated on its surface or inside. For these combinations foil, latex, synthetic rosin hardening with heat, synthetic dispersions, synthetic solutions and synthetic hot-melts can be used.
- the other way to produce paper-synthetics systems is the modification of cellulose itself. This modification can be done by esterification, alkalization, nucleophil substitution, oxidation, or copolymerization.

As the cellulose itself has a polymer structure, it can only produce block or graft copolymers. The synthesis of cellulose block copolymers cannot be widely applied for the chemical modification of cellulose, since it essentially changes the physical structure of cellulose.

The **chemical modification of cellulose fibre by graft copolymerization** is of great importance because, by grafting a relatively small amount of monomers, this natural macromolecular material can be provided with a number of new advantageous properties. Graft copolymerization makes it possible **to produce industrial cellulose fibres which can be successfully applied for making special, marked papers, and it can be also used as binder fibres of synthetic papers.**

## 2. BACKGROUND TO THE RESEARCH

Cellulose fibre is a natural raw material recurring in a great quantity. The demand to use it more and more widely is increasing. The production of cellulose by-products started as early as the 19th century, however the modification of these materials meant the breaking up the fibrous structure, which made their use more difficult in paper industry. **The modified cellulose made by graft copolymerization, however, keeps its fibrous character, which provides a great advantage regarding its use.**

Analysing the literature on the techniques of graft copolymerization, we can state that initiation by redox systems for initiating the reaction is the technique that can be carried out in the easiest way in industry. Among these techniques the best seems to be the one in which the role of a reducing component is acted by the cellulose itself, and the oxidizing component is a metal of changing valence.

In most cases the grafting reaction was carried out on regenerated cellulose pulp, fibres and films. In some cases cellulose derivatives, above all cellulose esters and ethers were

used. The high price of cellulose and cotton cellulose pulp makes it necessary that a cheaper industrial-technical cellulose fibre should be examined on its grafting ability.

The grafting efficiency of industrial cellulose is affected, to a great extent, by the lignin left behind in the cellulose pulp during exploration or bleaching. This lignin acts as an inhibitor in the grafting reaction of the cellulose pulp, since it retards the formation of macro-radicals required for the beginning of grafting. According to the publications it was stated in the publications studied that the greater amount the lignin the greater the induction period of the reaction.

Few papers deal with the effect of the specific surface of the cellulose pulp on grafting. I have found no papers dealing with the effects of the incrusting materials of industrial cellulose, the content and origin of hemi cellulose on graft copolymerization.

There have been found papers only on indirect measurements in connection with the study of radicals formed in graft copolymerization, but no research has been carried out on the measurement of the susceptibility of radicals.

In Hungary experiments have been going on in the Research Institute of Paper Industry in connection with the grafting of cellulose pulp in paper industry, and experiments related to graft copolymerization in textile industry have been carried on in the Research Institute of Textile Industry, since 1966.

At the Paper Industry Department of the Technical College of Light Industry, which is the predecessor of the Budapest Polytechnic, Rejtő Sándor Faculty of Light Industry, we have been dealing with grafting of industrial cellulose pulp with vinyl monomers since 1974.

### 3. OBJECTIVES OF THE RESEARCH

**My doctoral thesis aims at the study of grafting of industrial cellulose pulp with vinyl-acetate monomer**, which allows for the production of grafted wood cellulose fibres that have a thermoplastic layer on their surface. **The binder fibre (fibrid) produced in this way can be excellently used for producing synthetic papers.** The surface of cellulose-poli(vinyl-acetate) fibres melts in the range of 433-453 K therefore, having undergone thermo shock treatment on a heated calender, it will be adapted for fastening various synthetic and artificial fibres, as well as cellulose fibre.

Textures and papers made of synthetic fibre are widely used, such as geotextiles, special coated printing papers, paper diapers. The material is under bearing force, e.g. mechanical and chemical effects, damaging caused by light or micro-organisms. For the production of materials resistant to the given effects, it is indispensable to carefully select the fibre material and the production technology.

If among the fibrous materials a proper bond cannot be established, thermoplastic powders, fibres or binder dispersions will have to be added. Fibrids can easily mix with other fibres, as they are similar in content and characteristics to those that are used for binding them. The formation of the junction is based on the fact that the synthetic part of the binder fibre melts from heat and, in the course of hardening, a junction among the fibres is established.

**My studies aimed at finding the answers to the questions whether industrial wooden cellulose can be grafted with vinyl-acetate and how the grafting reaction depends on the composition and properties of cellulose.**

#### **4. EXPERIMENTAL**

In my first experiments I dealt with choosing the parameters of graft copolymerization which are best suited to various uses.

Using an initiation method, which has already been studied in literature, that is using Ce(IV) salt, I grafted vinyl-acetate on bleached cellulose and **examined the dependence of graft yield on the conditions of reaction**, such as, temperature, reaction time, concentration of monomer and initiator, and liquor ratio.

In the following part of my investigations **I studied the dependence of graft reaction on the composition and properties of industrial cellulose applied**. I examined the effect of the lignin content, hemi cellulose content and the specific surface of industrial cellulose on graft copolymerization.

The selection of the suitable reaction parameters was followed by **the study of reaction speed and activation energy**.

Next I analysed **the formation of free radicals acting in radical copolymerization of cellulose; then I analysed the whole copolymerization process by tracing the changes in magnetic susceptibility**.

The grafted copolymer produced was examined by means of infrared spectroscopy, thermal analysis and photos taken on microscope.

Finally, **I measured the mechanical properties of sheet structures made of a mixture of copolymer binder fibre and synthetic cut fibre, under conditions standard for paper industry**.

#### **5. RESULTS OF THE RESEARCH**

Among the reaction parameters of the binder fibre, which is the most convenient for practical use, **the adequate temperature has been chosen** in the range of 293-333 K. The results have shown that by increasing the temperature in the reaction the yield also increases, however the increase of the temperature from 323 K to 333 K only results in a few per cent of increase in the volume of the grafted product. For this reason I propose 323 K to be applied for industrial use.

A similar tendency is seen when **the reaction time is examined**. The yield of grafting has been measured in the range of 5-60 minutes of reaction time. According to my measurements the amount of the grafted product increases when reaction time is increased, however after 30 minutes the rise of curve starts to level out and, if the reaction time is increased from 40 to 60 minutes, it will only result in a minor increase of yield. For this reason I propose a reaction time of 40 minutes to be applied for producing binder fibre.

**To find the adequate initiator concentration** I measured the yield of graft copolymerization in the range of  $10^{-3} - 5 \cdot 10^{-3}$  mol/dm<sup>3</sup> Ce(IV) ammonium-sulphate concentration. I experienced the greatest value when  $2 \cdot 10^{-3}$  mol/dm<sup>3</sup> initiator concentration was set. If the concentration is lower, it is unlikely for the radicals to occur in a sufficient quantity, on the other hand a higher concentration may increase the speed of primary recombination. On the basis of this, I propose the use of  $2 \cdot 10^{-3}$  mol/dm<sup>3</sup> Ce(IV) ammonium-sulphate concentration for the industrial production of graft copolymerization.

**The effect of the monomer concentration** of vinyl-acetate on the yield of graft copolymerization **was examined** in the range of 0,25–2,00 mol/dm<sup>3</sup> concentration and I stated that the yield of reaction increased up to the value of 1,00 mol/dm<sup>3</sup> monomer concentration, above this value however, a decrease was seen, which was probably caused by a proportional increase in the number of chain-transmission reactions. For graft copolymerization the use of 1,00 mol/dm<sup>3</sup> vinyl-acetate concentration has been found.

**Examining the interrelation between the yield of graft reaction and liquor ratio** I have stated that until the liquor ratio reaches the 200 value, the yield rapidly increases, however above this value it decreases. It is supposed that an extreme mixture content has an unfavourable effect on the reaction. As a grafted product produced with a liquor ratio of more than 100 value is already sticky, difficult to treat and unapt for making sheets, I propose a liquor ratio of 100 value for practical use.

Having stated the adequate parameters of graft copolymerization, I studied the effect of the lignin content, the hemi cellulose content and the pulping grade of industrial cellulose, also its specific surface on the graft reaction, under the conditions chosen in my earlier experiments.

The grafting of holocellulose is affected by the quantity of lignin left over in the fibrous material during bleaching. The lignin behaves as an inhibitor as it retards the formation of macro-radicals required for starting the process of grafting. **The effect of copolymerization reaction on the lignin content of cellulose was examined** by grafting unbleached cellulose, the lignin content of which was decreased in five steps by treating it with sodium hypochlorite. I stated that in the case of an industrial cellulose containing lignin less than 2 % the extent of grafting was satisfactory. To examine the inhibition effect of lignin, I also grafted unbleached cellulose containing 12 % lignin, using a reaction time in the range of 0–120 minutes. Owing to the inhibition effect of lignin the grafting reaction starts with an induction period of 70–80 minutes for such lignin content.

Grafting is also affected by hemi cellulose, which occurs, beside pure alpha cellulose, in the carbohydrate (holocellulose) content of cellulose fibres used in paper industry. **I studied the effect of hemi cellulose content on grafting reaction** by grafting bleached cellulose that contained 5 various types of hemi cellulose. As the different specific surfaces of the different kinds of cellulose has an effect on grafting I have always done the grafting of the alpha cellulose pulp gained from the different kinds of cellulose in order to test the tendency typical for the effect of hemi cellulose. I have found that an increasing quantity of hemi cellulose increases the yield, which is proven by the fact that by removing the hemi cellulose pulp from industrial cellulose, the grafting efficiency of the alpha cellulose obtained is lower than that of industrial cellulose containing hemi cellulose too. This can be explained by the greater reactivity and better availability of hemi cellulose.

**The effect of the specific surface of cellulose on grafting reaction was examined** by using bleached sulphate pine cellulose, which was pulped in a time range of 10–60 minutes and the pulped cellulose samples were grafted with vinyl-acetate. To define the external specific surface of cellulose fibre, I applied the relatively rapid and accurate colloid-kongo colouring adsorption technique. The yield of grafting reaction was the greatest when a cellulose with a specific surface of 4,41 m<sup>2</sup>/g was grafted, which was achieved by a pulping time of 35 minutes. My experiments have shown that a further increase of pulping time leads to a decrease in yield, because in the case of an identical initiator concentration, while the specific surface steeply increases, the number of radicals per unit surface decreases. This is supported by the fact that the maximum yield of grafting reaction occurred at a cellulose with a greater specific surface (8,512 m<sup>2</sup>/g) when the initiator concentration was increased.

**For studying the kinetics of grafting reaction** I carried out the grafting at temperatures 293, 303, 313 and 323 K. From the results **I calculated the average activation energy values** for each temperature, using the Arrhenius equation, which is well suited to approximate calculations. The results have shown that – according to my expectations – when the temperature increases, the average activation energy of the reaction decreases.

As it has already been mentioned, the reaction goes on in a complicated heterogeneous system thus, apart from the kinetics of the chemical reaction, certain macro-kinetic factors affecting the speed of material transmission may also have an effect on the gross speed of the reaction. From the results of the experiments on parameters **I have calculated the gross speed of the reaction** in the range of 20–60 reaction time after the induction period and, by analysing the curves, I have defined the equations for the changes of speed in time. These are:

at temperature 293 K.	$v = 2,32114 \cdot 10^{-5} t^2 - 0,00269 t + 0,09354$
at temperature 303 K.	$v = 2,45545 \cdot 10^{-5} t^2 - 0,00283 t + 0,09528$
at temperature 313 K.	$v = 2,66932 \cdot 10^{-5} t^2 - 0,00298 t + 0,09543$
at temperature 323 K.	$v = 3,04886 \cdot 10^{-5} t^2 - 0,00336 t + 0,10166$

When the invert of the momentary monomer concentration is plotted against reaction time, it can be stated that **the function is a linear one at those four temperatures studied, which proves the fact that the gross reaction is a second order reaction.**

**The free radicals taking part in graft copolymerization were examined by measuring the changes in magnetic susceptibility** with the equipment for the measurement of susceptibility, available at the magnetic laboratory of the Hungarian Academy of Science Central Research Institute for Physics. Having defined the magnetic susceptibility of the materials taking part in the reaction, tests on kinetics have been carried out by use of the method.

As in our case, due to a low polymerization speed, no essential copolymerization could occur, the negative change of susceptibility arising from the copolymer did not compensate the positive change arising from the formation of free radicals or from the transformation  $Ce^{4+} \rightarrow Ce^{3+}$ . It can be stated, therefore, that **the measuring of magnetic susceptibility is well suited for studying the free radicals arising from the grafting of cellulose pulp with vinyl-acetate.** The results of the tests carried out by use of measuring the changes in the magnetic susceptibility of graft copolymerization completely justify the results achieved in chemical micro kinetics.

**The thermal and infrared spectroscopy examinations** of the grafted product have proved the formation of cellulose-poly-(vinyl acetate) copolymer. **On the photos taken with polarization microscope it can be well seen** that the cellulose contains a significant quantity of poly-(vinyl-acetate) agglomerate, the role of which is significant in its use as a fibrid. **On the photos taken with scanning electron microscope** synthetic sections taking place on the cellulose fibres **can be also experienced**. It can be also seen on the photos that the thermoplastic layer on the fibre surface melts during the heat treatment of grafted fibre, thus it is apt for making junctions between the cellulose and synthetic fibres occurring in the structure of synthetic paper.

**The mechanical tests of the experimental papers** have been carried out with techniques required by the standards in paper industry. In this way dry and wet tearing strength, tearing index, tearing lengthening, stiffness and the dual-folding number have been defined. The mechanical tests of the experimental papers prove that **the cellulose grafted with vinyl-acetate can be applied for producing synthetic papers when it is used as a copolymer fibrid**.

## 6. MAIN RESULTS

1. On the basis of my investigations I have stated that the grafting of industrial cellulose fibre with vinyl-acetate monomer, cerium(IV)-ammonium-sulphate initiator can be carried out.

In the case of sulphate-cellulose fibre, which I examined, the reaction parameters of binder fibre, best suited to practical use are:

- temperature: 323 K
- reaction time: 40 minutes
- monomer concentration: 1 mol/dm<sup>3</sup>
- initiator concentration: 2·10<sup>-3</sup> mol/dm<sup>3</sup>
- volume rate: 100 cm<sup>3</sup>/g cellulose

2. The yield of graft copolymerization depends on the lignin content of industrial cellulose, the lignin behaves as an inhibitor in the reaction. I have stated that when grafting industrial cellulose with vinyl-acetate, a sufficient yield can be reached only if the lignin content of cellulose fibre is less than 2 %. As a result of lignin content of industrial cellulose pulp, the graft copolymerization starts with an induction period, the duration of which amounts to 70-80 minutes if the lignin content is 12 %.

3. I have stated that the hemi cellulose content of industrial cellulose fibre increases the yield of graft copolymerization, the grafting efficiency of the alpha cellulose obtained is lower than that of industrial cellulose containing hemi cellulose too. This can be explained by the greater reactivity and better availability of hemi cellulose.

4. In the case of the examined reaction, the increase of the specific surface also increases – up to a given limit – the reaction yield. The examined cellulose pulp showed a maximum increase in mass with an external specific surface of 4,41 m<sup>2</sup>/g. My experiments have shown that a further increase of the pulping time results in a decrease of yield because, in the case of an identical initiator concentration, while the specific surface rapidly increases, the number of



radicals per unit surface decreases. This is supported by the fact that, by increasing the initiator concentration, the maximum yield of graft reaction has occurred at the grafting of a cellulose fibre of greater surface (8,51 m<sup>2</sup>/g).

5. When studying the kinetics of the process I have stated that the gross reaction of grafting industrial cellulose with vinyl-acetate monomer is a second order reaction, which is proven by the fact that the invert of the momentary monomer concentration of the system plotted against time is a linear function. The rise of the curves, that is, the reaction speed increases when the temperature in the range of 293–323 K is increasing, while the average activation energy decreases.

6. If the reaction speed is small, the measurement of magnetic susceptibility can be applied for the examination of free radicals arising from the grafting of cellulose pulp with vinyl-acetate.

7. The cellulose fibre grafted with vinyl-acetate can be used to produce synthetic papers, which is proved by the results of sheet structure tests.

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