



BENECEL®

high purity
METHYLCELLULOSE
METHYLHYDROXYPROPYLCELLULOSE

PHYSICAL AND CHEMICAL PROPERTIES

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BENECEL®

METHYLCELLULOSE
METHYLHYDROXYPROPYLCELLULOSE

BENECEL products are high purity, water-soluble, nonionic cellulose ethers designed for use in regulated industries as water retention aids, thickening and filmforming agents, protective colloids, suspending and emulsifying agents, binders and stabilizers. In this brochure the main characteristics of BENECEL have been compiled. It summarizes areas of application and contributes to the professional use of BENECEL.

For the Aqualon Division, the chemistry of cellulose ethers and their technical application are major subjects for research and development.

The range of water- and solvent-soluble cellulose ethers for regulated industries produced and supplied by Aqualon currently includes the following products:

BENECEL	purified methylcellulose and methylhydroxypropylcellulose
AQUALON®/BLANOSE® Cellulose Gum*	purified sodium carboxymethylcellulose
KLUCEL®	hydroxypropylcellulose

Some literature describes or designates methylhydroxypropylcellulose (MHPC) as hydroxypropylmethylcellulose (HPMC); both chemically and physically the two are identical, and distinct in designation only.

* AQUALON and BLANOSE are both registered tradenames of Hercules' Cellulose Gums
AQUALON CMC is produced in the USA and BLANOSE CMC is produced in France

CHEMISTRY OF CELLULOSE ETHERS

The starting material for the manufacture of cellulose ethers is highly purified and bleached cellulose.

Cellulose is a polysaccharide composed of anhydroglucose units, which are linked through Beta-glycosidic bonds. The number "n" of anhydroglucose units in the polymer chain is defined as the degree of polymerisation (DP).

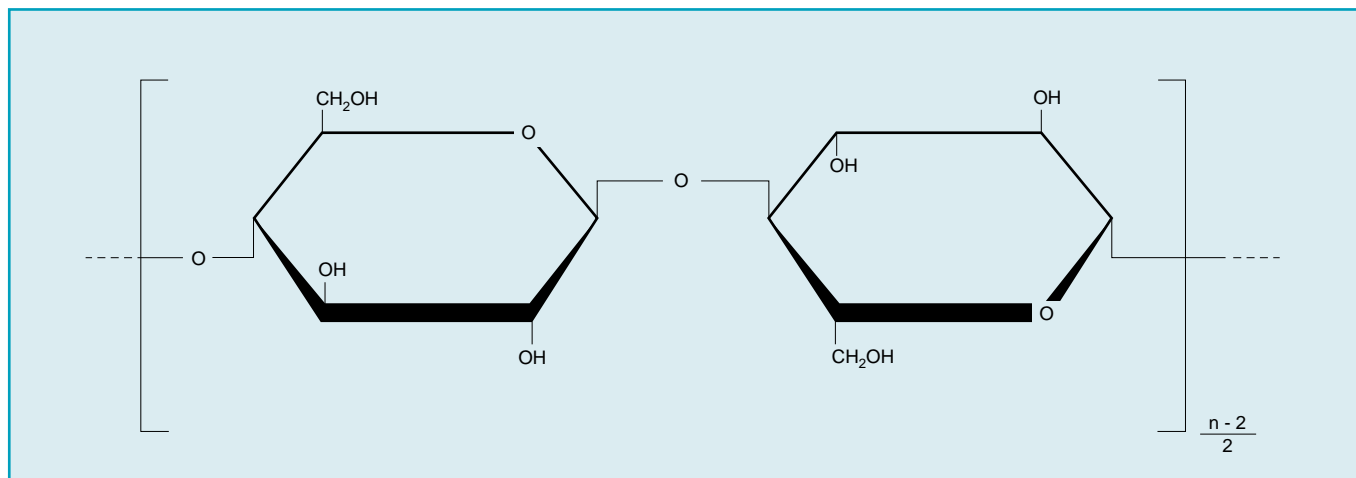
Each anhydroglucose ring carries three free OH-groups at positions 2,3 and 6. Ethers of cellulose are formed by substituting one or more of the three hydroxyl groups. The distribution of the substituents introduced onto the polymer chain is largely determined by the relative reactivity of these three OH-groups.

The number of substituted hydroxyl groups per anhydroglucose unit is expressed as DS or average degree of substitution. The DS can vary between 0 and 3.

In the case of alkoxylation the molar ratio of alkoxy groups in the side chains to cellulose is specified and expressed as the average molecular substitution (MS).

Instead of DS and MS the weight percent of the substituents in the cellulose ether is often quoted. The use of one etherification agent in the substitution process results in a simple cellulose ether, whereas using different kinds of agents leads to mixed ethers. Industrial cellulose ethers are alkyl, alkylhydroxyalkyl, hydroxyalkyl, and carboxyalkyl ethers.

Figure 1.
Structure of cellulose



Cellulose ethers are divided into ionic and nonionic types. Nonionic cellulose ethers like BENECEL methylcellulose and KLUCEL hydroxypropylcellulose carry electrically neutral substituents. The ionic cellulose ethers, e.g. AQUALON®/BLANOSE® sodium carboxymethylcellulose*, contain substituents which are electrically charged.

Nonionic cellulose ethers can be further subdivided according to their solubility in cold, warm or hot water, and in solvents, see figure 11 at the inside back cover.

Abbreviations of the chemical names have been introduced, as shown below. They will also be used in this brochure.

The manufacturing process of BENECEL is carried out in either one or several steps, the reactions taking place are alkalization and etherification, followed by purification.

BENECEL is produced either with a mixture of reactants or by stepwise addition of the substituents. Subsequently it is purified by washing with hot water, dried, ground and screened to the desired particle size.

All lots of BENECEL are analysed in accordance with governmental regulations to ensure their compliance with local and industry quality standards.

Table 1.
Aqualon cellulose ethers for food applications.

Chemical Term	Abbreviation	Aqualon Trademark
Methylcellulose	MC	BENECEL®
Methylhydroxypropylcellulose	MHPC	BENECEL®
Hydroxypropylcellulose	HPC	KLUCEL®
Sodium carboxymethylcellulose	CMC	AQUALON®/BLANOSE® *

* AQUALON and BLANOSE are both registered tradenames of Hercules' Cellulose Gums
AQUALON CMC is produced in the USA and BLANOSE CMC is produced in France

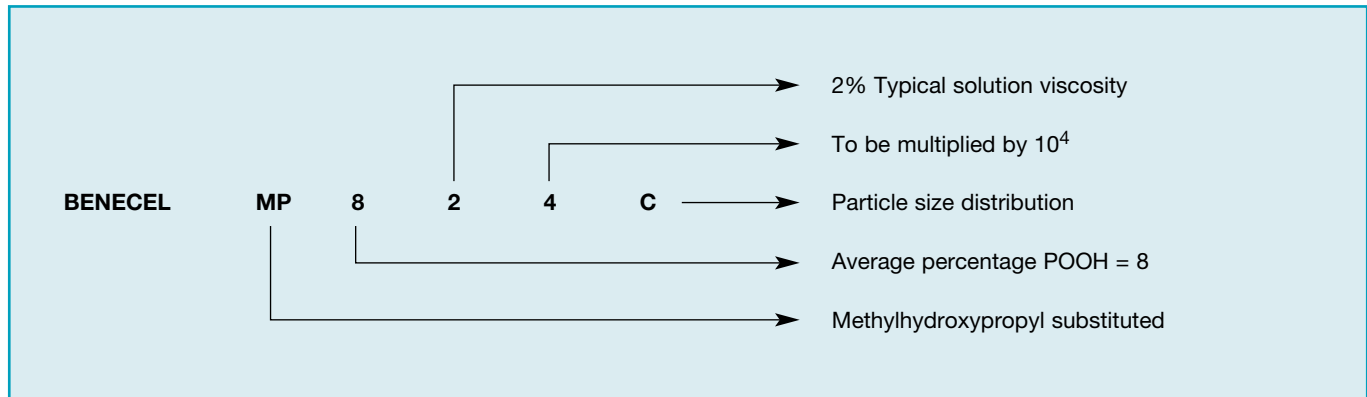
DESIGNATION OF BENECEL® PRODUCTS

BENECEL is the Aqualon trademark for MC and MHPC that meet the governmental regulations for their application in food, pharmaceutical, and personal care products.

The designation of BENECEL products includes the following elements:

- chemical identity of the substituent(s), using
 - M : methyl
 - MP : methylhydroxypropyl
- the average % of second substituent, using the first digit
- the 2% solution viscosity, using digits two and three
- C denotes a selected particle size (see table 5 on page 8)

In the example below the designation of a course grind, 20,000 mPa.s type MHPC is clarified:



SPECIFICATION OF BENECEL®

The principal purity criteria for BENECEL methyl(hydroxypropyl)-cellulose have been listed in table 3. BENECEL products are available in types that comply with the purity requirements for methylcellulose and methylhydroxyalkylcellulose as laid down either in the latest edition or amendment of:

- the European Pharmacopoeia
- the United States Pharmacopoeia
- EC Commission Directive 98/86/EC. amending Commission Directive 96/77/EC, laying down specific purity criteria on food additives other than colours and sweeteners
- Food Chemical Codex
- the Codex Alimentarius

Table 2 summarizes these compliances. Available types of BENECEL products series and their specifications can be obtained from Product Data Sheet 56.100.

Table 2.
Regulatory clearances of BENECEL products.

	BENECEL	
	M	MP
Eur.Pharm.	+	+
USP	+	+ ¹⁾
EC (food)	+	+
FCC	+	+
Codex	+	+

1) Users are invited to review the deviation of some MP grades from some minor aspects of the US Pharmacopoeia with Aqualon.

Table 4.
Types and substitution ranges of BENECEL.

BENECEL type	Reactant	Approximate substitution range	
		% OCH ₃	% POOH
M	CH ₃ Cl	28 – 31.5	
MP 3	CH ₃ Cl , C ₃ H ₆ O	25 – 30	3 – 6
MP 8	CH ₃ Cl , C ₃ H ₆ O	19 – 24	7 - 12

Each shipment of BENECEL is accompanied by an analysis report to certify compliance with the regulations for the end-use.

Table 4 lists the substitution ranges of the BENECEL products series. Further physical data on dry BENECEL products are mentioned in table 5.

Table 3.
BENECEL purity criteria.

Loss on drying	: < 5.0%
pH (1% solution)	: 5.5 - 8.0
Sulphated ash/Residue on ignition	: < 1,0%
Chlorides (Cl-)	: < 0.5%
Heavy metals: Total	: < 10 ppm
- Lead	: < 5 ppm
- Arsenic	: < 3 ppm
- Cadmium	: < 1 ppm
- Mercury	: < 1 ppm

Exception: for the MP C-types, a max. 10% for loss on drying, should be taken into account (does not comply with USP).

Table 5.

Physical data on BENECEL®.

Appearance		white to off-white powder or granules
Sieve analysis		
Standard	on 0,125 mm (ASTM no. 120)	8% max.
	on 0,8 mm (ASTM no. 20)	1% max
C-types	on 0,2 mm (ASTM no. 70)	55 – 90 %
Biological oxygen demand BOD ₅ , mg O ₂ /mg product		0 - 0.1
Bulk density, g/l		200 - 500
Decomposition temperature, °C		>220

Please consult your representative on types that may serve your specific needs

Table 6.

Properties of BENECEL solutions.

Viscosity-Ranges of 2% aqueous solutions at 20°C and 20 rpm, mPa.s, Brookfield RV	
BENECEL M	10 - 4.000
BENECEL MP	3 - 50.000
Flocculation range of 0,5% aqueous solutions	
BENECEL M	50 - 75°C
BENECEL MP	60 - 90°C
Surface tension at 20°C of 0,1% solutions mN/m	45 - 55
Interfacial tension at 20°C of 0,1% solution versus paraffin oil, mN/m	app.20
pH-value of 1% solutions at 20°C	typically 5.5 - 8.0
pH-stability at 20°C	pH 2 to 11
Freezing point of 2% aqueous solutions	0°C
Density of 2% aqueous solutions at 20°C, g/ml	1.0032

Some of the analytical methods used are described in the appendix, page 20.

PROPERTIES OF BENECEL®

BENECEL cellulose ethers are white to off-white powders of several particle size ranges, with neutral taste and odor. They are soluble or able to swell in cold water to form a colloidal solution. Some are also soluble in special organic solvent mixtures.

SOLUBILITY IN WATER

All BENECEL types are soluble in water in a colloidal state. The solubility depends on the BENECEL concentration, the temperature of the solution, and on the presence of other components such as salts or organic solvents. BENECEL is not soluble in hot water, as discussed later.

HYGROSCOPY

Cellulose ethers absorb water if stored in humid conditions, this is again released in dry air. BENECEL products are shipped in moisture-proof containers, which protect them from moisture pick-up.

TEMPERATURE STABILITY

Although BENECEL products are organic compounds, they exhibit excellent temperature stability. Darkening of color and slow softening only occur at temperatures above 140°C. Decomposition occurs at temperatures above 220°C.

REGULATORY STATUS

BENECEL is produced in accordance with the guidelines of the Food and Drug Administration (FDA) and according to Good Manufacturing Practices (GMP). MC and MHPC are also listed in the Food Chemicals Codex as approved food additives. These products are also listed in the Codex Alimentarius.

BENECEL IN PHARMACEUTICALS

The European Pharmacopoeia describes the required purity criteria for MC and MHPC. MC and MHPC are components of the current edition of the United States Pharmacopeia. BENECEL types that meet these requirements are available. Where applicable local regulations have to be taken into consideration.

BENECEL IN FOOD PRODUCTS

In Europe the characteristics of MC (=461) and MHPC (=E464) have been described as food additives in Council Directive 98/86/EC and its amendments.

The users in each member state and more generally in any country are invited to check what are the additional regulations for their specific use in the food recipes, or to consult Aqualon.

Based on all available data, JECFA (FAO/WHO) has classified the Acceptable Daily Intake (ADI) of MC and MHPC as "non specified" and can be used within the bounds of Good Manufacturing Practices (GMP).

BENECEL products, which meet the Food Chemical Codex are available. BENECEL M and MP products comply under several CFR numbers.

BENECEL IN COSMETICS AND PERSONAL CARE PRODUCTS

MC and MHPC have good skin and mucous membrane compatibility. They have been used for many years in cosmetic preparations with no known risks. Local regulations are to be taken into consideration.

TOXICOLOGY

BENECEL is not metabolized in the digestive tract of the body. It is used as an additive for food, pharmaceuticals and cosmetics.

ECOLOGY

An analysis of the toxicity of BENECEL® in fish, algae and daphnia revealed LC₅₀ values of >1000 mg/l (Goldorfen). The LC₅₀ value indicates that concentration of the material tested at which 50% of the test animals survive. BENECEL cellulose ethers show no biological oxygen demand after 5 days (BOD₅). They do not degrade biologically during this time.

SAFETY DATA

As with all organic products there is a risk of fire and dust explosion when BENECEL products are being handled. Fire can be caused by external sources or selfignition at temperatures over 360°C.

A dust explosion hazard always exists when accumulations of fine dust occur. Clouds of fine dust of BENECEL - when ignited- explode violently.

Spilled BENECEL should be cleaned up dry. Floors subject to dusting with BENECEL can become slippery when wet.

A Safety Data Sheet is available upon request.

PREPARATION OF SOLUTIONS OF BENECEL®

Dissolution of BENECEL takes place through progressive solvation of the polymer molecules. Complete wetting and good dispersion of the BENECEL particles are important.

AQUEOUS SOLUTIONS

The lower the temperature of the water used and the higher the stirring speed, the better the solubility of BENECEL. However, care should be taken to avoid air entrainment when preparing solutions, particularly since highly viscous BENECEL solutions de-aerate very slowly. This may influence viscosity reading. The relationship between rate of hydration and stirring intensity is illustrated in figure 2.

Figure 3 illustrates the influence of the temperature of the make-up water on final solution viscosity.

Figure 2.
Viscosity development of BENECEL MP 324C (2% aqueous solution) as a function of stirring speed.

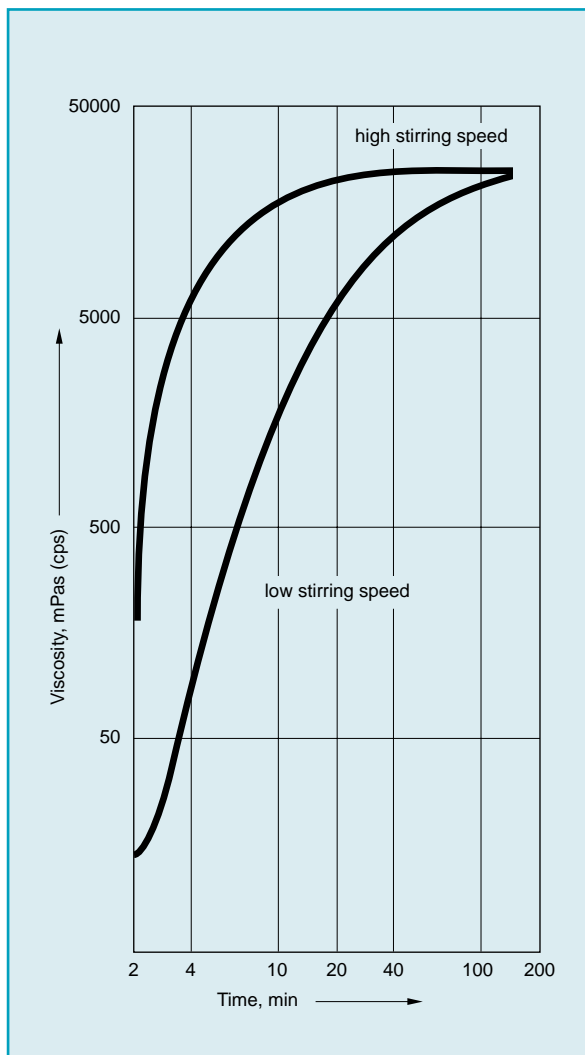
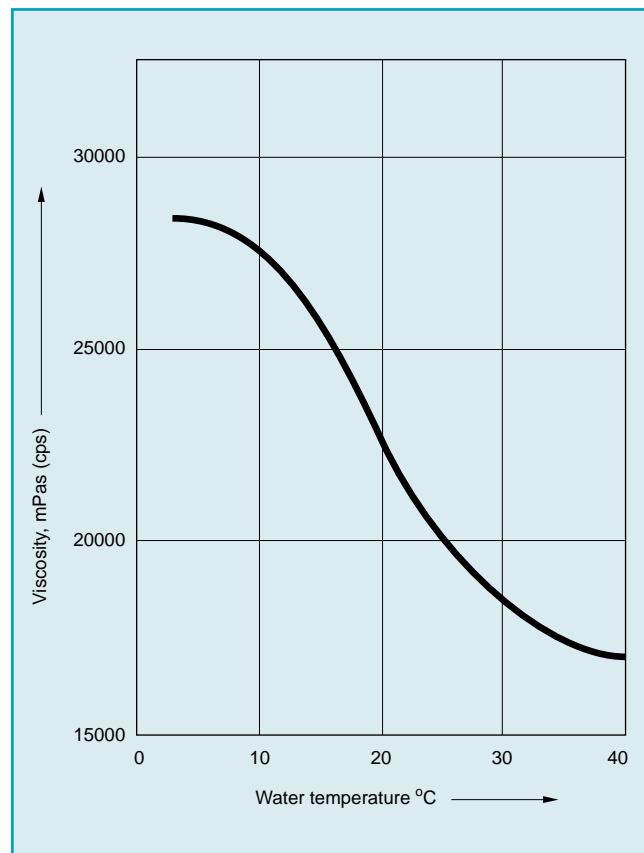


Figure 3.
Solution viscosity at 20°C of 2% BENECEL MP 324C in relation to the temperature of the make-up water.



For the preparation of aqueous BENECEL solutions different procedures are used for granules and powder.

Granular types

BENECEL® should be added quickly to water, which is stirred mechanically or by hand. Continue until swelling occurs. After that only occasional stirring is necessary until the cellulose ether has dissolved completely. If the addition is too slow, a part of the granules dissolves, which increases the viscosity of the water and hinders the dispersion of the remaining product.

Powders

BENECEL powders have been developed for dry blending with other powdered materials. Such dry blends allow lumpfree dispersion of BENECEL in water.

For dissolving pure BENECEL powder the following method is recommended:

Hot-Cold Solution

BENECEL powder is stirred into water and dispersed at a temperature of at least 70°C. A third of the water needed for the whole process is sufficient for this purpose. After the powder has been completely wetted, the rest of the water, this time cold water or ice, is added and stirring is continued until the powder has completely dissolved.

PROPERTIES OF SOLUTIONS

VISCOSITY OF AQUEOUS SOLUTIONS

As BENECEL[®] dissolves, the viscosity of the water increases. This thickening effect is dependent upon the degree of polymerisation/molecular weight and on the BENECEL concentration.

The higher the degree of polymerisation (and thus the molecular weight), the higher the solution viscosity. See figure 4 and table 7 below.

Figure 4. 2% solution viscosity of various types of BENECEL as a function of molecular weight (MW) and degree of polymerisation (DP).

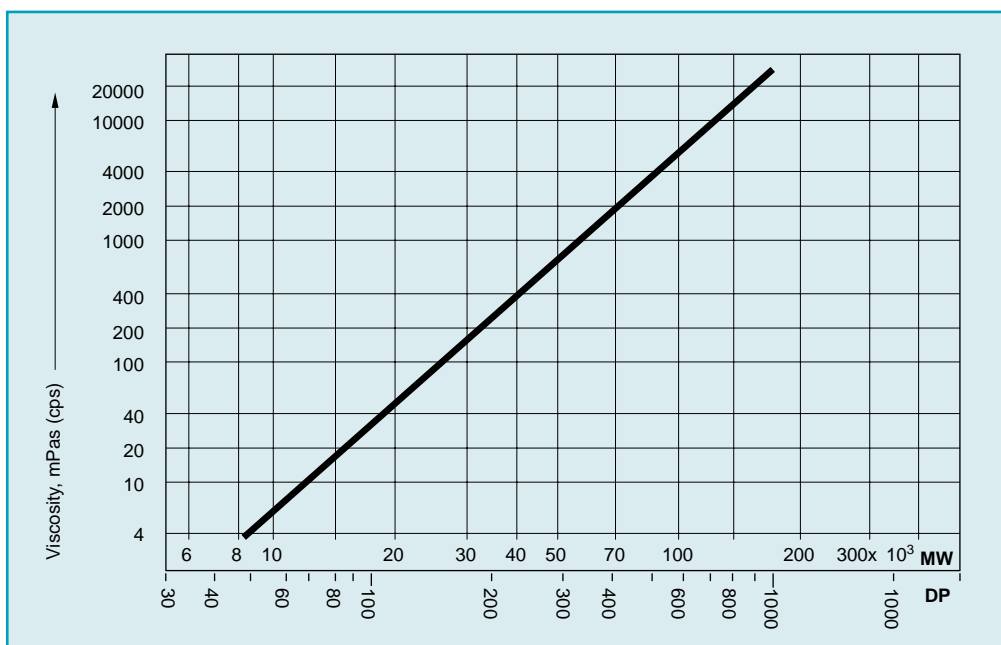


Table 7. Viscosity and molecular weight of methylcellulose.

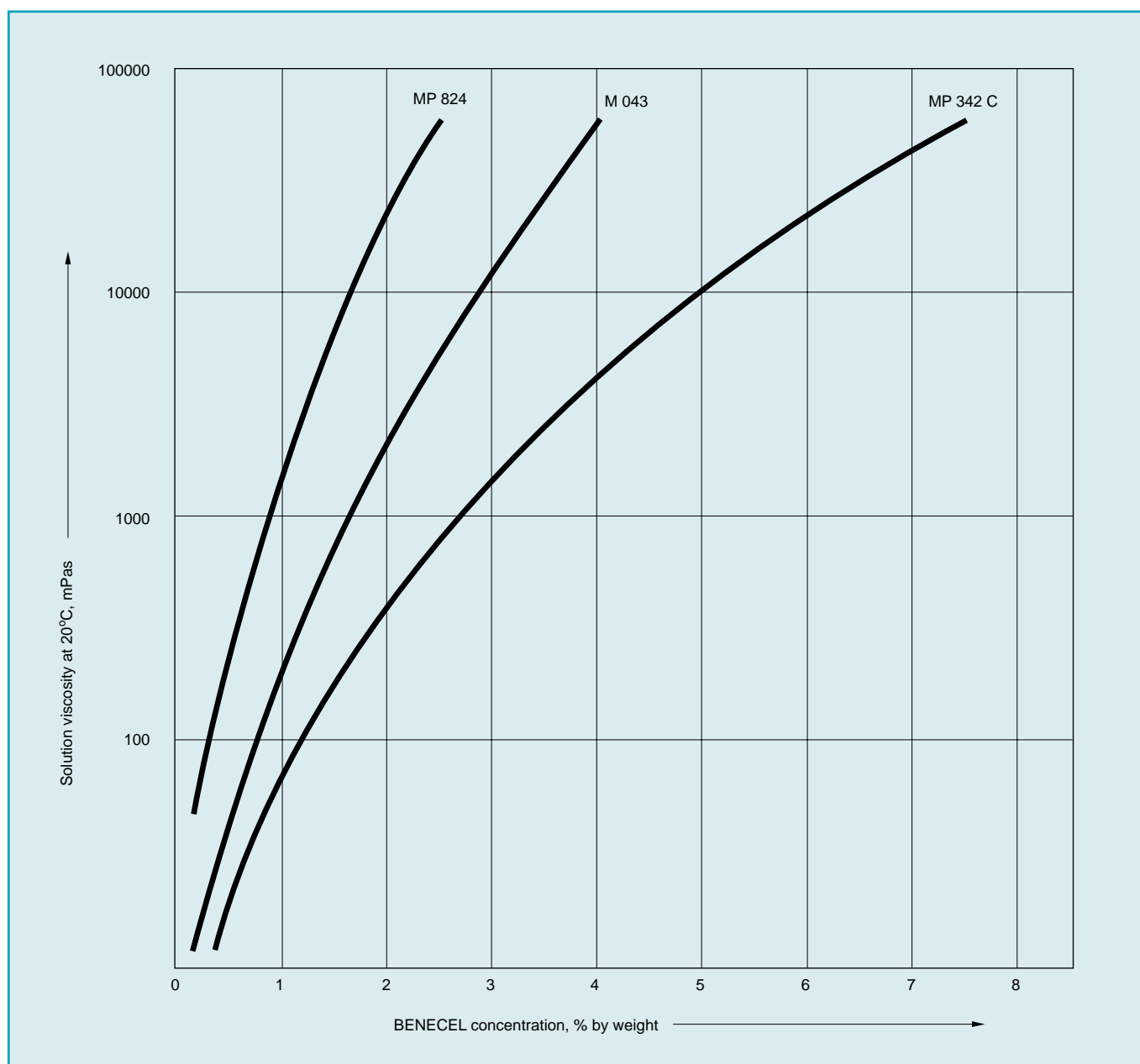
Solution viscosity 2%, 20°C, mPa.s	Intrinsic viscosity η dl/g	Average molecular weight, Dalton	Average degree of polymerisation, DP
10	1,4	13 000	70
40	2,05	20 000	110
100	2,65	26 000	140
400	3,9	41 000	220
1500	5,7	63 000	340
4000	7,5	86 000	460
8000	9,3	110 000	580
15000	11,0	120 000	650
19000	12,0	140 000	750

Fluctuations in the production process cause solution viscosities of cellulose ethers to vary in defined ranges. The viscosity is determined in a 2% aqueous solution at 20°C with the Brookfield rotational viscometer, type RV, at 20 rpm.

The viscosity of aqueous BENECEL® solutions increases exponentially with the concentration of cellulose ether.

This increase in viscosity is particularly pronounced for BENECEL types with high molecular weight, as shown in figure 6. The maximum concentrations, used in practice are between 2-3% for high-molecular weight cellulose ethers and between 5-8% for low-molecular weight products.

Figure 5.
Viscosity/concentration relationship for some BENECEL types.



RHEOLOGY OF BENECEL® SOLUTIONS

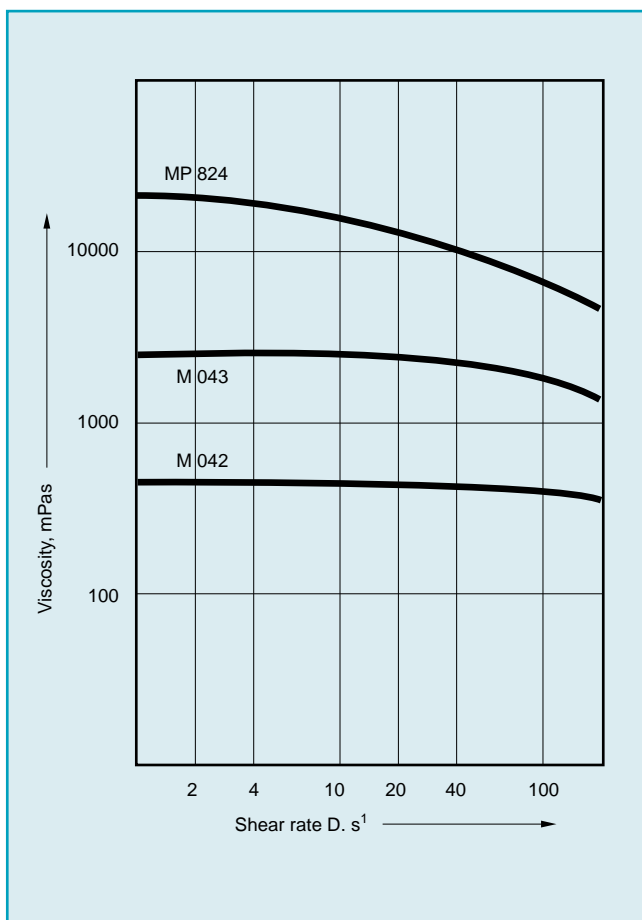
BENECEL solutions behave like non-Newtonian liquids being pseudoplastic. The rheological properties of the solutions are affected by the degree of substitution, the molecular weight and concentration of the cellulose ether.

Pseudoplasticity

The viscosity of pseudoplastic solutions decreases with an increase in shear rate. When shearing is stopped the original viscosity is instantaneously regained. Pseudoplasticity increases with the molecular weight of the cellulose ether and with the concentration of the solution see figure 7. Solutions of low molecular weight BENECEL behave almost like Newtonian fluids.

Figure 6.

Relationship between viscosity of 2% BENECEL solutions and shear rate.



Thixotropy

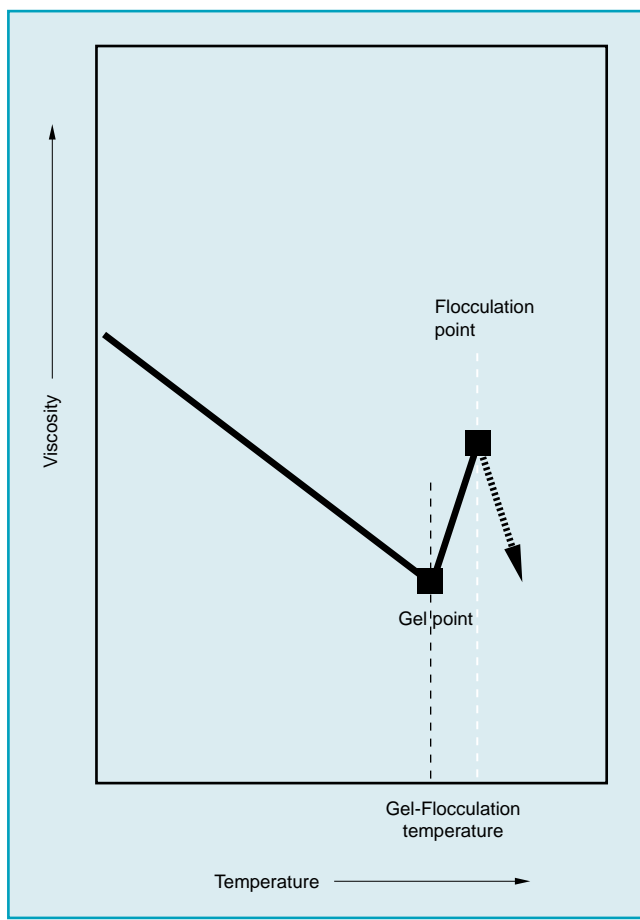
Thixotropic systems are solutions with structural viscosity that, after removal of shear, regain their original viscosity after a certain time. Solutions of BENECEL show little thixotropy only.

EFFECT OF TEMPERATURE ON BENECEL SOLUTIONS

The solution viscosity of cellulose ethers is dependent on the temperature. As the solution temperature increases, the viscosity initially decreases. Once the gelling temperature has been reached, the viscosity sharply rises until the flocculation temperature is reached. See figure 7.

Figure 7.

Viscosity curve at increasing temperature up to flocculation temperature.



The flocculation temperature is dependent upon type of substituent, degree of substitution and on solution concentration. See figure 9.

This also applies to the gelation process as illustrated in figure 10. Upon heating, BENECEL® starts to become less soluble in water and will build up a gel. The resulting gel strength and the gel temperature is determined by its chemistry and concentration in solution.

The flocculation temperature and gelation process is strictly reversible; upon cooling BENECEL will redissolve again.

The presence of salts will reduce flocculation and gel temperature, whereas alcohols such as methanol and ethanol will lead to an increase in flocculation.

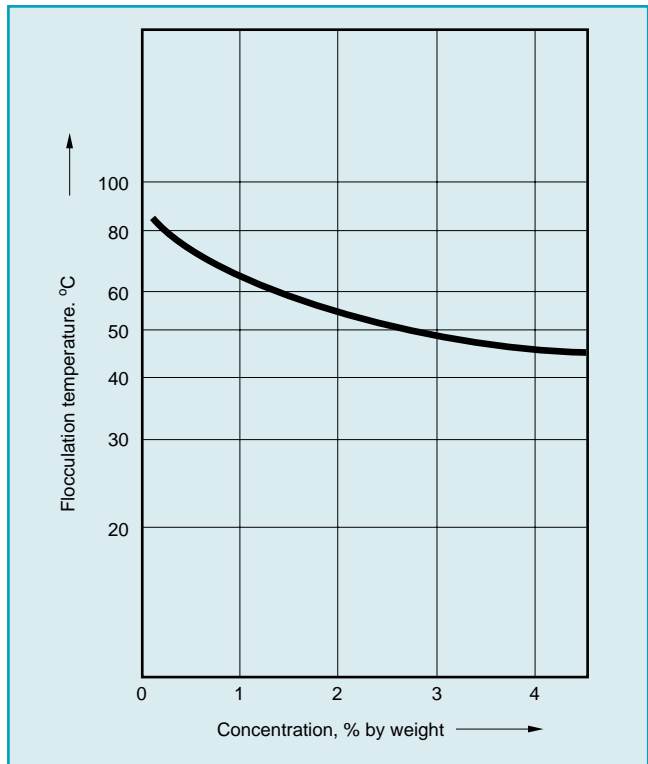
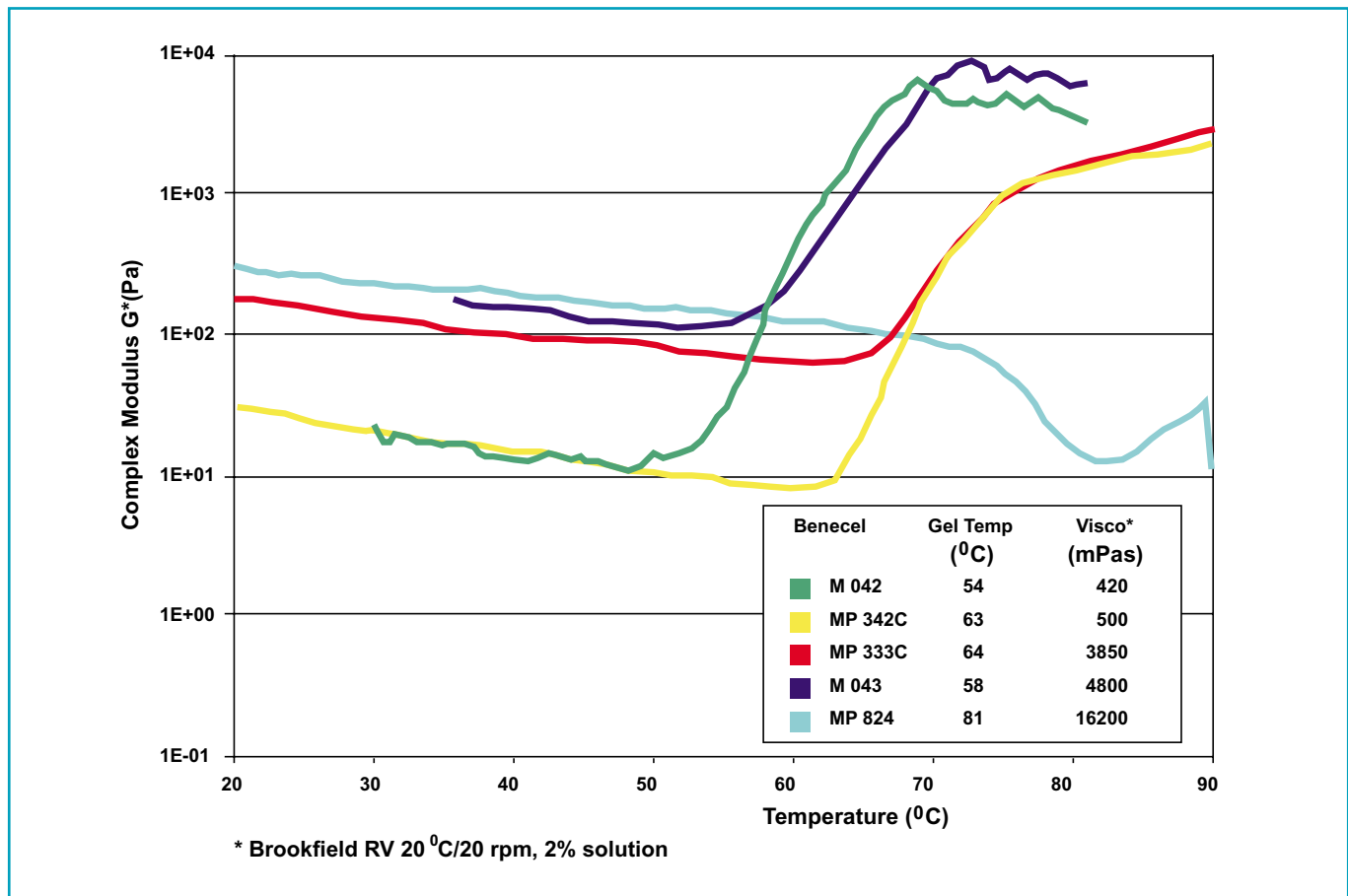


Figure 8.
Influence of concentration on the flocculation temperature of BENECEL.

Figure 9.
Gelcurve of different cellulose ethers (4% solutions).



pH STABILITY

The viscosity of BENECEL® solutions is nearly independent of the pH over a wide range. In strongly acidic or alkaline systems BENECEL may degrade slowly.

SURFACE AND INTERFACIAL ACTIVITY

BENECEL solutions are surface active or interfacially active depending upon type of substituents and degree of substitution. Because of this surface active properties, solutions of BENECEL tend to foam.

The influence of the type of substituent on surface and interfacial tension of aqueous BENECEL solutions is demonstrated in the following table (0,1% aqueous solutions at 20°C; water for comparison).

	Surface tension, mN/m	Interfacial tension versus paraffin oil, mN/m
water	72	45
M	50 – 55	18 – 21
MP	46 – 51	17 – 18.5

To avoid foam formation, BENECEL solutions can be treated with defoamers.

COMPATIBILITY OF BENECEL

BENECEL products are compatible with many materials. Foreign substances, nevertheless, affect the solubility of BENECEL, as with all hydrocolloids.

Influence of salts

Compatibility with salt depends on type and concentration of the salt, and on type and concentration of BENECEL. Low salt concentrations have little effect on viscosity. If a specific salt concentration is exceeded BENECEL is salted out, this is reversible by dilution, see figure 10.

Influence of other water-soluble polymers

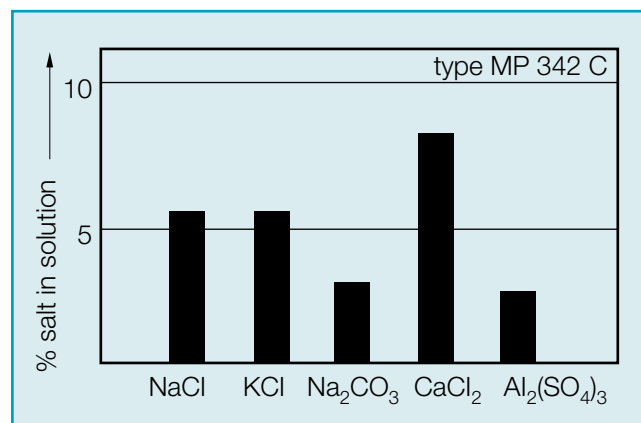
BENECEL is compatible with solutions of most other water-soluble polymers, such as AQUALON/BLANOSE® CMC*, starch, guar, karaya and locust bean gum, carrageenan, pectin and alginates. A synergistic increase of viscosity will be observed upon addition of CMC or xanthan.

PRESERVATION

Solid BENECEL is highly resistant to microorganisms. However, preservation of BENECEL solutions is recommended, if they are to be stored for a longer period, or are mixed with non-sterile additives. Examples of preservatives are the esters of p-hydroxy benzoic acid, benzoic acid, sodium benzoate, sorbic acid and potassium sorbate.

Manufacturers' instructions for use of preservatives should be carefully observed, as well as regulatory restrictions of its use.

Figure 10.
Salt compatibility of 2% BENECEL solutions.



* AQUALON and BLANOSE are both registered tradenames of Hercules' Cellulose Gums
AQUALON CMC is produced in the USA and BLANOSE CMC is produced in France

THE EFFECTS OF BENECEL®

BENECEL cellulose ethers have a great number of valuable properties, which find application in numerous industrial areas. Below is a list of these properties.

THICKENING ACTION

The viscosity of various aqueous, aqueous-organic, and organic solutions, slurries, and dispersions can be adjusted to the desired level by adding only a small amount of BENECEL.

RHEOLOGY

The rheology and consistency of solutions are controlled either thru the molecular weight of BENECEL or with its concentration.

FILM FORMATION

Films with high transparency and tensile strength can be prepared from BENECEL solutions. These films are soluble in water and resistant to most organic solvents, fats and oils.

SUSPENDING ACTION

Depending on the type of substituent, the degree of substitution and the solution viscosity BENECEL is a suitable suspending agent.

WATER RETENTION

High viscosity types of BENECEL exhibit excellent water retention. This property is beneficially used for preventing syneresis. The water retention capacity is proportional to the concentration and solution viscosity of the BENECEL-type used.

BINDING ABILITY

Due to their polymeric structure BENECEL cellulose ethers have good binding ability.

CONTROLLED RELEASE PROPERTIES

Like other Aqualon cellulose ethers, BENECEL when brought in contact with aqueous systems, forms stable gels that dissolve very slowly only. Therefore BENECEL is used as a sustained release agent in matrix tablets.

The use of MHPC has been described in several patents along the recent years.

SURFACE ACTIVITY

Depending on the type of substituent and degree of substitution, BENECEL cellulose derivatives are suitable as suspension- and emulsifying agents and as protective colloids due to their surface activity.

STABILIZING ACTION

By adding small amounts of BENECEL the stability of emulsion systems is improved.

GELATION

When heating solutions of BENECEL, gelation will occur at temperatures above 50°C. This gelation is beneficial to a number of food preparations, like the control of fat pick-up during deep-frying, and the control of texture of foam-aerated products.

APPLICATIONS

The most important areas of application for BENECEL® cellulose ethers are:

Food

BENECEL products are metabolically inert and have a neutral taste and odor. Because they offer combinations of many functional and some very specific properties BENECEL is used in a variety of food products. These include bakery products, to gain volume, texture and longer freshness by the incorporation of BENECEL in the paste or dough.

When frying deep frozen products like extruded croquettes BENECEL helps to retain their shape, and improves the adhesion of the paste to the meat, fish or shrimp filling. The heat-gelling property of BENECEL effectively helps to reduce fat pick-up.

The surface activity and water retention of BENECEL in solution explain its use in emulsion-type sauces, and in whipped toppings and creams.

Other food products that make use of the properties of BENECEL are ice-cream, low-calorie products, instant products, bakery creams.

	EC E461, E464	European Pharma- copoeia	United States Pharmacopeia (HPMC = MHPC)*
Heavy metals total, ppm max	20	20	10
- ppm mercury, max.	1		
- ppm cadmium, max.	1		
- ppm lead, max.	5		
- ppm arsenic, max.	3		3
% chloride, max.		0.5	
% sulphated ash, max.	1.5 MHPC ≤50 mPa.s: <3.0	1.0	
% residue on ignition, max.			MC : 1.5 HPMC 1828 : 5.0 HPMC <50 mPa.s : < 3.0 HPMC others : 1.5
% loss on drying	10	10	5
pH, 1% solution	5 - 8	5.5 - 8	5 - 8
Molecular weight	MC 20000 - 380000 MHPC 13000 - 200000		
Compliance with GMP	yes	yes	yes
% viscosity range of specified viscosity	-	75 - 140	80 -120 visc.<100 mPa.s: 75 - 140
Substitution range	MC: 25 - 33% methoxyl max 5% EOOH MHPC 19-30% 3 - 12% POOH		MC: 27.5 - 31.5% HPMC: % methoxyl/POOH type 2208 : 19 - 24/4 - 12 type 2906 : 27 - 30/4 - 7.5 type 2910 : 28 - 30/7 - 12 type 1828 : 16.5 - 20/23 - 32
for HPMC only propylenechlorohydrin	< 0.1 ppm		

*) See also page 3

Pharmaceutical industry

When formulating solid or liquid dosage forms, the functional properties of BENECEL® products are extremely interesting to the pharmacist.

BENECEL are excellent binders and film formers when used as granulation and film coating agents. They also control the release of the active ingredients in matrix tablets. Their use for parenteral applications is not recommended.

Cosmetic and personal care industry

The same balance of functional properties and characteristics make the BENECEL products of great interest when formulating for the hair care, skin care and other toiletry products. BENECEL products are used in shampoos, emulsions, creams and lotions.

BENECEL products are also used in specific medical applications.

Legal rights and regulations

The protective rights of third parties must be taken into consideration upon application of BENECEL products. The use of BENECEL in pharmaceutical, cosmetic, food and food-related products should comply with the respective regulations which apply in many countries.

APPENDIX

QUALITATIVE ANALYSIS OF BENECEL CELLULOSE ETHERS

Qualitative identification tests on BENECEL products include precipitation with tannin, heat coagulation, the diphenylamine test, the reaction on hydroxyalkyl groups with sodium nitroferricyanide and the Ninhydrin test.

QUANTITATIVE DETERMINATIONS

Moisture content

A quantity of 10 g cellulose ether is carefully weighed, and dried at 105 - 110°C until its weight remains constant. The amount of moisture is the quotient of the loss in weight and the original weight, given as a percentage.

Recommended viscosity ranges of spindles

Spindle	Range, mPa.s
1	20 – 350
2	350 – 1450
3	1450 – 3500
4	3500 – 7000
5	7000 – 15000
6	15000 - 45000
7	>45000

BENECEL viscosity procedure

The viscosity of BENECEL is measured on the basis of dry product. Readings obtained are converted into mPa.s, the value of the latter equals that of the former cps.

1. Equipment

- Brookfield RVT rotary viscometer (Brookfield Inc., Stoughton Mass., U.S.A.). For viscosities below 20 mPa.s the Brookfield UL-adapter should be used. Readings are taken at 20°C and 20 rpm
- Mechanical stirrer IKA RW 18 or RW 20 (Janke & Kunkel, Staufen, W-Germany) with 4-blade impeller stirrer (diameter: blades 50 mm, shaft 5 mm)
- Analytical balance, accuracy $\pm 0,01$ g
- 600 ml beaker, low form

For viscosities below 350 mPa.s: 1000 ml beaker, low form.

- Thermostat (water bath) at 20°C
- heating plate
- watch glass (glass cover)

Only deionized water is used for make-up.

Moisture content of BENECEL has to be determined in advance

2. Concentration of BENECEL® solutions

2%-solutions are standard. For highly viscous products with a 2%-viscosity higher than 20000 mPa.s a 1%- solution should be prepared. For low viscosity products with 2%-solution below 350 mPa.s a 4%-solution should be used additionally for viscosity measurements.

3. Make-up of solutions

Solutions for granular, retarded and powdered BENECEL-types are prepared differently.

For a 2%-solution:

10 g BENECEL as is, $10 \times \frac{(98\% \text{ moisture})}{2}$ g water

600 ml beaker.

If 2%-viscosity below 350 mPa.s:

16 g BENECEL as is, $16 \times \frac{(98\% \text{ moisture})}{2}$ g water

1000 ml beaker.

For a 1%-solution:

5 g BENECEL as is, $5 \times \frac{(99\% \text{ moisture})}{2}$ g water

600 ml beaker.

For a 4% solution:

32 g BENECEL as is, $32 \times \frac{(96\% \text{ moisture})}{2}$ g water

1000 ml beaker.

Stirring rate

The stirring rate has to be adjusted to get maximum possible mixing without introducing too many air bubbles into the solution.

3.1 BENECEL C-types

- Add total amount of water (temperature approx. 18°C) to beaker;
- add BENECEL within 30 s under stirring;
- stirring continues for 30 minutes;
- thermostat solution at 20°C for 2 hours;
- before viscosity reading, stir solution with IKA stirrer during 3 minutes.

3.2 BENECEL Standard

- One fifth of the required amount of water is heated to 80-90°C. Cover beaker with watch glass;
- add BENECEL within 30 s under stirring;
- add rest of water that has a temperature of 5°C; stirring continues for 30 minutes;
- BENECEL M is subsequently cooled to 10°C during one hour.
- thermostat solution at 20°C for 2 hours;
- before viscosity reading, stir solution with IKA stirrer for 3 minutes.

Sodium chloride content

The sodium chloride content is calculated from the amount of chloride determined. The chloride determination can be carried out according to the Mohr or Volhard method.

Mohr method: The cellulose ether is weighed and dissolved in distilled water, mixed with 3 drops of a 25% solution of potassium chromate and titrated with 0,1 N silver nitrate solution. At the end point the colour changes from yellow into rust brown.

Volhard method: The cellulose ether solution is acidified with 2 N nitric acid, mixed with an excess of a 0,1 N silver nitrate solution, then titrated with 0,1 N ammonium thiocyanate using ferric ammonium sulfate as an indicator from colourless to weak rust brown.

With highly-viscous cellulose ethers it is best to ash a weighed amount of test sample at 600°C and then subject the ash to the chloride test.

Sulfated ash

2 g of cellulose ether are ashed at 600°C to a constant weight with 5 ml of 98% sulfuric acid. The residual sodium sulphate is weighed and related to the original weight (expressed as a percentage).

Ash content

The weighed sample is ashed in a porcelain or platinum crucible at 600°C to constant weight. The ash content is expressed in percent and is the ratio between the ash and the original weight.

Degree of substitution

Cellulose ethers are subjected to a modified Zeisel ether cleavage at 140°C with hydroiodic acid. The resulting products can be determined quantitatively with a gas chromatograph. The determination of the degree of substitution is also possible by nuclear magnetic resonance (N.M.R.) measurements.

Screen analysis

This test is carried out with an air-stream sieve; for example, the Alpine type A 200 LS, at 150 mm water column vacuum with 20 g of sample for 3 minutes.

The use of shaking screens may give incorrect results, especially in the fine particle size region due to electrostatic charging of the cellulose ethers.

Flocculation point

The determination of the flocculation point is based on the turbidity measurement. A 0,5% solution of cellulose ether is heated at a rate of 2°C per minute. Temperature and turbidity of the solution are registered with a two-channel recorder.

The flocculation point is the temperature at which the turbidity curve goes through a turning point. For flocculating cellulose ethers the turbidity curve forms an irregular pattern starting at the flocculation point.

High viscosity types of BENECEL® are tested as 0,25% solutions.

Surface/interfacial tension

The surface or interfacial tension can be measured with the ring method according to Du Noy on 0,1 % cellulose ether solutions at 20°C. Low viscosity types are tested at a concentration of 0,5 to 2%.

Determination of gel/fiber content

2 g of cellulose ether are dissolved in 98 g of distilled water with agitation. Then 100 ml of a 2% aqueous sodium chloride solution and 5 drops of a 0.75% Congo red solution (solvent: water/ethanol = 30/70) are added. After stirring the sample for 1 hour, 33 g of the coloured solution are poured into a centrifuge tube, which has a capillary at its bottom, and centrifuged for 15 minutes at 3200 rpm (2000 g). The height of gel particles and fibres, which are settled as a coloured layer in the capillary is measured and expressed in mm.

Turbidity

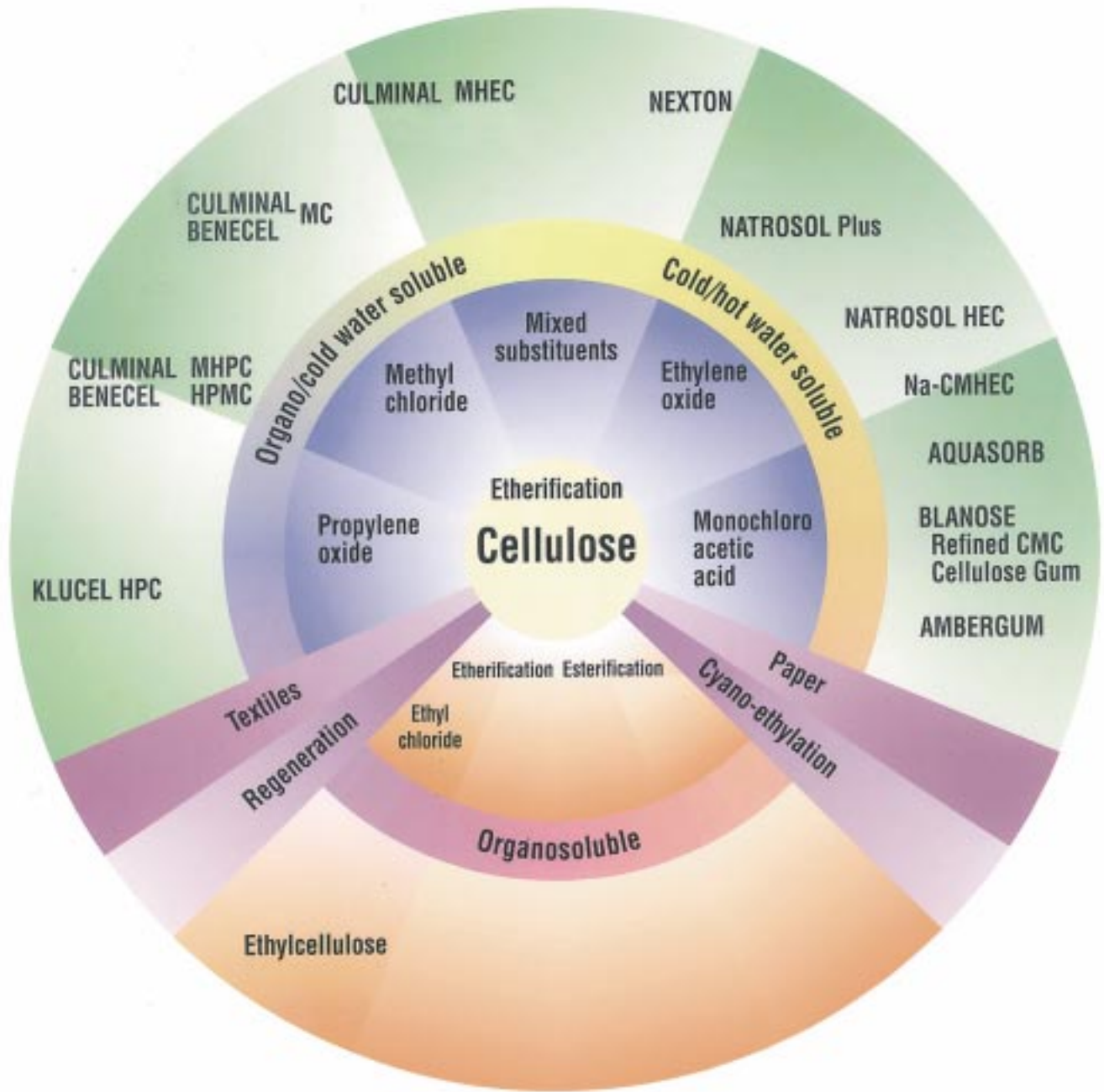
The turbidity of 2% solutions of BENECEL is determined using commercial turbidity measuring equipment. These function according to the light transmission principle and measure the amount of light which is diffused or transmitted. The turbidity is the ratio between diffused light and incident light, expressed in percent.

PACKAGING AND STORAGE

BENECEL is supplied in multiply paper bags with polyethylene inner liner, with net weights of respectively 18 kgs, 18,14 kgs (40 lbs) and 20 kgs.

BENECEL is a non-perishable product. It is recommended to use the product in rotation on a first-in first-out basis. The product should be stored under dry and clean conditions in its original packing and away from heat. The product is hygroscopic. The packaging is selected in a way to avoid ingress of moisture, but the water content of the packed product will/may increase if not stored dry.

Figure 11.
Cellulose and its derivatives (Sun)



**For further information please visit
our website www.aqualon.com or
contact any of the following offices**

Hercules Incorporated
Aqualon Division
Hercules Plaza
1313 North Market Street
Wilmington, DE 19894-0001
USA
Tel. : +1 302 - 594 5000
Fax : +1 302 - 992 7287

Hercules International Limited LLC
Aqualon Division
Veraartlaan 8
NL-2288 GM Rijswijk
P.O.Box 5822
NL-2280 HV Rijswijk
THE NETHERLANDS
Tel. : +31 70 - 413 4341
Fax : +31 70 - 319 1187

Hercules GmbH,
Aqualon Division
Paul-Thomas-Strasse 56
D-40599 Düsseldorf
GERMANY
Tel. : +49 211 - 749 10
Fax : +49 211 - 749 1200

Hercules Moscow
1-st Tverskaya-Yamskaya, 23
Business Centre "Parus"
125047 Moscow
RUSSIA
Tel. : +7 095 - 931 9547 (-49)
Fax : +7 095 - 931 9546

Hercules CZ spol s.r.o
Na Výspě 22
CZ-147 00 Prague 4
CZECH REPUBLIC
Tel. : + 420 2 - 4400 1295
Fax : + 420 2 - 4447 1912

Hercules Polska Sp. z o.o
ul. Migdalowa 4
PI-02-796 Warsaw
POLAND
Tel. : +48 22 - 645 1286/7
Fax : +48 22 - 645 1289

Hercules Italia S.p.A.,
Aqualon Division
Via Bondanello 15
P.O. Box 1726
I-40013 Castelmaggiore (Bologna)
ITALY
Tel. : +39 051 - 715 333
Fax : +39 051 - 714 734

Hercules Chemicals Singapore Pte. Ltd
Aqualon Division
200 Pandan Loop
#07-01 Pantech 21
SINGAPORE 128388
Tel. : +65 6775 5366
Fax : +65 6776 9690

Hércules Química S.A.,
Aqualon Division
Carretera de Valencia, 5/9
P.O. Box 32
E-43080 Tarragona
SPAIN
Tel. : +34 977 - 558 800
Fax : +34 977 - 558 814

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