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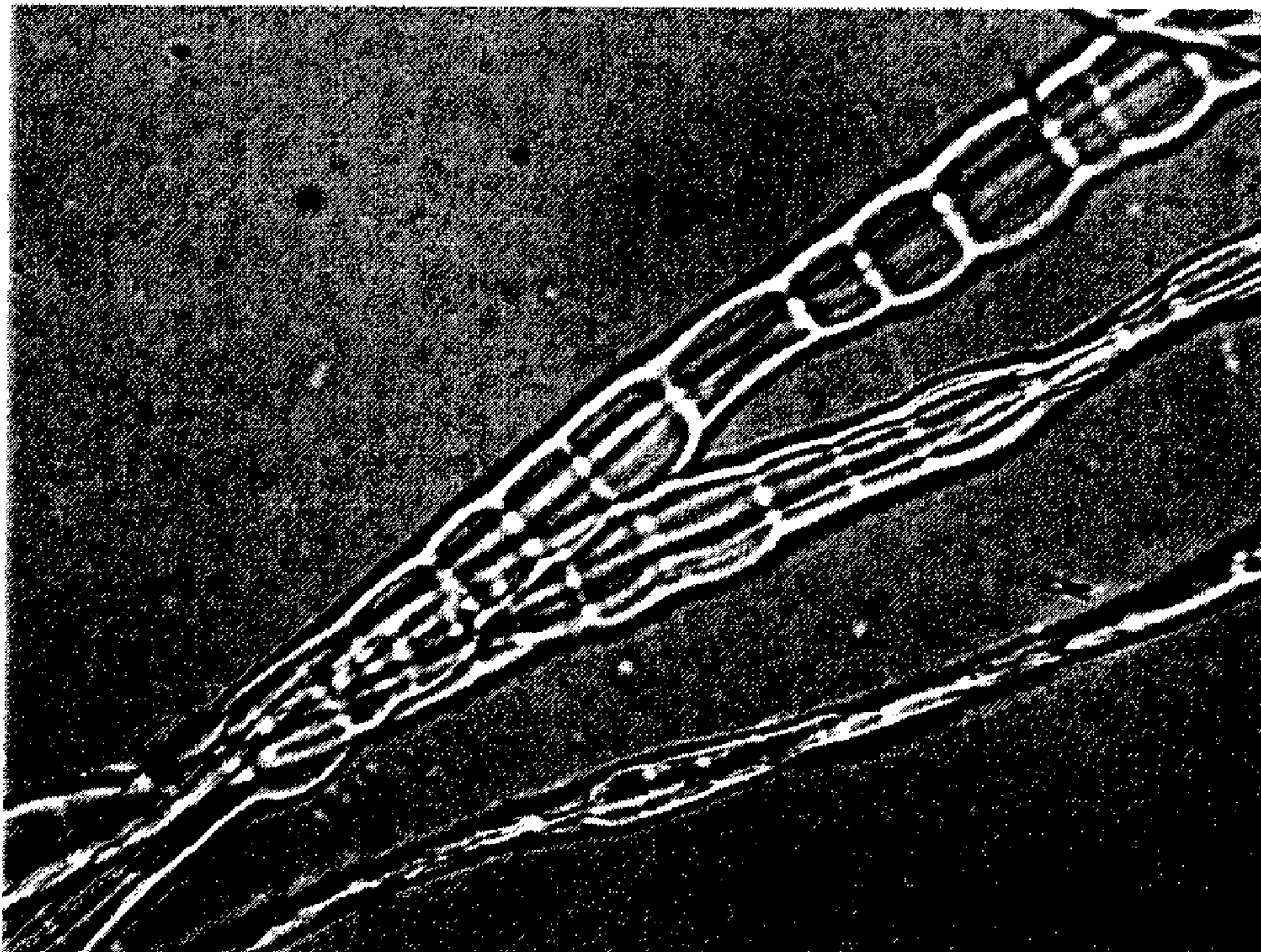
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(54) Title: REGENERATED CELLULOSE FIBRE



(57) Abrégé/Abstract:

The present invention relates to a regenerated cellulose fibre, which is characterized by the combination of the following features:
- the fibre has in its dry condition a collapsed hollow cross-sectional structure - the fibre has in its wet condition a cross-sectional structure with cavities - the fibre is segmented in the longitudinal direction by dividing walls - there is incorporated into the fibre an absorbent polymer, in particular carboxymethylcellulose. The fibre may be obtained by a process, wherein there is admixed a carbonate as well as an absorbent polymer, in particular carboxymethylcellulose, to a viscose dope.

Abstract:

The present invention relates to a regenerated cellulose fibre, which is characterized by the combination of the following features:

- the fibre has in its dry condition a collapsed hollow cross-sectional structure
- the fibre has in its wet condition a cross-sectional structure with cavities
- the fibre is segmented in the longitudinal direction by dividing walls
- there is incorporated into the fibre an absorbent polymer, in particular carboxymethylcellulose.

The fibre may be obtained by a process, wherein there is admixed a carbonate as well as an absorbent polymer, in particular carboxymethylcellulose, to a viscose dope.

Regenerated cellulose fibre

The present invention relates to a regenerated cellulose fibre prepared by the viscose process.

In particular the invention relates to a regenerated cellulose fibre with a collapsed hollow cross-sectional structure.

For sanitary uses such as, for example, tampons or absorbent cores in general, fibres are desirable, which have an especially high water storing capacity in order to in this way allow for a possibly high absorption capacity of the sanitary product.

Conventional super-absorbent materials have the disadvantage that they usually form a gel upon contact with water and thus reduce the integrity of the absorbent core and, furthermore, block the further transport of liquid through the absorbent core. The integrity of the absorbent core, hence, is important in these applications, as there is prevented, by means of this material, the exit of the absorption material and thus the contamination of, for example, wounds.

It is also desired for wound dressings that the absorbing material does not agglutinate with the wound so that it is achievable to remove the dressing from the wound without causing pain.

Another demand for the fibre material in these fields of application is the flawless processability in conventional production techniques for the fleece production.

Viscose fibres with a high portion of carboxymethylcellulose (CMC) have been known. This is a mixed fibre that is obtained by spinning in carboxymethylcellulose into the viscose dope. Such fibres have also been commercially produced (US 4,199,367 A, US 4,289,824 A).

The production of viscose fibres with a hollow cross-sectional structure ("hollow fibres") by spinning in sodium carbonate into the viscose dope has already been known since 1920. Fibres of this type were previously commercially produced by various producers. Hollow fibres of the "superinflated" type with high water retention capacity have been produced especially for sanitary applications (GB 1,333,047 A, GB 1,393,778 A).

A comprehensive survey on development and history of hollow viscose fibres may be found with: C.R. Woodings, A. J. Bartholomew; "The manufacture properties and uses of inflated viscose rayon fibres"; TAPPI Nonwovens Symposium; 1985; pp. 155-165.

Viscose-CMC mixed fibres show in their production the disadvantage that due to their high absorbance and the strong adherence of the fibres to each other in a wet condition (gel effect on the fibre surface) the fibres in the conventional viscose process, from an integration of about 15% CMC based on the cellulose on, tend to sink in the baths of the subsequent treatment and not to float like conventional viscose fibres. This substantially hinders the production of these fibres in a common commercial process.

A desired effect of these fibres is their gel-like surface consistency in a wet condition. This effect in touch, however, is partly reduced by the fact that these fibres have an extremely grooved surface structure – like conventional viscose fibres.

The maximum increase of the water retention capacity of such fibres is restricted, as during the spinning in of >50 % CMC there arise great problems in the processing step thereof.

Also with hollow viscose fibres the increase of the water retention capacity is limited. Due to a stronger inflation of the fibre, there will not be obtained any higher water retention capacity anymore from a certain level on, and there are rather formed band-like fibres with a flat cross-section having low water retention capacity, wherein the post-treatment thereof in a staple fibre method is rather associated with problems due to their large and smooth surface and the high fibre-fibre binding capacity associated therewith.

There are further known carboxymethylated fibres, i.e. fibres that have been produced by subsequent carboxymethylation of viscose fibres (commercially available as "Aquacell™" fibres of the company Convatec; US 6,548,730 B1; AU 757461 B; WO00/01425 A1; EP 1 091 770 A1). The fibres allow for a high amount of water absorption but form upon contact with water a gel with simultaneous complete loss of the fibre structure, which is not desirable for all possible applications.

The US 3,318,990 describes a method for the production of flat viscose fibres, wherein there is admixed to the viscose, on the one side, sodium carbonate and, on the other side, a high-molecular substance swelling in water, e.g., CMC.

The resulting fibres are described as completely flat. The collapsed cross-sectional structure retains its form also in a wet condition. The fibres are suitable for the production of paper according to US 3,318,990.

The present invention has the objective to provide a viscose fibre with a possibly high water retention capacity, a possibly high absorption under pressure and an additional surface gel effect. The fibre is to be produced in the conventional methods for the production of viscose fibres and to be processed according to the conventional method for the production of fleece (for example, carding, solidification by needling, hydro-entanglement, thermal solidification, calandration).

This task is solved by a regenerated cellulose fibre as described herein. The process described herein serves for the production of the cellulose fibre according to the invention. Preferred embodiments are described herein.

SHORT DESCRIPTION OF THE FIGURES

Figure 1 shows the longitudinal section of a dried cellulose fibre according to the invention. Figure 2 shows a much enlarged longitudinal section of a dried cellulose fibre according to the invention.

Figure 3 shows the cross-section of dried cellulose fibres according to the invention.

Figure 4 shows the tubular cross-section of a cellulose fibre according to the invention upon contact with water.

DETAILED DESCRIPTION OF THE INVENTION

The present invention provides a regenerated cellulose fibre with a collapsed hollow cross-sectional structure, which is characterized by the combination of the following features:

- the fibre has in its dry condition a collapsed hollow cross-sectional structure
- the fibre has in its wet condition a cross-sectional structure with cavities
- the fibre is segmented in the longitudinal direction by dividing walls
- there is incorporated into the fibre an absorbent polymer.

The absorbent polymer is preferably carboxymethylcellulose (CMC). Other suitable absorbent polymers may be selected from the group of polymers that are compatible with the

viscose process, such as, for example, viscose carbamate, carboxymethylcellulose, alginates or homo- and co-polymers of acrylic acids (such as, for example, described in US4399255).

By means of the present invention, there is, for the very first time, provided in the preferred embodiment a combination of a hollow viscose fibre, which has a tubular structure with cavities again upon re-wetting, and a viscose mixed fibre with CMC.

The expert understands that a regenerated cellulose fibre with collapsed hollow cross-sectional structure is a viscose fibre, the cross-section of which has been so strongly inflated that the cross-sectional structure collapses in itself. The production of such fibres (through admixture of sodium carbonate) is described in GB 1,333,047 A and GB 1,393,778 A.

The fibre according to the invention has in its dried condition preferably an irregular multilobal cross-section or an irregularly grooved cross-section. Such fibres are also designated as "SI" fibres (for superinflated). In contrast to the fibres described in the US 3,318,990 A the cross-section will re-open upon wetting of the fibres and it will regain its tubular form, with cavities being formed.

The fibre according to the invention is different to the fibres described in US 3,318,990 A in particular due to the presence of a segmentation in the longitudinal direction as a result of dividing walls.

The distance between the dividing walls thus is preferably between 0.3 and 3 times, preferably 0.5 to 2 times, the fibre width averaged over the fibre length. There is assumed that the presence of these dividing walls is in part responsible for the fact that upon wetting of the fibres, there is re-formed a tubular structure with cavities.

This tubular structure is probably responsible for the fibres according to the invention having excellent absorbance compared to the fibres of the US 3,318,990 that remain flat also upon re-wetting.

By the measure that there is incorporated an absorbent polymer, in particular CMC; into the fibre the expert understands as being integrated, in the matrix of the (upon regeneration of the fibre) underivatized cellulose, the absorbent polymer, for example CMC. This is – in contrast to the application of CMC onto the already fabricated fibre – possible, especially, by spinning in CMC into the viscose dope.

The cellulose fibre according to the invention has a very high water retention capacity; the fibre character, however, still remains the same in its wet condition. Furthermore, there has been shown that the fibre according to the invention, in contrast to conventional viscose-CMS mixed fibres, may be well produced and in particular processed in a conventional viscose process.

The portion of absorbent polymer, in particular of CMC, in the fibre according to the invention is preferably 5 to 50 % by weight, especially preferably 15 to 40 % by weight, most preferably 20 to 30 % by weight, based upon the underivatised cellulose.

The cellulose fibre according to the invention is preferably available in the form of a staple fibres and may be produced in all common titre areas. Preferably, the fibre titre may be 0.5 dtex to 8 dtex, preferably from 1.3 to 6 dtex.

The fibre length of the fibre according to the invention may be from 2 mm to 80 mm and is dependent in particular on the field of application. For a wet-laid process, there are suitable in particular fibre lengths of 2 to 20 mm, for a carding process, fibre lengths from 20 to 80 mm are suitable.

The tenacity of the fibre according to the invention is typically above 10cN/tex; the tear elongation is above 15%.

The cellulose fibre according to the invention has preferably a water retention capacity of at least 300%, measured according to DIN 53814.

There was, for example, measured in the fibres according to the invention with a portion of 35 % by weight CMC a water retention capacity of up to 400% WRC.

A cylindrical tampon prepared from the fibre according to the invention with a mass of 2.72 g, a length of 44 mm and a diameter of 13 mm has preferably a Syngina value of at least 5.2 g/g, measured according to EDANA / INDA Standard Test Methods for the Nonwovens and Related Industries ERT 350.0 or WSP 350.1, respectively.

In this way, the fibre has a higher Syngina value than the currently commercially most important absorbent viscose fibre, a viscose fibre with a regular trilobal cross-section marketed under the trade name "Galaxy"® (EP 0 301 874 A1).

For the production of a regenerated cellulose fibre according to the present invention, there is used a process, comprising the steps

- providing a viscose dope
- admixing a carbonate compatible with viscose into the viscose dope
- admixing of an absorbent polymer into the viscose dope
- spinning the viscose dope in a spinning bath, thereby forming fibres,

wherein

- the degree of ripening of the viscose dope before spinning is less than 15° Hottenroth, preferably 10° to 14° Hottenroth
- the content of H_2SO_4 in the spinning bath is 8% to 10%
- the content of ZnSO_4 in the spinning bath is 0.3% to 0.5% and
- the content of Na_2SO_4 in the spinning bath is 25% to 30%.

The process according to the invention, hence, combines the so far known step of spinning in a carbonate, in particular sodium carbonate, for producing a hollow cross-section, with the step of spinning in an absorbent polymer.

In comparison with the method described in US 3,318,990, there is spun in the process according to the invention a viscose with a lower degree of ripening (expressed in Hottenroth) and the spinning bath has a smaller portion of H_2SO_4 and ZnSO_4 . There is made the assumption that these different procedures result in the differences mentioned above between the fibre according to the invention and the fibre described in the US 3,318,990, in particular in terms of cross-sectional structure and also in terms of the other characteristics.

The absorbent polymer is preferably CMC. The following embodiments relate to this preferred embodiment; they apply, however, *mutatis mutandis* also to other absorbent polymers that are compatible with the viscose process.

As carbonate, there is suitable any carbonate, in particular an alkali metal carbonate, which is compatible with the conditions of the viscose process. Especially suitable is sodium carbonate. Other suitable carbonates are potassium carbonate, calcium carbonate as well as in general all carbonates releasing carbon dioxide under the influence of an acid.

Exerting influence on the cross-sectional form of the fibre (inflation and collapse) in a way so that there is developed a collapsed hollow cross-sectional structure, for example, by selecting the appropriate conditions in the spinning bath, selection of temperature and, of course, amount of carbonate added, is known to the expert in particular from the patent publications cited above.

Preferably, the carbonate is admixed in an amount of 11 % by weight to 23 % by weight (calculated as $(\text{CO}_3)^{2-}$), based on the cellulose in the viscose dope. In the specific case of sodium carbonate, the preferred amount is from 20 % by weight to 40 % by weight based on cellulose.

Further preferably, the absorbent polymer, in particular the carboxymethylcellulose, is admixed in an amount of 5 % by weight to 50 % by weight, especially preferably 15 % by weight to 40 % by weight, most preferably 20 % by weight to 30 % by weight, based on the cellulose in the viscose dope.

The carbonate and/or the carboxymethylcellulose may preferably be admixed in the form of a solution.

In particular, the carboxymethylcellulose may be admixed in the form of an alkaline solution, containing 2 % by weight to 9 % by weight, preferably 3 % by weight to 5 % by weight NaOH and 5% by weight to 15 % by weight, preferably 6 % by weight to 12 % by weight, carboxymethylcellulose.

The carbonate and the carboxymethylcellulose may also be admixed together, in particular in the form of a joint solution.

For the production of a cellulose fibre according to the invention there may also be used a conventional viscose dope.

A typical embodiment of the process according to the invention comprises the following measures:

To a viscose dope produced according to conventional methods (cellulose content = 9-10%, NaOH content 5-6%; viscosity 30-50 falling ball seconds, degree of ripening 10-15° Hottenroth) there are added 25-35 % by weight Na_2CO_3 (to be varied according to fibre titre), based on the cellulose in the viscose dope, in the form of a 20% solution of Na_2CO_3 .

There are further added to the dope 5 % by weight to 45 % by weight, preferably 20 % by weight to 30 % by weight, based on the cellulose in the viscose dope, carboxymethylcellulose in the form of an alkaline solution (2-9 % by weight NaOH; preferably 3-5 % by weight). The concentration of the solution is 5 % by weight to 15 % by weight, preferably 8-12 % by weight. The carboxymethylcellulose is a commercially available product with a degree of substitution DS of 0.6 – 1.2, preferably 0.65 – 0.85 and a viscosity (2% solution; 25°C) of 30-800 mPas; preferably 50-100 mPas.

The additive solutions (carbonate and CMC) are preferably injected into the already spinnable viscose dope, in particular immediately before the spinning. The viscose is subsequently homogenized, preferably via a dynamic mixer.

The viscose is spun out and subjected to a post-treatment at parameters common for textile fibres.

Examples:

For the production of regenerated cellulose fibres described in the following there was used a conventional viscose dope.

Experiment A: spinning of the viscose dope without additives

Experiment B: spinning of the viscose dope with 30% Na₂CO₃ based on cellulose

Experiment C (according to the invention): spinning of the viscose dope with 30% Na₂CO₃ and 20% CMC (DS=0.81; viscosity 2% = 53 mPas, Type Blanose 7M1F, producer: company Aqualon France (Hercules)), each based on the cellulose present in the viscose dope.

The dopes were spun out into fibres with a titre of 4.4 dtex with identical spinning parameters.

Experiment D: spinning of the viscose dope without additives

Experiment E: spinning of the viscose dope with 20% CMC (specification as in experiment C) based on cellulose

Experiment F: spinning of the viscose dope with 30% CMC (specification as in experiment C) based on cellulose

The dopes were spun out into fibres with a titre of 3 dtex with identical spinning parameters.

Morphology of the produced fibres

Viewed with a microscope, the fibres B and C are hollow fibres of the SI type (superinflated, segmented). The fibres A, D, E and F are fibres with a grooved and round cross-section that is typical for viscose fibres.

Longitudinal section and cross section of the dried fibre C according to the invention are illustrated in the figures 1, 2 and 3.

In the figures 1 and 2 there is clearly visible the segmentation in the longitudinal direction, which is typical for the fibre according to the invention. The segmentation is the result of dividing walls, which – as may be recognized in figure 2 in an increased magnification – in fact result in a rigid and membrane-like sub-division.

In figure 3 there is visible the collapsed hollow cross-sectional structure, which is typical for "SI" fibres, with the formation of a plurality of sides or irregularly grooved cross-sections.

Figure 4 shows the tubular cross-section of the fibre according to the invention upon wetting of the dry fibre.

Spinning performance:

All fibres may be spun with the conventional parameters. Whereas the fibres E and F tend to deposit in the baths of the post-treatment of the fibres, this effect is not observable in the fibre C according to the invention.

Water retention capacity:

For all fibres, the water retention capacity was measured in % according to DIN 53814.

Fibre A:	80 %
Fibre B:	220 %
Fibre C:	335 %
Fibre D:	93 %
Fibre E:	149 %
Fibre F:	193%

As the results show, the water retention capacity in absolute terms may be increased by spinning in 20% CMC based on the cellulose by about 50%, by spinning in 30% CMC by 100%. (comparison of the fibres D -> E -> F).

By spinning in 30% Na₂CO₃ based on cellulose, the water retention capacity may be increased by 140 %. (comparison of the percentage, fibre A -> B).

By spinning in 20% CMC and 30% Na₂CO₃ based on cellulose (according to the invention), the water retention capacity, however, may be increased by 255%. This shows that the effects of the two additives do not increase additively but rather synergistically:

The effect of the addition of CMC alone or Na₂CO₃ alone, respectively, amounts to:

A->B = +140%

D ->E = +56%

The expected value for the fibre C, hence, would be $80 + 140 + 56 = 276\%$

As the measured water retention capacity of the fibre amounting to 335% is higher by the factor 1.2, the two additive components surprisingly, hence, act in a synergistic way.

Gel effect:

The surface gel effect (slimy and soapy touch) is somehow stronger in the fibres C according to the invention than in the comparative sample E (with the same content of CMC). This gel effect is only developed in a wet condition.

Fibre values:

The fibre C according to the invention obtains at a titre of 4.4 dtex a tenacity of 13.1 cN/tex with a tension of 22.9 %, which is acceptable for a use in the field of fleece processing.

Absorption under pressure:

From the fibres A, B and C there were pressed tampon plugs, each amounting to 2.72g, and there was carried out an Syngina measurement according to WSP 351.0. As an additional reference, there was produced an identically produced plug made of 100% commercial trilobal viscose fibre ("Galaxy"®).

Results:

Fibre A:	4.56 g/g
Fibre B:	4.75 g/g
Fibre C:	5.73 g/g
100% Galaxy:	5.05 g/g

The result shows that with the fibres according to the invention there may be obtained a significantly higher Syngina absorption even in comparison with commercial trilobal fibres.

The regenerated cellulose fibre according to the invention, hence, is especially suitable for the use in absorbing products, sanitary products, in particular tampons, sanitary aids for incontinence, sanitary napkins and panty liners, packaging of foodstuff, in particular for meat products, papers, in particular filter papers, clothes (e.g., clothing textiles for the moisture management in combination with other fibres or as multi-layered construction) and wound dressings.

Claims:

1. A regenerated cellulose fibre, which is characterized by the combination of the following features:
 - the fibre has in its dry condition a collapsed hollow cross-sectional structure
 - the fibre has in its wet condition a cross-sectional structure with cavities
 - the fibre is segmented in the longitudinal direction by dividing walls
 - there is incorporated into the fibre an absorbent polymer in an amount of 5% by weight to 50% by weight based on underivatised cellulose.
2. A cellulose fibre according to claim 1, characterized in that the cross-section of the fibre in the dry condition is multilobal and/or is irregularly grooved.
3. A cellulose fibre according to claim 1 or claim 2, characterized in that the distance between the dividing walls is between 0.3 and 3 times the fibre width averaged over the fibre length.
4. A cellulose fibre according to claim 3, characterized in that the distance between the dividing walls is between 0.5 to 2 times the fibre width averaged over the fibre length.
5. A cellulose fibre according to any one of claims 1 to 4, characterized in that the portion of absorbent polymer in the fibre is 15 % by weight to 40 % by weight, based on underivatised cellulose.
6. A cellulose fibre according to claim 5, characterized in that the portion of absorbent polymer in the fibre is 20 % by weight to 30 % by weight, based on underivatised cellulose.
7. A cellulose fibre according to any one of claims 1 to 6, characterized in that the absorbent polymer is carboxymethylcellulose.
8. A cellulose fibre according to any one of claims 1 to 7, characterized in that it is present in the form of a staple fibre.
9. A cellulose fibre according to any one of claims 1 to 8, characterized in that the fibre titre is 0.5 dtex to 8 dtex.
10. A cellulose fibre according to claim 9, characterized in that the fibre titre is 1.3 to 6 dtex.

11. A cellulose fibre according to any one of claims 1 to 10, characterized in that the fibre length is from 2 mm to 80 mm.

12. A cellulose fibre according to any one of claims 1 to 11, characterized by a water retention capacity of at least 300%, when measured according to DIN 53814.

13. A cellulose fibre according to any one of claims 1 to 12, characterized in that a cylindrical tampon produced from the fibre has a Syngina value of at least 5.2 g/g, when measured according to EDANA / INDA Standard Test Methods for the Nonwovens and Related Industries ERT 350.0 or WSP 350.1, the tampon having a mass of 2.72 g, a length of 44 mm and a diameter of 13 mm.

14. A process for the production of a regenerated cellulose fibre according to any one of claims 1 to 13, comprising the steps

- providing a viscose dope
- admixing a carbonate compatible with viscose into the viscose dope
- admixing an absorbent polymer into the viscose dope
- spinning the viscose dope in a spinning bath, thereby forming fibres,

wherein

- the carbonate is admixed in an amount of 11 % by weight to 23 % by weight (calculated as $(\text{CO}_3)^{2-}$), based on the cellulose in the viscose dope,
- the absorbent polymer is admixed in an amount of 5 % by weight to 50 % by weight, based on the cellulose in the viscose dope,
- the degree of ripening of the viscose dope before spinning is less than 15° Hottenroth,
- the content of H_2SO_4 in the spinning bath is 8% by weight to 10% by weight,
- the content of ZnSO_4 in the spinning bath is 0.3% by weight to 0.5% by weight, and
- the content of Na_2SO_4 in the spinning bath is 25% by weight to 30% by weight.

15. A process according to claim 14, wherein the degree of ripening of the viscose dope before spinning is 10° to 14° Hottenroth.

16. A process according to claim 14 or 15, characterized in that the absorbent polymer is admixed in an amount of 15 % by weight to 40 % by weight, based on the cellulose in the viscose dope.

17. A process according to claim 16, characterized in that the absorbent polymer is admixed in an amount of 20 % by weight to 30 % by weight, based on the cellulose in the viscose dope.
18. A process according to any one of claims 14 to 17, characterized in that the carbonate and/or the absorbent polymer is/are admixed in the form of a solution.
19. A process according to any one of claims 14 to 18, characterized in that the absorbent polymer is carboxymethylcellulose.
20. A process according to claim 19, characterized in that the carboxymethylcellulose is admixed in the form of an alkaline solution, containing 2 % by weight to 9 % by weight NaOH and 5% by weight to 15 % by weight, carboxymethylcellulose.
21. A process according to claim 20, characterized in that the alkaline solution contains 3 % by weight to 5 % by weight NaOH.
22. A process according to claim 20 or 21, characterized in that the alkaline solution contains 8 % by weight to 12 % by weight carboxymethylcellulose.
23. A process according to any one of claims 20 to 22, characterized in that the carbonate and the carboxymethylcellulose are admixed together.
24. A process according to claim 23, characterized in that the carbonate and the carboxymethylcellulose are admixed together in the form of a joint solution.
25. A use of a regenerated cellulose fibre according to any of claims 1 to 13 in absorbing products, sanitary products, sanitary aids for incontinence, sanitary napkins and panty liners, packaging of foodstuff, papers, clothes and wound dressings.
26. A use according to claim 25 in packaging for meat products.
27. A use according to claim 25 in filter papers.
28. A use according to claim 25 in tampons.

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FIG. 1

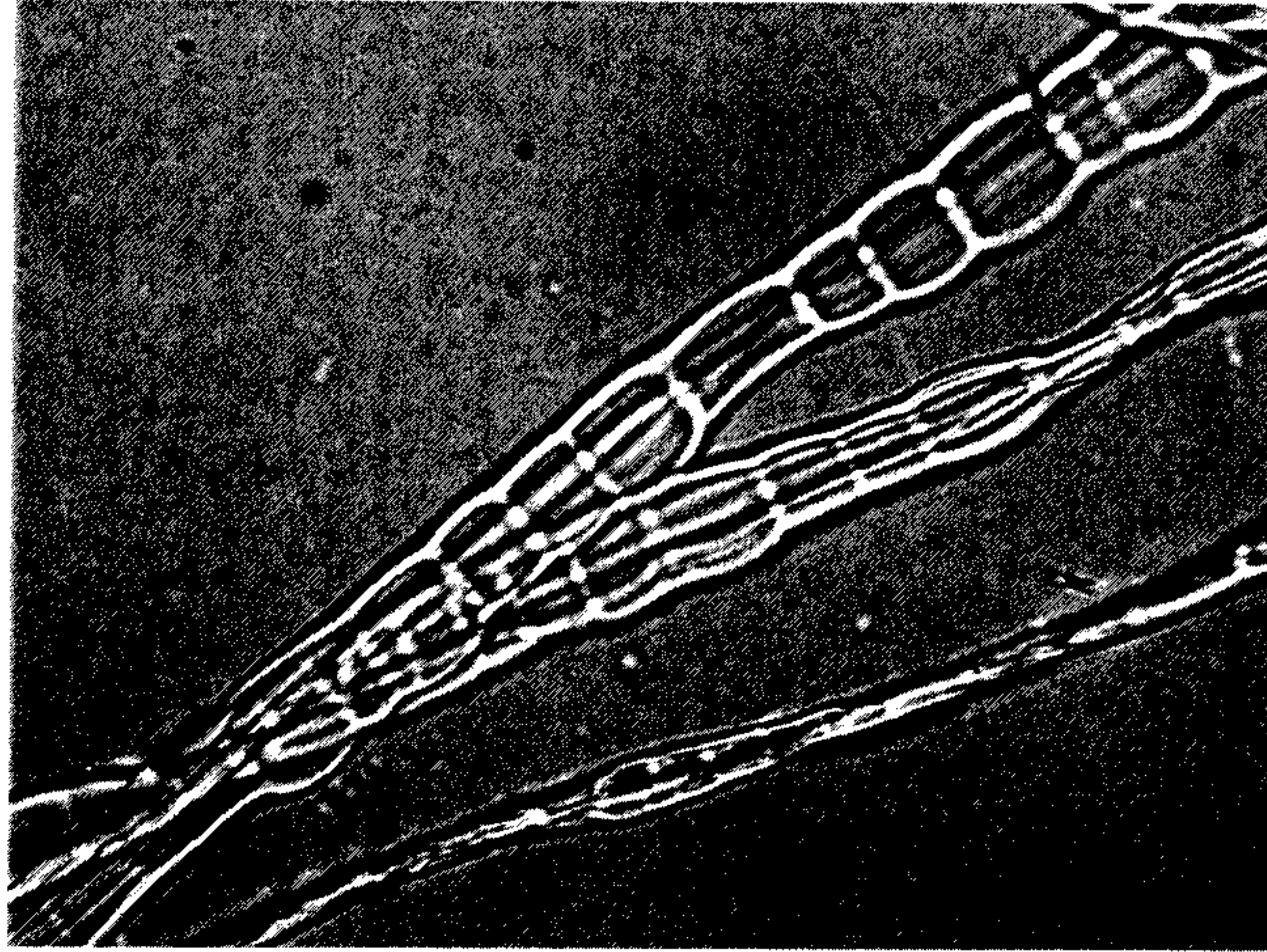
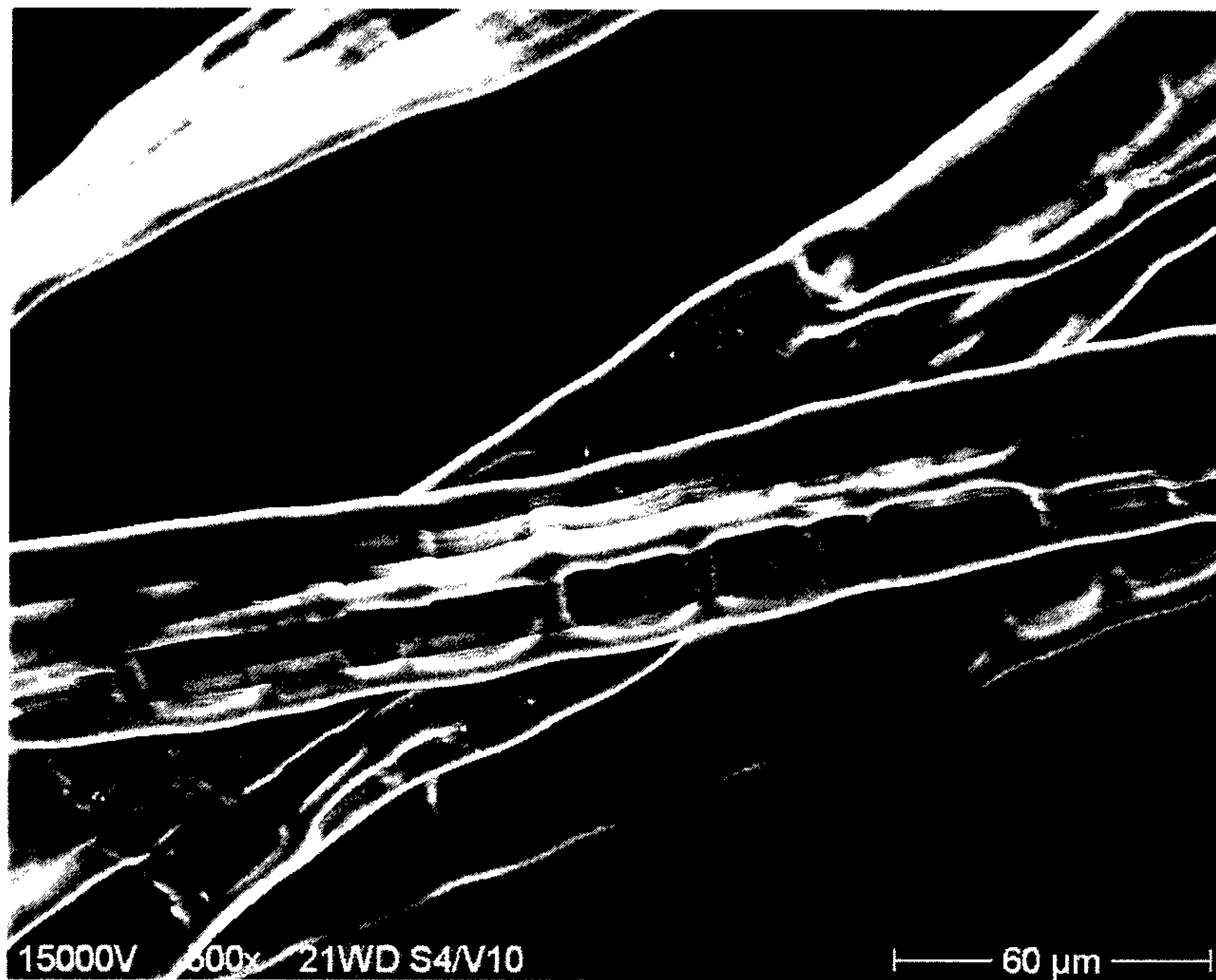


FIG. 2



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FIG. 3

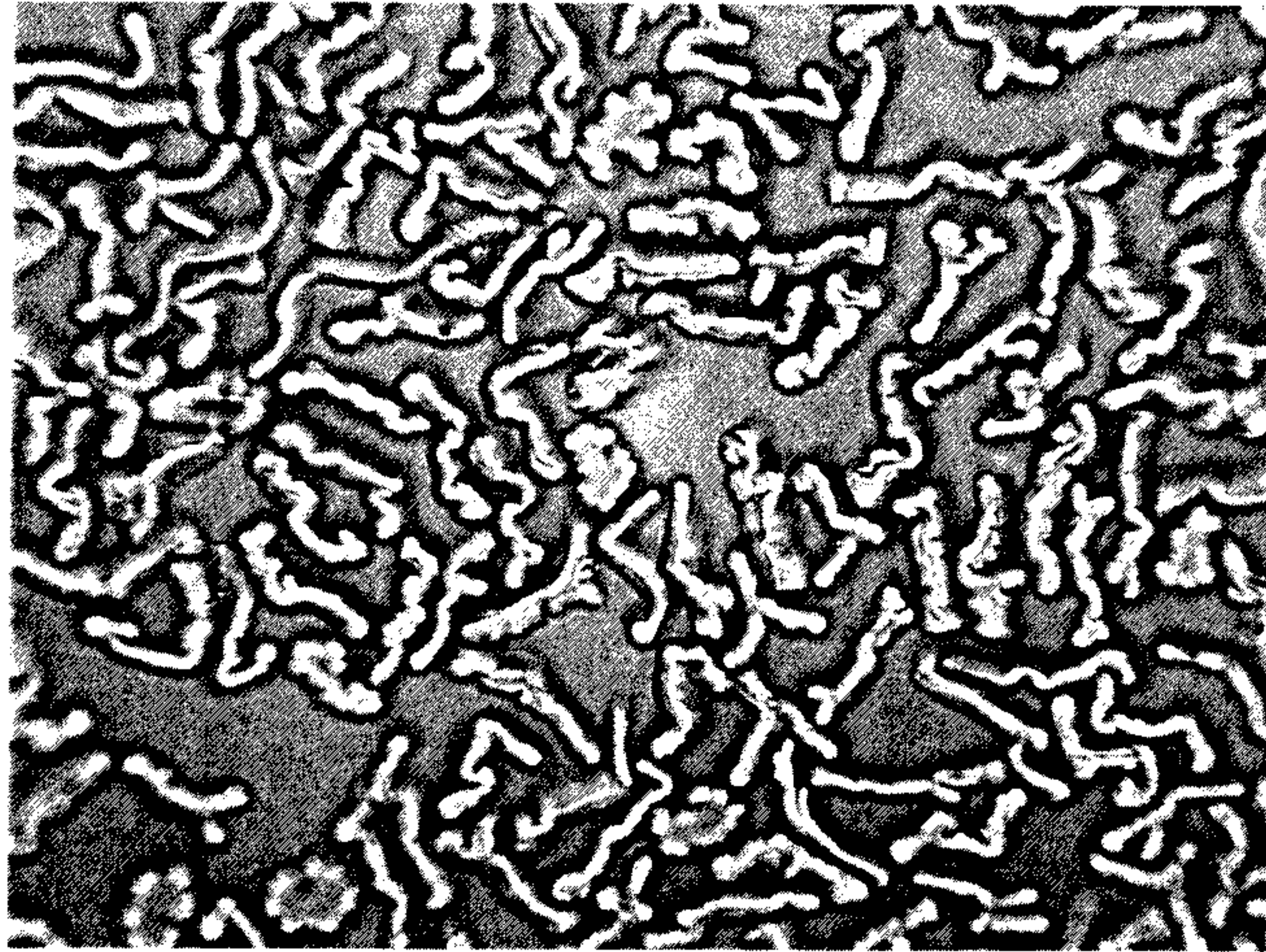


FIG. 4

