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(54) Title: PROCESSING OF CHEMICALLY MODIFIED CELLULOSIC FIBRES

(57) Abrégé/Abstract:
A chemically modified cellulosic fibre or filament having a moisture content of at least 7% by weight obtained by a process comprising the steps of (i) obtaining cellulosic fibres or filament and chemically modifying the cellulose by substitution to increase its absorbency; (ii) washing the fibres after step (i) in a mixture comprising water and up to 99% by weight of water-miscible organic solvent; (iii) drying the fibres to a moisture content of at least 7% by weight, tart abstract here

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(54) Title: PROCESSING OF CHEMICALLY MODIFIED CELLULOSIC FIBRES

(57) Abstract: A chemically modified cellulosic fibre or filament having a moisture content of at least 7% by weight obtained by a process comprising the steps of (i) obtaining cellulosic fibres or filament and chemically modifying the cellulose by substitution to increase its absorbency; (ii) washing the fibres after step (i) in a mixture comprising water and up to 99% by weight of water-mis - cible organic solvent; (iii) drying the fibres to a moisture content of at least 7% by weight, tart abstract here

Processing of Chemically Modified Cellulosic Fibres

This invention relates to chemically modified cellulosic fibres and processes for making nonwoven fibre webs from them. In particular it relates to chemically modified cellulosic fibres which may be carded on a carding machine to produce a web. The fibres may be ultimately processed into nonwoven fabrics for instance by needling or entangling the web, the fabrics being suitable for use in absorbent personal products or absorbent medical products such as wound dressings. Also described are carding processes suitable for forming webs of chemically modified cellulosic fibres or blends of chemically modified fibres with other non-gelling fibres, for example viscose or Tencel or other cellulosic fibres.

Absorbent cellulosic fibres are well known. For instance GB-A-2220881 and GB-A-2094802 describe the production of carboxymethyl cellulose fibres from regenerated cellulose fibres (viscose rayon) fibres or from cotton. It is also known that carboxymethyl cellulose fibre of greater absorbency and strength can be produced from solvent-spun cellulose fibre. Such fibres are described in EP 0616650 and are manufactured by reacting solvent spun cellulose fibre with a strong alkali and a monochloroacetic acid reagent. It is also known that alternative chemical modification of cellulose fibres is possible and also has the effect of increasing the absorbency of the cellulose fibre. The cellulose fibre can for instance be modified by sulphonation, for example by substitution with an alkyl sulphonate at one or more of the hydroxyl groups on the anhydroglucose monomers that make up the cellulose backbone forming ether linkages. Modified cellulose of this type is known as cellulose sulphonate or cellulose alkyl sulphonate some of the insoluble forms of which are described in WO2012/061225.

Modifying the cellulose fibre requires the fibre to be exposed to one or more reagents which modify the cellulose by substitution, the degree of substitution determining the absorbency and solubility of the fibre. As the degree of substitution is increased the modified cellulose becomes increasingly soluble. As the solubility increases, absorbency also increases.

For some applications, for instance for absorbent gelling wound dressings, the chemically modified fibres need to be insoluble and retain some of their fibrous form when exposed to wound fluid and consequently a balance needs to be found between solubility and absorbency. The chemically modified fibre can be in the form of a tow, filament, sliver, yarn or staple fibre, woven, non-woven or knitted fabric.

Once the modification and neutralisation in acid has taken place, the fibres need to be washed to remove any unreacted alkali, chloroacetate, alkylsulphonate, other modifying agent or any by-products such as sodium chloride or sodium glycollate. An aqueous wash is generally used initially, preferably a mixture of water with a water-miscible organic solvent such as water and IMS, the major portion of the wash being organic solvent. The wash may contain an organic hydroxyl compound, a surfactant, and/or acid. The organic hydroxyl compound is a compound containing at least one alcoholic hydroxyl group, for example ethanol, methanol or another low molecular weight alcohol and/or a compound such as ethylene glycol or propylene glycol.

The final wash or finish typically comprises a large percentage of alcohol for instance, 99% industrial alcohol and 1% emulsifier or 100% alcohol. After washing, the fibres are dried at low temperature for instance as described in EP 0680344, by forced air drying or radiant heat drying.

As the washing step or steps to which the fibres are subjected following modification involve the use of relatively high percentages of organic solvents such as industrial alcohol, the drying of the fibres requires the solvents that are released to be managed from an environmental and safety perspective. It would be desirable to reduce the percentage of organic solvents used in the washing process so that environmental and safety standards can be readily met. There are also potential cost and efficiency advantages to using a lower percentage of organic solvents as the cost of materials and duty are reduced.

We have found that reducing, for instance, the percentage of organic solvent in the wash liquor used to wash modified cellulose fibres results in fibres that have a lower tensile strength making them less suitable for processing on textile machinery such as a carding machine. The lower tensile strength can lead to the problem of fibre

breakage on carding which gives higher levels of airborne lint or dust (fly) and greater shedding of broken fibre from the finished fabric. The higher level of fly necessitates increased cleaning and environment management in the fabric production process which in turn increases cost. There is also more wastage in the process as fibres tend to drop out of the fabric during the process of manufacture.

Fibre breakage can also adversely affect the staple length which has the effect of reducing the strength of a carded web and nonwoven fabric made from the fibres. It is possible that fibre breakage leads to a failed card process where no non-woven mat is produced. A weaker product can be more difficult to remove from the wound as the integrity of the dressing can be compromised.

We have now found that it is possible to mitigate the problem of low tensile strength in modified fibres by controlled management of the moisture content of the fibres.

Accordingly the invention provides a chemically modified cellulosic web of fibres having a tensile strength of at least 3N per cm (N/cm) in the cross direction produced by processing fibres having a moisture content of at least 7% by weight.

Controlling the moisture content of the fibres, either by controlling the drying process following washing of the modified fibres as part of the chemical modification process or by conditioning the fibres to increase their moisture content to at least 7% subsequent to drying, increases the tensile strength of the fibres to the point where it is possible to process the fibres for example on a carding machine to produce a non-woven fibre web without the production of excessive fly or shedding from a fabric produced from the web. Preferably the moisture content of the fibres is kept at between 11 and 20% by controlling the drying process following washing of the modified fibres as part of the chemical modification process.

Preferably the tensile strength is measured in the cross direction on needled webs having a basis weight in the range of 80 to 120 gsm.

Accordingly the invention provides, in a further aspect, a process for producing a non-woven web of chemically modified cellulosic fibres comprising the steps of:

(i) obtaining chemically modified cellulosic fibres with a moisture content of at least 7%;
(ii) carding the fibres to make a nonwoven web of fibres.

The fibres are typically those resulting from a modification process using a wash or final rinse comprising less than 99% by weight such as 95% by weight of organic solvent in the wash liquor. By controlling the moisture content, the fibres can be processed into a consolidated form that allows a fabric to be produced by conventional carding means while mitigating the problem of fibre breakage and fly in the environment of the fabric production plant. A consolidated form could for example be a yarn, sliver, woven, non-woven or knitted fabric.

When the fibres are to be processed using textiling machinery, if it is found that the moisture content of the fibres is too low, the moisture content of the modified cellulosic fibres is increased by exposing the fibres to a moisture rich environment for at least 24 hours. Alternatively the drying of the fibres at the end of the chemical modification process can be controlled so that the moisture content of the fibres is maintained above 7% until the fibres are subjected to further processing such as with textiling machinery for instance by carding and needling.

Preferably the carded, needled fibre web has a tensile strength of from 3N/cm to 25 N/cm, more preferably from 7N/cm to 25N/cm and most preferably from 10N/cm to 25N/cm.

The tensile strength of the carded, needled web is measured by cutting five specimens to a size of 25 mm x 75 mm with the long dimension being in the direction to be tested. Tensile testing is carried out at a gauge length (effective test fabric length) of 50mm. One end of the sample is fixed in an upper jaw of a tensile testing machine and allowed to dangle into the lower jaw. The sample is then fixed by closing the jaws without stretching the sample or allowing it to be slack. The crosshead speed is set to 100mm/min and the sample pulled to break. The tensile strength is calculated by dividing the force at break by the sample width in cm.

Preferably the moisture content of the fibres prior to processing with textiling machinery is from 7% to 20% by weight and more preferably from 11% to 18%. More preferably the moisture content is from 11.5% and 15% by weight and most preferably it is between 12 and 15% by weight. A moisture content of 12.5% to 15% is particularly preferred and 15% is most preferred.

By the term moisture content is meant the amount of moisture (measured by weight) contained in the sample of fibres as a percentage of its conditioned weight and measured by the loss on drying.

The moisture content of the fibres is measured by calculating the loss on drying using a moisture balance operated in accordance with the instrument manual. For convenience, during the process for making the fibres, comparative measurements of moisture content can be made using a moisture meter for instance an Aquaboy (TEMI).

By the term carding in the present invention is meant a mechanical process that disentangles, cleans and intermixes fibres to produce a continuous web or sliver suitable for further processing.

In a further aspect of the invention, the invention provides a chemically modified cellulosic fibre having a moisture content of at least 7% by weight.

The invention will now be illustrated by the following examples.

<u>Example 1</u>

Comparison of the moisture content of fibres vs their tensile strengths once carded and needled was made by measuring the initial moisture content of dried modified cellulosic fibres produced using washes of varying alcohol content. That initial moisture content was then increased by conditioning the fibres in a moist atmosphere for 24 hours at a relative humidity of 55 to 60% and the tensile strength of the needled fabric measured again. Figure 1 shows the fibre moisture content vs tensile

strength of carded needled fabric in the transverse (cross) direction before and after moisture conditioning to increase the moisture content.

The results show that the moisture content of the fibres influences the tensile strength of the resulting carded and needled web. The column headed IDA in the table of Figure 1 refers to the percentage of IDA (industrial denatured alcohols) used in the wash liquor as part of the modification process. The columns headed % refer to the % moisture in the fibre and N/cm to the tensile strength of the resulting carded needled web in the transverse direction. It can be seen from Figure 1 that decreasing the % of IDA in the wash liquor has a negative influence on tensile strength for carded and needled webs made from unconditioned fibres. It can also be seen that once the moisture content of those fibres is raised to more than 11%, such as 12.5 to 14% moisture those tensile strength values of the carded needled web increase.

Example 2

Effect of moisture content on the tensile strength of silver containing wound dressings.

Dressings were prepared by modification of solvent spun cellulose tow to a degree of substitution of 0.3 to form carboxymethylcellulose, neutralising to a pH of 5.5 with an organic acid. Adding 1.2% cationic silver by an ion exchange process in a largely organic solvent such as by the process described in Ep1343510, washing in an aqueous organic solution containing sodium chloride and di-sodium EDTA for light stabilisation and to entrain approximately 0.4% EDTA. Followed by washing in organic solvent wash containing fibre finishing agents including tween 20 and benzethonium chloride (to give 0.135% wt/wt BeCl on the finished product) and subsequently warm air drying, cutting to staple and processing into a nonwoven felt by carding and a needle punching process. The dressings were cut to size from the web and packaged in a light, moisture and vapour impermeable heat sealed foil pouch.

The dressings were removed from the packs and then subjected to various controlled environments.

Controlled environments

Ambient as packed, tested without any preconditioning
Zero humidity: Stored in a square desiccator with 3 perforated perspex shelves above a layer of silica gel desiccant, conditioned for a minimum of 5 days
25°C/60%RH, for a minimum of 6 days
30°C/65%RH, for a minimum of 6 days
40°C/75%RH, for a minimum of 6 days

Samples (ambient as packed) were tested immediately after opening the packs. Samples removed from the other environments were sealed into plastic bags during removal, and then tested immediately. The plastic bags (also preconditioned in corresponding controlled environments) were used to maintain the humidity of the environment of the samples until the point of testing.

Loss on Drying (LOD)

LOD of the samples was determined using the Ohaus moisture balance MB23 operated in accordance with the instruction manual. A sample mass of greater than 1 gram was used. Samples were cut to fit within the weighing pan, ensuring there was adequate clearance from the heating element. A standardised method was used with a maximum temperature limit of 110°C. The endpoint was determined automatically when the sample mass stopped reducing and was stable. Under these conditions the fabric did not char. Typically, samples would be subjected to a 10 minute cycle.

Fabric Thickness (Loft)

Samples were tested using the Hampden Soft Materials Thickness Gauge, Model FMTml-4D, S/N 14082. Fabric thickness (sometimes referred to as loft) was determined for 6 dressings per batch.

Fabric Dry Tensile Strength

2.5cm x 7.5cm rectangular strips were cut from along the length (machine direction) and across the width (transverse direction) using a ribbon cutting die and press. Samples were conditioned as described Table 1. The peak force and the extension at which that force occurred were recorded when a 50mm test length was stretched at a constant separation rate of 100mm per minute.

Results

Table 1

Relationship between absolute and relative humidity					
Conditioning	Dry	Ambient	25°C/60%RH	30°C/65%RH	40°C/75%RH
Moisture (g/m^3)	0.00	9.50	13.81	19.71	38.29
Thickness (mm)	0.170	0.190	0.198	0.183	0.202
LOD%	9.28	11.93	14.17	15.27	18.10
Tensile Machine (N/cm)	3.10	5.19	5.80	5.36	7.75
Tensile Transverse (N/Cm)	5.15	6.53	11.12	10.23	14.28
GSM (g/m2)	91	102	105	98	108

Loss on drying is the summation of all the volatile substances that can be removed by heating at 110°C. These include ethanol, water and to some degree acetic acid.

For this particular fibre, for this example, textile trials have shown that fibres for use in wound dressings according to the invention can be successfully textiled between 42% and 50% RH at around 18 to 20°C. Trials suggest that fibres with 10.5% to 11.5% w/w moisture content can be carded efficiently.

The results show that tensile strength, loft and LOD are all functions of equilibrium moisture content. The results suggest that fibres with a moisture content of greater than 9% will be able to be textiled to produce dressings suitable for use in the present invention.

Example 3

Materials

Two gelling fibre types were used in this study; cellulose ethylsulphonate (CES) and carboxymethyl cellulose (CMC).

Table 1 Summary of gelling fibres used

Gelling Fibre
CES fibre tow
CMC fibre tow

Methods

Single fibres were mounted onto card windows as described in BS EN 5079:1996.

In addition, tow bundles were prepared to assess the moisture content.

Fibres were oven dried at 105°C for one hour prior to being conditioned at the chosen relative humidity for a minimum of 16 hours. The mass of the oven dried tow bundles (W1) and the conditioned tow bundles (W2) was taken to assess the moisture content of the fibres, using Equation 1.

Equation 1: $$Moisture\ Content\ (\%) = \frac{W2 - W1}{W2} x\ 100$$

Single fibre tensile strength of the conditioned samples was undertaken using the method described in BS EN 5079:1996.

Fibres were tested at 45% RH, 65% RH, and 85% RH.

Results

Results found that a significantly higher ($P \leq 0.05$) tensile strength was seen in the samples conditioned at 65% RH than those conditioned at 45% RH and 85% RH as shown in Table 2.

Table 2 Summary of results

Relative Humidity of Atmosphere (%) Ambient		45	65	85
CES	Moisture Content in fibre (%) w/w	9	15	22
	Mean Fibre Breaking Strength (cN)	4.85	6.16	3.79
	Standard Deviation (cN)	1.08	3.61	1.21
	T-Test against 65% RH data P value	0.016	N/A	0.000
CMC	Moisture Content in fibre (%)	12	15	21
	Mean Fibre Breaking Strength (cN)	6.04	8.24	4.16
	Standard Deviation (cN)	2.65	6.06	1.55
	T-Test against 65% RH data P value	0.029	N/A	0.000

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Claims

1. A chemically modified cellulosic web of needled fibres having a tensile strength of at least 3N per cm measured in the cross direction obtained by processing fibres having a moisture content of at least 7% by weight.

2. A chemically modified cellulosic fibre having a moisture content of at least 7% by weight.

3. A chemically modified web of fibres or fibre as claimed in claim 1 or claim 2 having a moisture content from 11% to 18% by weight.

4. A chemically modified web of fibres or fibre as claimed in claim 1 or claim 2 having a moisture content from 12% and 15% by weight.

5. A chemically modified web of fibres or fibre as claimed in claim 1 or claim 2 having a moisture content of 15% by weight.

6. A chemically modified web of fibres or fibre as claimed in any preceding claim wherein the fibres are obtained from a chemical modification process comprising a wash step that uses a wash composition comprising less than 99% by weight of a water-miscible organic solvent.

7. A chemically modified web of fibres as claimed in claim 1 or any of claims 3 to 6 processed by carding and needling having a tensile strength of 3N/cm to 25N/cm.

8. A chemically modified web of fibres as claimed in claim 7 having a basis weight of between 80 and 120 gsm.

9. A chemically modified web or fibres as claimed in claim 7 or 8 having a tensile strength of from 7N/cm to 25N/cm.

10. A chemically modified web or fibres as claimed in claim 7, 8 or 9 having a tensile strength of from 10N/cm to 25N/cm

11. A process for producing a non-woven web of chemically modified cellulosic fibres comprising the steps of:

(i) obtaining chemically modified cellulosic fibres with a moisture content of at least 7%;

(ii) carding and needling the fibres to make a nonwoven web of fibres.

12. A process as claimed in claim 11 wherein the fibres are obtained from a chemical modification process which comprises a step wherein the fibres are washed with a composition comprising less than 99% by weight of an organic solvent.

13. A process as claimed in claim 12 wherein the wash composition comprises less than 95% by weight of a water-miscible organic solvent.

14. A process as claimed in any of claims 12 to 13 wherein after the washing step, the fibres are dried to a moisture content of at least 7%.

15. A process as claimed in claim 12 wherein the fibres are obtained by conditioning the fibres in air having a relative humidity of at least 40% to increase the moisture content of the fibres to least 7%.

16. A process as claimed in claim 18 wherein the relative humidity is from 45% to 85%.

17. A process as claimed in claim 18 or 19 wherein the relative humidity is from 50% to 65%.

18. A process as claimed in any of claims 11 to 17 wherein the fibres have a moisture content of from 11% to 18% by weight.

19. A process as claimed in any of claims 11 to 18 wherein the fibres have a moisture content from 12% and 15% by weight.

20. A process as claimed in any of claims 11 to 19 wherein the fibres have a moisture content of 14% to 15% by weight.

21. A process for producing a non-woven web of chemically modified cellulosic fibres comprising the steps of:

(i) obtaining cellulosic fibres or filament and chemically modifying the cellulose by substitution to increase its absorbency, then;

(ii) washing the fibres with a composition comprising water and up to 99% by weight of water-miscible organic solvent; and then

(iii) drying the fibres in a controlled atmosphere to a moisture content of at least 7% by weight.

22. A process as claimed in claim 21 wherein the wash composition comprises less than 95% by weight of an organic solvent.

23. A process as claimed in claim 21 or 22 wherein the process comprises the additional step of needling the web to form a wound dressing.

24. A process as claimed in claim 23 wherein the process comprises the additional step of sealing and sterilising the dressing in a pouch that maintains the dressing in a sterile and controlled environment.

25. A process as claimed in any of claims 21 to 24 wherein the fibres have a moisture content of from 11% to 18% by weight.

26. A process as claimed in any of claims 21 to 25 wherein the fibres have a moisture content from 12% and 15% by weight.

27. A process as claimed in any of claims 21 to 26 wherein the fibres have a moisture content of 14% to 15% by weight.

28. A chemically modified cellulosic fibre or filament having a moisture content of at least 7% by weight obtained by a process comprising the steps of

(i) obtaining cellulosic fibres or filament and chemically modifying the cellulose by substitution to increase its absorbency;

(ii) washing the fibres after step (i) in a mixture comprising water and up to 99% by weight of water-miscible organic solvent;

(iii) drying the fibres to a moisture content of at least 7% by weight.

29. A process as claimed in claim 28 wherein the wash composition comprises less than 95% by weight of an organic solvent.

30. A process as claimed in claim 28 or 29 wherein the process comprises the additional step of carding and needling the fibre to form a wound dressing.

31. A process as claimed in claim 30 wherein the process comprises the additional step of sealing and sterilising the dressing in a pouch that maintains the moisture content of the dressing in a sterile environment.

32. A process as claimed in any of claims 28 to 31 wherein the fibres or filaments have a moisture content of from 11% to 18% by weight.

33. A process as claimed in any of claims 28 to 32 wherein the fibres or filaments have a moisture content from 12% and 15% by weight.

34. A process as claimed in any of claims 28 to 33 wherein the fibres or filaments have a moisture content of 14% to 15% by weight.

35. A process for producing a non-woven web of chemically modified cellulosic fibres comprising the steps of:

(i) obtaining cellulosic fibres or filament and chemically modifying the cellulose by substitution to increase its absorbency, then;

(ii) washing the fibres with a composition comprising up to 99% by weight of an organic solvent; and then

(iii) conditioning the fibres in an atmosphere having a relative humidity of at least 40% to increase the moisture content of the fibres to least 7%.

36. A process as claimed in claim 35 wherein the wash composition comprises less than 95% by weight of an organic solvent.

37. A process as claimed in claim 35 or 36 wherein the conditioning is in air having a relative humidity of 45% to 85%.

38. A process as claimed in claim 35 to 37 wherein the relative humidity is from 50% to 65%.

39. A process as claimed in any of claims 35 to 38 wherein the fibres have a moisture content of from 11% to 18% by weight.

40. A process as claimed in any of claims 35 to 39 wherein the fibres have a moisture content from 12% and 15% by weight.

41. A process as claimed in any of claims 35 to 40 wherein the fibres have a moisture content of 14% to 15% by weight.

42. A process as claimed in claims 35 to 41 wherein the process comprises the additional step of carding and needling the fibres to form a wound dressing.

43. A process as claimed in claim 42 wherein the process comprises the additional step of sealing and sterilising the dressing in a pouch that maintains the moisture content of the dressing in a sterile environment.

Moisture vs. Tensile strength

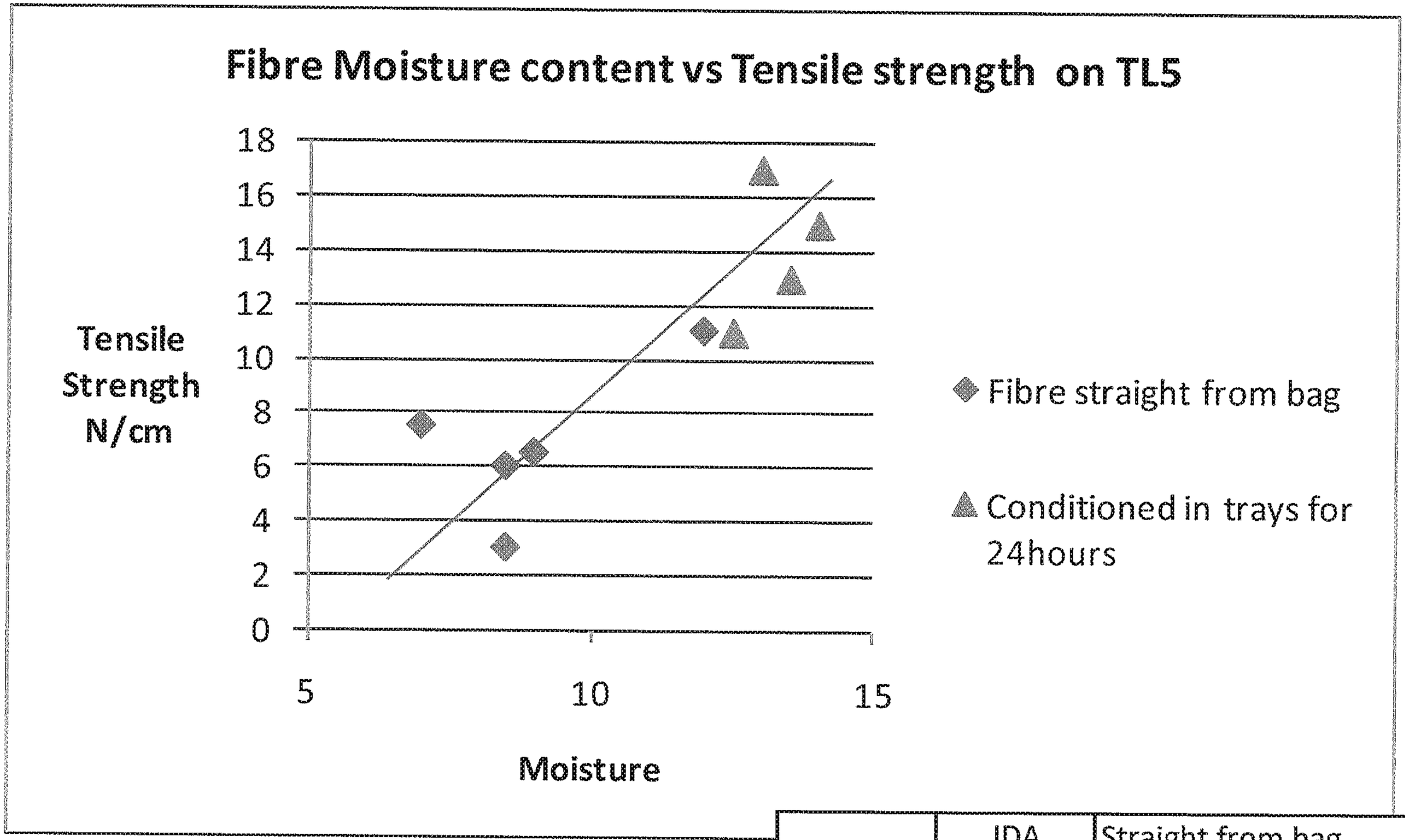

Figure 1

	IDA	Straight from bag		Conditioned 24hrs	
		%	N/cm	%	N/cm
2G00360	99%	12	11	13	17
2D01372	96%	8.5	3	14	15
2C03198	96%	8.5	6	12.5	11
2E01891	96%	9	6.5		
2G01295	99%	7	7.5	13.5	13

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