MANUFACTURE, CHARACTERIZATION AND USE OF MICROCRYSTALLINE PULP IN THE FOOD INDUSTRY

C. Stanciu
University “Dun[rea de Jos”, Galaţi, Faculty of Engineering, Brăila, Romania

INTRODUCTION

Microcrystalline pulps (micronized) are materials made of smaller sized particles of tens microns obtained by pulp mechanical or hydrolytic destruction, or by the combination of these two treatments. Pulp is a polymer with a two-phase supermolecular structure, characterized by the existence of the crystalline and non-crystalline fields. By pulp mechanical dispersing two phenomena take place: reduction of particle geometrical sizes by their splitting, simultaneously with diminishing the degree of polymerization due to splitting of main chains. The main drawbacks of producing microcrystalline pulps using the mechanical process have been noticed to consist of metal contamination of products, coming from equipment material and consequently, their range of uses is restricted. Hence, chemical processes have an intensive development. Chemical micronization of pulp consists of its treatment with various chemicals, able to destroy certain fields (non-crystalline) of supermolecular structure. Fig. 1 shows different ways of producing microcrystalline pulps (Stanciu, 2005).

PULP

Mechanical process	Thermomechanical process	Chemical process			Precipitation from solution
		Acid hydrolysis	Alkali-oxidative treatment	Alkali hydrolysis	
Fibre fragments		Microcrystalline pulp			Non-crystalline powder

Figure 1. Different ways of producing microcrystalline pulps.

Chemical micronized pulp has a maximum degree of crystalinity and a maximum density in relation with the other types of cellulose materials being made of aggregates with high orientation, bound by steady intermolecular bonds (figure 2).

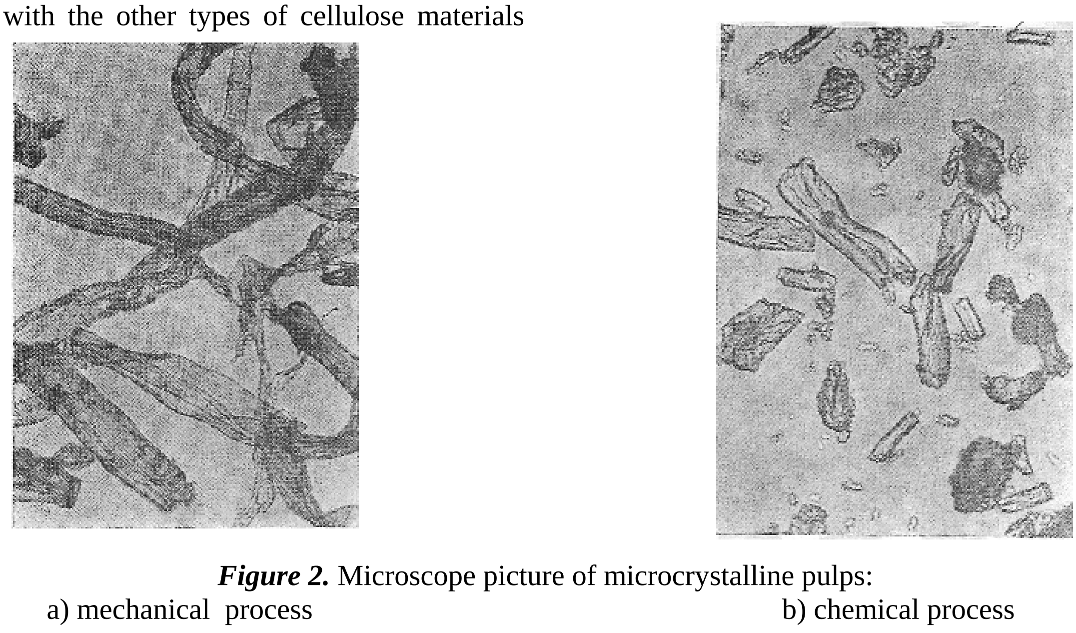

Figure 2. Microscope picture of microcrystalline pulps:
a) mechanical process b) chemical process

Reduction of particle sizes in the micronization process has as a result the change of specific area of cellulose materials, its value varying between 4000-6000 cm²/g. The increase of specific area in connection with a major modification of the ratio between crystalline and non-crystalline fields results in the significant modification of filtration coefficient and ability of micronized materials to retain fine impurities from different media. Table 1 shows the characteristics of some microcrystalline pulps from various origins

Table 1. Characteristics of some microcrystalline pulps

Commercial name	**Degree of polymerization**	**Crystallinity index**	**Filtration coefficient x 10^{-3} cm³/scm²**	**Sum of fractions less than 10 microns %**	**Ash %**
ARBOCEL type 800/3	1017	37.0	0.475	14.7	0.27
SOLKA FLOC –BW- 40	976	53.5	0.631	21.5	0.10
SOLKA FLOC –BW-20	1164	55.5	0.888	9.3	0.10
AVICEL	299	67.0	1.786	85.2	0.18
Pulp (Finland)	339	54.3	1.000	33.2	0.32

Microcrystalline pulp is used as filler in pharmaceuticals like tablets (Steige 1976, Sokolor 1986). Added in small amounts in cold milk or in ice cream, it preserves their taste for a long time. It is assumed that microcrystalline pulp prevents the growth of ice crystals. In other cases, this pulp is used as films for a long fruit protection or for encapsulation and stabilization of unsteady substances. The paper shows the manufacture technology, the characteristics of microcrystalline pulp obtained for food industry as well as the results achieved at oil and wine filtration.

1. MATERIALS AND RESEARCH METHODS

Parameters of the microcrystalline pulp making process are illustrated in Table 2.

Table 2. Parameters of microcrystalline pulp making process.

Process parameters	**Values**
Raw material	Beech dissolving pulp
Previous sulphuric acid concentration	45.6%
Liquor to wood ratio	1/20
Reaction temperature	43-45°C
Reaction time	4 hrs.
Filtration time	4.5 hrs.
Washing time	7.5 hrs.
Drying time	110°C
Drying time	24 hrs.
Yield	86.2

The pulp produced has been characterized by the crystallinity index found by IR spectroscopy, the filtration coefficient found by grading measurement (fractioning on vibrating sieves with sizes between 40 and 400 microns) and microscope controlled.

The filtration coefficient shows from the quantity point of view, the filtration capacity of a cellulose material being defined as the volume of filtration aid passing in time unit through the surface unit of a certain thickness layer. For tests, the Brecht device has been used.

2. RESULTS AND DISCUSSIONS

Table 3 shows the characteristics of microcrystalline pulps produced.

Table 3. Characteristics of microcrystalline pulps.

Characteristics	P1	P2
Ash,%	3.2	3.5
Particle size distribution (fractions less than 71 microns),%	69	58
Molecular weight	553	871
Filtration coefficient, x10^{-3}, cm³/s·cm²	1.19	2.04
Cristallinity index, %	37.76	37.2
Moisture	3.2	3.5

The content of heavy metals has not increased over 10 ppm/g microcrystalline pulp. As a result of micronization, a significant rise of the filtration coefficient is noticed. Beech dissolving pulp has a filtration coefficient of $0.48 \cdot 10^{-3}$cm³/s·cm², after micronization this coefficient is 1.19-2.04 cm³/s·cm². Microcrystalline pulps have been used with good results in the oil industry in the processes of oil separation from wax and stearin, in combination with activated diatomite (2.1 kg/70 t oil, day). During these processes, it is important that the filter material should be dry (moisture does not allow the pass through a filtration metallic sieve), should have a certain granularity (not to "*plug*" at low granularities), not to react with the filter and finally provides the oil specific "*brightnes*s" and "clearness". Research carried out highlighted that, pulp grading can be modified by changing acid concentration, reaction time and temperature (crystallinity index and filtration coefficient increase with temperature showing an intensification of hydrolisis at a temperature of 60-70°C). Good results have also been attained when using the microcrystalline pulp, as substituient of Kiselgur on the wine filtration plant, in the Schenek type powdery layer filter.

3. CONCLUSIONS

Microcrystalline pulps produced by the chemical process cant be used in various fields in the food industry. For various applications in food industry, microcrystalline pulps should present specific characteristics (grading, specific area, filterability etc.). A special attention should be given to the content of heavy metals, that has to be as lower as possible. Capability of oil separation from wax and stearin was not influenced by the replacement of Crystal Theorit with microcrystalline pulp.

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