Title of the Invention: Process
Abstract Title: Process for making cellulose articles

A process for producing cellulose shaped articles comprises the steps of steeping a non-dissolving cellulose-containing pulp in a basic liquid, xanthating the steeped cellulose-containing pulp with carbon disulphide, dissolving the xanthated cellulose-containing pulp in a basic liquid to form a viscose solution, and casting a cellulose shaped article from the viscose solution, wherein one or more of the following optimised processing conditions are employed: steeping is carried out, at least partially, at a temperature of about 30°C to about 50°C, steeping is carried out in a basic liquid having a concentration of base of about 16 to about 18% by weight, xanthation is carried out using about 30 to about 40% carbon disulphide by weight of cellulose present, xanthation is carried out, at least partially, at a temperature of about 20°C to about 30°C, dissolution is carried out, at least partially, at a temperature of about 10°C to about 25°C, dissolution results in a viscose solution having a base concentration of between about 5.5 and 7.5% by weight of the solution, and/or dissolution results in a viscose solution having a cellulose concentration of between about 8.5 and about 9.5% by weight of the solution.
Figure 10

Figure 11A
Figure 11B
PROCESS

The present invention provides an optimised method of processing cellulose in which a non-dissolving cellulose-containing pulp is used to prepare a viscose solution for the preparation of cellulose shaped articles, such as fibres.

In addition to its utility in the industrial production of paper, cellulose-containing pulp has a number of other applications, including the industrial manufacture of cellulose shaped articles, such as fibres.

There are numerous sources of cellulose-containing pulps, for example wood, (especially softwood), cotton, flax, hemp and bamboo. Pulp is generally prepared from those raw materials by treatment to separate out and remove lignin and hemicellulose, so as to maximise the cellulose content of the pulp.

There are two general pulping techniques known to those skilled in the art. Firstly, pulp can be prepared mechanically, by milling or grinding the raw material to physically separate cellulose fibres from hemi-cellulose and lignin.

Alternatively, the raw material can be chemically treated to dissolve lignin and hemicellulose, ideally without disrupting the cellulose fibres native to the raw material. Examples of chemical pulping processes include the Kraft process and the Sulphite process.
There are also a number of hybrid pulping processes where chemical processing steps are employed in a mechanical pulping process, or vice versa. Examples of such processes include thermomechanical processes, where, in addition to mechanical comminution, wood chips or other raw materials are also exposed to heat, and chemithermomechanical processes, where wood chips are firstly exposed to chemicals used in chemical pulping processes before comminution and exposure to heat.

For those skilled in the art of cellulose regeneration, there are two classes of pulp obtained from chemical pulping processes. Firstly, there are ‘dissolving pulps’ or ‘dissolving grade pulps’, which are used as feedstocks in the viscose process. Dissolving pulps are characterised by a high cellulose content, around 90% or higher. As their name suggests, they are soluble in solvents or dopes used in commercial cellulose regeneration processes.

The other class of pulps are ‘non-dissolving pulps’, examples of which include ‘market pulps’ or ‘fluff pulps’. These have limited utility in the viscose process and in other processes for producing cellulose shaped products because, as their name suggests, they are not soluble in conventionally used processing solutions or dopes. Accordingly, they are principally used in applications other than cellulose regeneration. For example, over 80% of all fluff pulp is used in the production of baby diapers.

Owing to the lower cost of non-dissolving pulps, as compared to dissolving pulps, there have been attempts made previously to employ non-dissolving pulps in the
viscose process. However, as a result of their deleterious effects on the properties of the resulting viscose solutions, they can only be used in low proportions, as filler materials, making up no more than about 5 to 10% of the total pulp used.

Examples of commercially available non-dissolving pulps include Pearl 429 (Weyerhauser), Peach (Weyerhauser) and Fluff 416 (Weyerhauser).

In addition to their dissolution properties, non-dissolving pulps differ from dissolving pulps in a number of ways. For example, they generally have a higher hemi-cellulose content, a lower alpha-cellulose content, are less refined, have higher degrees of polymerisation (DP) and / or have lower quality control than dissolving pulps.

As those skilled in the art will be aware, the viscose process as generally practiced includes the steps of dissolving or slurrying a pulp in caustic soda, steeping it in the caustic solution, xanthating the cellulose with carbon disulphide, and re-dissolving it in an aqueous caustic solution to form viscose.

Viscose is typically filtered and refiltered in order to maximise the purity of the material to improve product quality. It is then formed into a desired shape using techniques known to those in the art, for example by extruding it through a spinnerette to form a fibrous material , which is then contacted with an acidic casting solution to regenerate the cellulose from viscose.
In certain applications, between the steeping and xanthation steps, the steeped cellulose slurry or solution may be subjected to mercerisation, where a portion of the caustic liquid is removed, for example by pressing, to achieve an alkali cellulose having a target cellulose and soda content. The purpose of this step is to improve the properties of the cellulose, notably by reducing its degree of polymerisation (DP).

If a non-dissolving pulp is subjected to these steps as conventionally practiced, the resulting viscose solution will have unacceptably low filterability for shape forming applications. It is this adverse effect on the filterability of viscose solutions which has prevented non-dissolving pulps being used in the viscose process in proportions greater than about 5 to 10% by weight of the total pulp.

Dissolving pulps, on the other hand, which are optimised for use in the viscose process can be used to produce viscose liquids exhibiting a high level of filterability.

Filterability can be measured using a number of different testing methods. The clog constant (Rv) is a calculated by taking the slope of the curve generated by plotting the weight of viscose passed through a filter cloth in five minute intervals.

TVW is a measure of the total amount of viscose filtered within a thirty minute period.

Typically, the higher the Rv and the TVW values, the higher quality the viscose as it does not clog or block the filter cloth.
Ball fall velocity (BFV), measured in seconds, is a measure of the time it takes for a steel ball weighing 0.13g +/- 0.02g to sink to the bottom of a sample of viscose solution having a depth of 205mm.

As an alternative technique for quantifying the quality of a viscose solution, the fibre count method can be used, in which the quantity of residual fibres in the solution is measured.

There remains a need in the art to optimise the viscose process to enable less costly non-dissolving pulps to be used in cellulose processing methods.

Thus, according to a first aspect of the present invention, there is provided a process for producing cellulose shaped articles comprising the steps of steeping a non-dissolving cellulose-containing pulp in a basic liquid, xanthating the steeped cellulose-containing pulp with carbon disulphide, dissolving the xanthated cellulose-containing pulp in a basic liquid to form a viscose solution, and casting a cellulose shaped article from the viscose solution, wherein one or more of the following optimised processing conditions are employed: steeping is carried out, at least partially, at a temperature of about 30°C to about 50°C, steeping is carried out in a basic liquid having a concentration of base of about 16 to about 18% by weight, xanthation is carried out using about 30 to about 40% carbon disulphide by weight of cellulose present, xanthation is carried out at a temperature of about 20°C to about 30°C, dissolution is carried out, at least partially, at a temperature of about 10°C to
about 25°C, dissolution results in a viscose solution having a base concentration of between about 6 and about 7% by weight of the solution, and / or dissolution results in a viscose solution having a cellulose concentration of between about 8.5 and about 9.5% by weight of the solution

Advantageously, the optimised processing conditions of the present invention can be used to form high-quality viscose solutions, which are suitable for use in the preparation of cellulose shaped articles, from non-dissolving pulps.

The shaped articles which are produced according to the processes of the present invention are most preferably fibres. Other products which may also be formed include ropes, yarns, cloths or cigarette filters. These other products may be formed directly from the cellulose solution, or may be formed from fibres spun from the cellulose solution.

For the avoidance of any doubt, the term ‘shaped articles’ shall not encompass cellulose sheets, films, laminates or the like.

For the avoidance of doubt, the presence of additional processing steps may be, and most likely will be, performed prior to step a) and / or between steps a) to d). Such steps will be discussed below in greater detail. It will be recognised that such steps, may alter the appearance or properties of the pulp. Thus, where reference is made, in steps b) and c) to cellulose-containing pulp, it will be appreciated that the pulp will not necessarily be in the form of a pulp per se, but may be in the form of a processed version of the pulp, for example a slurry, solution or crumb prepared from the non-dissolving pulp.
As mentioned above, the skilled artisan will understand what is meant by a non-dissolving pulp. S/he will identify that such pulps exhibit one or more of the following properties: relatively high hemi-cellulose content (typically about 10% or greater), relatively low alpha-cellulose content (typically about 90% or less, more typically about 83 to about 89%), less refined, relatively high degrees of polymerisation (DP) (typically about 700 to about 1200) and/or have relatively low quality control, as compared to dissolving pulps.

Prior to step a), a slurrying or dissolution step is preferably performed. This step involves forming a solution or slurry of cellulose-containing pulp in a basic liquid, for example a caustic solution, typically comprising an alkali metal hydroxide such as sodium hydroxide. Conventionally, the concentration of the caustic solution used with dissolving pulps is 18 to 20% by weight. The concentration of the cellulose in the solution or slurry is typically in the range of about 2 to 15% by weight.

In step a), a solution or slurry of cellulose is steeped in a basic solution for a period of time. This results in the basic solution penetrating the cellulose, resulting in the partial formation of the alkali salt of cellulose, for example the sodium salt. The basic liquid is generally a caustic solution, typically comprising an alkali metal hydroxide such as sodium hydroxide.

Conventionally, the concentration of the caustic solution used with dissolving pulps is 18 to 20% by weight. The concentration of the cellulose in the solution or slurry is typically in the range of about 2 to 15% by weight. Steeping is generally conducted
for about 10 to 120 minutes and at a temperature equal to or greater than about 50°C.

Some of these process conditions may be employed in the process of the present invention. However, surprisingly, it has been found that advantageous results can be obtained when non-dissolving pulp is exposed to milder conditions.

Thus, in preferred embodiments of the present invention, step a) is performed by steeping non-dissolving pulp in a caustic solution of about 16 to about 18% alkali metal hydroxide. In particularly preferred embodiments, the non-dissolving pulp is steeped in a caustic solution having a concentration of about 16.5% to about 17.5%, about 17.5% to about 18%, about 17% to about 18%, about 16.6% to about 17.0%, or about 17.2 to about 17.8% alkali metal hydroxide by weight. Caustic solutions of the same concentrations may be used in the slurring or dissolution step outlined above.

It has also unexpectedly been found that steeping non-dissolving pulp at lower temperatures than those used conventionally results in an alkali cellulose than can be used to form viscose solutions exhibiting improved properties. Thus, in preferred embodiments of the present invention, steeping is performed, at least partially, at a temperature in the range of about 30°C to about 50°C, about 35°C to about 48°C, about 35°C to about 45°C, about 38°C to about 42°C, or about 40°C to about 48°C.
It has been found that the preferred steeping conditions of the present invention, which can be used to produce viscose solutions exhibiting good filterability, may result in the removal of less hemicellulose from the cellulose pulp than if conventional steeping conditions are used with dissolving pulps. It was previously considered that the greatest possible amount of hemicellulose should be removed from the pulp to maximise filterability of the resulting viscose solution. However, it has unexpectedly been found that while a significant proportion of native hemicellulose should be removed from the pulp, retention of a small amount appears to actually contribute to the quality of the resulting viscose solution.

Thus, in preferred embodiments of the present invention, the viscose solution formed in step (c) preferably contains from 0.1% to about 1.0%, about 0.8%, about 0.6% or about 0.5% of hemicellulose, by weight of the total amount of cellulose present.

In the viscose process as conventionally practiced, manganese may be added to the steeping liquor to reduce the DP of the cellulose pulp. It is typically added in an amount of 40 to 500ppm (by weight of the mixture). Manganese may be employed in the same way in the process of the present invention.

One or more steeping additives may be present in or added to cellulose containing slurries or solutions employed in the process of the present invention. These function by holding open the cellulose structure and the skilled artisan will be familiar with such materials. Examples of steeping additives which may be used include glycerol and / or alkoxylated alcohols, especially ethoxylated alcohols. These may be added to the solution or slurry in any of the steps of the present invention, most preferably in step a) and / or any slurrying or dissolution step which is performed. An example of an especially preferred steeping additive is Berol 388. Where used, a
steeping additive such as Berol 388 is preferably added in an amount of about 0.1% to about 5.0%, about 0.5% to about 5%, about 1% to about 4%, or about 0.5 to about 2% by weight of cellulose.

Additionally or alternatively, one or more swelling agents may be present or added to the slurry or solution processed in the present invention. Preferably, the one or more swelling agents are added to the slurry or solution in step a) and / or any slurrying or dissolution step which is performed. Specific examples of swelling agents that may be employed in the process of the present invention include propylene glycols, polyethylene glycols, polyvinyl alcohols or polyacrylates.

In preferred embodiments of the present invention, prior to the commencement of step b), a slurry or solution comprising cellulose pulp may be subjected to drainage steps (to remove excess basic liquid, for example by pressing), shredding and / or aging steps. If performed, the drainage step and / or the shredding step will preferably result in a cellulose crumb product.

The alkali cellulose product obtained from any drainage step which is performed preferably includes about 32% or less of cellulose and / or about 16% or less alkali material (most preferably alkali metal hydroxide, such as sodium hydroxide).

Conventionally, xanthation is achieved by contacting alkali cellulose obtained from a dissolving pulp at a temperature of 30°C or higher with 26 to 29% carbon disulphide, by weight of the cellulose present. For example, alkali cellulose may be added to a
churn into which gaseous, liquid or aqueous carbon disulphide is introduced (churn xanthation).

The use of higher quantities of carbon disulphide has been previously found to be problematic as the rate of formation of a sodium thiosulphate, an unwanted by-product of a reaction between carbon disulphide and caustic solutions, has been found to increase, especially when xanthation is conducted at elevated temperatures.

While non-dissolving cellulose pulp (or crumb, slurries or solutions formed from such pulp) may be subjected to some conventional processing conditions in the xanthation step performed in the process of the present invention, it has unexpectedly been found that optimal results are obtained when the conventional processing conditions are adjusted.

More specifically, if non-dissolving cellulose pulp, or crumb, slurries or solutions formed from such pulp, are subjected to a xanthation step, this is preferably carried out at a temperature of about 30°C or lower, more preferably at about 20°C to about 30°C, about 22°C to about 28°C, about 24°C to about 26°C, or about 25°C to about 30°C.

Additionally or alternatively, said pulp, crumb, slurry or solution is preferably contacted with about 30% or more carbon disulphide, by weight of the cellulose present, more preferably about 30% to about 40%, about 30% to about 35% carbon disulphide, about 32% to about 38% carbon disulphide or about 34% to about 38%
carbon disulphide. It has unexpectedly been found that increasing the quantity of carbon disulphide used during xanthation reduces the viscosity of viscose solutions formed from the xanthated cellulose.

In step c) of the process of the present invention, dissolution is achieved by dissolving the non-dissolving pulp (or crumb, slurry or solution prepared therefrom) in a basic liquid, for example a caustic solution, typically comprising an alkali metal hydroxide such as sodium hydroxide. The liquid is preferably aqueous.

A quantity and concentration of basic liquid is preferably used which results in a viscose solution having a cellulose content (CIV) of about 5 to 15%, about 5 to 13%, about 5 to about 10%, or about 7 to about 10% and / or a caustic content (SIV) of about 2 to about 10%, about 4 to about 8%, about 5.5 to about 7.5%, or about 6 to about 7%

Dissolution is preferably carried out at a temperature of about 10°C to about 25°C, about 15°C to about 20°C, about 18°C to about 22°C or about 16°C to about 18°C.

In the process of the present invention, a mercerisation step may be performed. This will preferably be performed following steeping and / or prior to xanthation. The purpose of the mercerisation step is to improve the properties of the cellulose contained in the pulp, for example, by reducing the degree of polymerisation in the cellulose.
As conventionally practiced as part of the viscose process, the mercerisation step involves removing a portion of an alkali liquid (and optionally adding fresh alkali liquid) from a slurry or solution of dissolving cellulose pulp in alkali liquid to attain a target cellulose content and / or a target alkali or soda content, for example at least about 33% cellulose and / or at least about 16% alkali. Such processing conditions may be employed in the process of the present invention.

Alternatively, in the process of the present invention, different target cellulose and / or alkali contents may be preferable. More specifically, in preferred arrangements of the present invention, an alkali cellulose product optimally includes about 32% or less of cellulose and / or about 16% or less alkali material (most preferably alkali metal hydroxide, such as sodium hydroxide).

In alternative embodiments of the present invention, the mercerisation step can be excluded from the process. In one embodiment of the present invention, a slurry or solution containing cellulose, subjected to a steeping step (step a) is cooled to prevent mercerisation occurring. For example, the solution or mixture may be cooled to a temperature of about 50°C or less, about 40°C or less, about 30°C or less, or most preferably, to a temperature of about 25°C or less.

The viscose solution formed from a pulp processed according to the method of the present invention preferably exhibits a K value of at least 400, more preferably of at least 500, at least 600, or at least 700.
Additionally or alternatively, the viscose solution formed from a pulp processed according to the method of the present invention preferably exhibits one or more of the following characteristics: an Rv of about 200 or higher, more preferably about 500 or higher, a TVW of about 100 or higher and a fibre count of about 100 fibres / gram or lower, or more preferably 20 fibres / gram or lower.

Generally, as a result of the advantageous and optimised conditions practiced as part of the present invention, viscose solutions formed entirely from non-dissolving pulps may be prepared. However, those skilled in the art may nevertheless wish to produce viscose solutions from a mixture of dissolving and non-dissolving pulps. Accordingly, in preferred processes of the present invention, at least about 15% of the pulp used in the process of the present invention will be non-dissolving pulp. In other words, at least about 15% of the cellulose steeped in step (a) and / or present in the viscose solution formed in step (c) and / or present in the viscose solution from which the cellulose shaped article is cast in step (d) will be obtained from a non-dissolving pulp.

In more preferred embodiments, at least about 25%, about 50%, about 70% about 90% about 95% or even about 98% of the cellulose steeped in step (a) and / or present in the viscose solution formed in step (c) and / or present in the viscose solution from which the cellulose shaped article is cast in step (d) will be obtained from a non-dissolving pulp.

The process of the present invention further comprises the step of casting a cellulose shaped article from the viscose solution. The techniques for doing so will be well known to those skilled in the art. For example, in preferred embodiments, where the shaped articles are cellulose fibres, those fibres are preferably formed by extruding
the cellulose solution through a spinnerette, to produce a fibrous material. However, any fibre-forming techniques and apparatus may be employed.

Likewise, in embodiments of the present invention, where cellulose shaped articles other than fibres are prepared from the cellulose solution, the cellulose solution may be moulded, formed or shaped into the desired arrangement using conventional techniques known to those skilled in the art.

Additionally, in embodiments where cellulosic articles are formed from fibres prepared from the cellulose solution, the cellulose fibres may be converted into those articles using any techniques known to those skilled in the art.

The shaped cellulose solution is preferably then transferred into a casting bath including a casting solution.

To maximise the quality of viscose solutions prepared according to the process of the present invention, the cellulose-containing pulp employed therein may be subjected to one or more treatment steps in addition to steeping, xanthation dissolution and casting.

For example, the cellulose-containing pulp may be subjected to electron beam treatment. The use of electron beam radiation to treat cellulose-containing pulps is known and has been employed in commercial processes for many years. The treatment of pulps with electron beam radiation has typically been carried out by passing a layer or sheet of pulp by a fixed electron beam source, for example, using a conveyor.
Electron beam radiation may be emitted from any number of locations, for example, from one, two, three, four, five or six locations. However, in preferred aspects of the present invention, electron beam radiation is emitted from two locations, most preferably, from above and below the pulp. The electron beam radiation may be emitted from a single, fixed or movable electron beam source, or from a plurality of electron beam sources which may independently be fixed or movable. The emission of electron beam radiation from the plurality of locations may be simultaneous or sequential.

Preferably, the voltage of the electron beam radiation to which the non-dissolving pulp is exposed will be the same as or lower than that used in conventional electron beam treatment processes. For example, the electron beam radiation emitted from one, some or all of the locations will be about 10 meV or lower, about 5 meV or lower, about 1.5 meV or lower, about 1.0 meV or lower, about 800 keV or lower, about 600 keV or lower, about 500 keV or lower, about 400 keV or lower, about 300 keV or lower, about 250 keV or lower, or even about 200 keV or lower.

Any electron beam emitting apparatus known to those skilled in the art which is capable of emitting the required voltage and dose of radiation may be employed in the process of the present invention.

For example, medium voltage equipment capable of delivering doses of electron beam radiation at a voltage of 750 keV to 1.5 meV can be used. Treatment services
employing such equipment are offered by a range of companies, for example AquaMed. Such apparatus is advantageous as the pulp processing speeds (i.e. the treatment speeds) are high.

Additionally, or alternatively, low voltage equipment may be employed. Such equipment is typically used in coating curing systems and examples of such apparatus are provided, at least in the UK, by PCT Engineering Systems. The apparatus is capable of delivering curtain beam doses of electron beam radiation at an voltage of up to 300 keV. The width of the beam is adjustable depending on the area of pulp to be treated and treatment is achieved with an acceptable level of consistency. As a result of the emission of relatively low voltage electron beam radiation, minimal shielding is required, making the apparatus more manipulative for treating pulp from a plurality of locations and / or using a plurality of said devices in different locations simultaneously and / or sequentially without any risk to the user’s health.

In certain embodiments, treatment of the non-dissolving pulp with equal amounts of electron beam radiation from the different locations is preferred. However, in alternative arrangements, the amount of radiation emitted from the different locations varies by about 100 keV or lower, about 80 keV or lower, about 60 keV or lower, about 50 keV or lower, about 40 keV or lower, about 30keV or lower, about 20 keV or lower, about 10 keV or lower, or about 5 keV or lower.

The overall dose of electron beam radiation to which the pulp is exposed may vary. In preferred aspects of the present invention, the pulp is exposed to a dose of
electron beam radiation in the order of about 0.5 to about 5.0 mRad (about 5 to about 50 kgy), about 1.0 to about 4.0 mRad (about 10 to about 40 kgy), about 1.5 mRad to about 3.5 mRad (about 15 to about 35 kgy), about 2.0 mRad to about 3.0 mRad (about 20 to about 30 kgy), or about 1.0 to about 2.0 mRad (about 10 to about 20 kgy). In embodiments where the pulp is fed past electron beam/s to expose the pulp to radiation, the feed speed can be controlled to ensure that the target dose is administered.

The dose and voltage of radiation to which the non-dissolving pulp is exposed may be varied depending on the source, density, area, thickness and / or weight of the pulp being treated. The pulp which is treated in accordance with the present invention may have a thickness ranging from about 0.1mm, about 0.5 mm, about 1.0mm, about 5.0mm, or about 10.0mm to about 100mm, about 50mm, about 20mm, about 15mm, or about 10mm. The weight of the pulp which is treated in the process of the present invention may range from about 200 g/m², about 400 g/m², about 600 g/m², or about 700 g/m² to about 2000 g/m², about 1500 g/m², about 1200 g/m², about 1000 g/m², or about 800 g/m².

The non-dissolving pulp may be exposed to the electron beam radiation on a continuous basis (e.g. through use of conveyor means) or on a batch basis.

The treated pulp will preferably exhibit a degree of polymerisation of about 600 or lower, about 500 or lower, about 400 or lower, or most preferably about 300 or lower. This constitutes a significant improvement over the degree of polymerisation of untreated non-dissolving pulps, which typically is around 800 to 1400. Surprisingly,
these advantageous results can be obtained using significantly lower voltage and / or doses of radiation than have been employed conventionally.

Additionally or alternatively there are other treatment steps to which the pulp employed in the process of the present invention may be subjected.

For example, in the case of fluff pulps, these may be supplied in sheet form and it may be desirable to fluff or shred them, for example using a high shear mixer.

Additionally or alternatively, the pulp may be subjected to a drying step, for example, by placing it in an oven and heating it to a temperature of, e.g., about 40°C to 100°C.

As a further treatment step, pulp may be delaminated.

In a preferred embodiment of the present invention, the pulp is subjected to reduced temperature treatment. Without wishing to be bound by theory, it is believed that by exposing pulp to cold temperatures, the fibrous nature of the wood pulp is disrupted, potentially breaking hydrogen bonding and making the cellulose fibres more accessible.

Reduced temperature treatment is achieved by exposing the pulp to low temperatures, for example 0°C or lower, -50°C or lower, -100°C or lower, -150°C or lower or -180°C or lower.
Reduced temperature treatment can be effected by storing or holding the pulp in a low temperature environment, for example, an industrial freezer. Additionally or alternatively the pulp may be exposed to a low temperature agent, for example cryogenic liquid such as liquid nitrogen, liquid helium, liquid hydrogen, liquid oxygen, liquid neon or mixtures thereof. If a liquid low temperature agent is employed, the pulp is preferably saturated and / or submerged therein.

In preferred arrangements, the reduced temperature treatment has a duration of about 60 minutes or less, more preferably about 30 minutes or less, about 20 minutes or less, or about 15 minutes or less.

According to a second aspect of the present invention, there is provided a cellulose shaped article obtained according to the method of the first aspect of the present invention.

For the avoidance of any doubt, references to features of the first aspect of the present invention discussed above are optionally applicable to the product of the second aspect of the present invention, where appropriate.

The invention will now be illustrated in the following examples which demonstrate that the optimised viscose processing conditions of the present invention can be used to produce high-quality viscose solutions from non-dissolving pulps.

**Example 1 – Optimised Steeping Conditions – NaOH Concentration of Steeping Liquor**
The effect of the concentration of the alkali liquid used to steep cellulose-containing pulps was investigated. A softwood dissolving pulp was steeped in sodium hydroxide solutions having a concentration of 18.3% and 17.5% by weight for 30 minutes at 50°C. The resulting slurry was mercerised, xanthated and dissolved using conventional processing conditions.

The resulting viscose solution exhibited the following properties:

Table 1:

<table>
<thead>
<tr>
<th>Steeping NaOH Concentration (%)</th>
<th>Rv</th>
<th>Fibre Count / g</th>
<th>Viscosity – BFV (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>18.3</td>
<td>12.1</td>
<td>160+</td>
<td>78</td>
</tr>
<tr>
<td>17.5</td>
<td>289.9</td>
<td>77</td>
<td>51</td>
</tr>
</tbody>
</table>

Thus, as can be seen, a minor reduction in steeping liquor NaOH concentration has a significant effect on the quality of the resulting viscose solution.

**Example 2 – Optimised Steeping Conditions – NaOH Concentration of Steeping Liquor**

Viscose solutions were prepared according to the following processing conditions. As can be seen, the only variable was the concentration of the caustic solution used to steep the cellulose.

Weyerhauser Pearl pulp (a non-dissolving pulp) was steeped for 15 minutes at 40°C in soda having a caustic concentration of 16%, 16.8%, 17.1%, 17.5% and 18.3%
Xanthation was then carried out using 31% CS₂ by weight of the cellulose present, at a reaction temperature of 30°C. The xanthated cellulose was then dissolved to produce a viscose solution having a final target Soda in Viscose (SIV) content of 6.5%, and a Cellulose in Viscose (CIV) content of 9.3%. The viscose exhibited the following properties which are also depicted in the chart provided as Figure 1:

Table 2:

<table>
<thead>
<tr>
<th>Steeping NaOH (%)</th>
<th>Rv</th>
<th>TVW (g)</th>
<th>BFV(s)</th>
<th>Fibre Count (fibres/gram)</th>
</tr>
</thead>
<tbody>
<tr>
<td>18.3</td>
<td>40.4</td>
<td>98.8</td>
<td>44</td>
<td>54.3</td>
</tr>
<tr>
<td>17.5</td>
<td>59.6</td>
<td>105.2</td>
<td>51</td>
<td>43</td>
</tr>
<tr>
<td>17.1</td>
<td>82</td>
<td>131</td>
<td>50</td>
<td>65</td>
</tr>
<tr>
<td>16.8</td>
<td>141</td>
<td>151</td>
<td>53</td>
<td>4</td>
</tr>
<tr>
<td>16</td>
<td>48.4</td>
<td>81.2</td>
<td>43</td>
<td>12.5</td>
</tr>
</tbody>
</table>

Again, this data confirms that a minor reduction in the NaOH concentration of steeping liquors from those conventionally used to steep dissolving pulps has a significant effect on the quality of the viscose solutions prepared from non-dissolving pulps.

**Example 3 – Optimised Steeping Conditions – Steeping Temperature**

Viscose solutions were prepared in accordance with the following processing conditions. As can be seen, the only variable was the temperature at which steeping was conducted.

Weyerhauser Pearl pulp (a non-dissolving pulp) was steeped in a liquor having a concentration of 17.1% NaOH for 15 minutes at 20°C, 40°C, 50°C and 60°C. This
was then xanthated using 31% CS₂ by weight of cellulose present at a temperature of 30°C. The resulting product was then dissolved at 17.3°C to obtain a viscose solution having a Cellulose in Viscose (CIV) content of 9.3% and a Soda in Viscose (SIV) content of 6.5%.

Figure 2 confirms that steeping temperature has a significant effect on final viscose quality, with the optimum temperature being 40°C for the pulp tested.

**Example 4 – Optimised Steeping**

Viscose solutions were prepared from Weyerhaeuser Pearl pulp (a non-dissolving pulp) using the following processing conditions:

<table>
<thead>
<tr>
<th>Conditions Set No.</th>
<th>Steeping Conditions</th>
<th>Xanthation Conditions</th>
<th>Dissolution Conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>NaOH Concentration – 17.5% Duration – 15 Minutes Temperature - 40°C</td>
<td>Percentage of CS₂ – 31 Temperature - 30°C</td>
<td>CIV – 9.3 SIV – 6.5</td>
</tr>
<tr>
<td>2</td>
<td>NaOH Concentration – 16.8% Duration – 15 Minutes Temperature - 40°C</td>
<td></td>
<td></td>
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<tr>
<td>3</td>
<td>NaOH Concentration – 17.1% Duration – 15 Minutes Temperature - 40°C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>NaOH Concentration – 16% Duration – 15 Minutes Temperature - 40°C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>NaOH Concentration – 18.3% Duration – 15 Minutes Temperature - 40°C</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The effect of the varying conditions on the properties of the resulting viscose solutions, presented in terms of their filtered weight shown on a 5 minute interval basis and cumulatively are provided as Figures 3a and 3b.
Example 5 – Optimised Xanthation – Amount of Carbon Disulphide

Viscose solutions were prepared in accordance with the following processing conditions. As can be seen, the only variable was the quantity of carbon disulphide employed in the xanthation step.

Weyerhauser Pearl pulp (a non-dissolving pulp) was steeped in a liquor having a concentration of 17.5% NaOH at 40°C for 15 minutes. This was then xanthated using 26, 31, 33 and 36% CS₂ by weight of cellulose present at a temperature of 30°C. The resulting product was then dissolved at 17.3°C to obtain a viscose solution having a Cellulose in Viscose (CIV) content of 9.3% and a Soda in Viscose (SIV) content of 6.5%.

The Rv, TVW and BFV values of the resulting viscose solutions are provided in Figure 4. As can be seen, increasing the amount of carbon disulphide beyond conventionally used levels advantageously resulted in an increase in TVW and Rv and a decrease in BFV.

Example 6 – Optimised Xanthation - Xanthation Temperature

Viscose solutions were prepared in accordance with the following processing conditions. As can be seen, the only variable was the temperature at which the xanthation step was conducted.
Weyerhauser Pearl pulp (a non-dissolving pulp) was steeped in a liquor having a concentration of 17.5% NaOH at 40°C for 15 minutes. This was then xanthated using 31% CS₂ by weight of cellulose present at temperatures of 25°C, 30°C and 40°C. The resulting product was then dissolved at 17.3°C to obtain a viscose solution having a Cellulose in Viscose (CIV) content of 9.3% and a Soda in Viscose (SIV) content of 6.5%.

The Rv, TVW and BFV values of the resulting viscose solutions are provided in Figure 5. As can be seen, performing xanthation at a temperature lower than those used conventionally for dissolving pulps resulted in an unexpected increase in TVW and Rv and a decrease in BFV.

**Example 7 – Optimised Xanthation**

Viscose solutions were prepared from Weyerhauser Pearl pulp (a non-dissolving pulp) using the following processing conditions:

**Table 4:**

<table>
<thead>
<tr>
<th>Conditions Set No.</th>
<th>Steeping Conditions</th>
<th>Xanthation Conditions</th>
<th>Dissolution Conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>NaOH Concentration – 17.5%</td>
<td>Percentage of CS₂ – 29.3 Temperature - 35°C</td>
<td>CIV – 9.3</td>
</tr>
<tr>
<td>2</td>
<td>Duration – 15 Minutes</td>
<td>Percentage of CS₂ – 31 Temperature - 30°C</td>
<td>SIV – 6.5</td>
</tr>
<tr>
<td>3</td>
<td>Temperature - 40°C</td>
<td>Percentage of CS₂ – 36 Temperature - 40°C</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td></td>
<td>Percentage of CS₂ – 36 Temperature - 30°C</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td></td>
<td>Percentage of CS₂ – 31 Temperature - 25°C</td>
<td></td>
</tr>
</tbody>
</table>
The effect of the varying conditions on the properties of the resulting viscose solutions, presented in terms of their filtered weight shown on a 5 minute interval basis and cumulatively are provided as Figures 6a and 6b.

Example 8 – Optimised Dissolution - SIV Content

Viscose solutions were prepared in accordance with the following processing conditions. As can be seen, the only variable was the SIV content of the viscose.

Weyerhauser Pearl pulp (a non-dissolving pulp) was steeped in a liquor having a concentration of 17.5% NaOH at 40°C for 15 minutes. This was then xanthated using 31% CS₂ by weight of cellulose present at a temperature of 25°C. The resulting product was then dissolved at 17.3°C to obtain a viscose solution having a Cellulose in Viscose (CIV) content of 9.3% and a Soda in Viscose (SIV) content of 5.5, 6, 6.5 and 7%

The Rv, TVW, BFV and fibre count values of the resulting viscose solutions are provided in Figure 7. As can be seen, an SIV content of around 6.5% results in optimal Rv, TVW and fibre count values for the viscose solutions tested.

Example 9 – Optimised Dissolution - SIV Content

Viscose solutions were prepared from Weyerhauser Pearl pulp (a non-dissolving pulp) using the following processing conditions:
Table 5:

<table>
<thead>
<tr>
<th>Conditions Set No.</th>
<th>Steeping Conditions</th>
<th>Xanthation Conditions</th>
<th>Dissolution Conditions</th>
</tr>
</thead>
</table>
| 1                  | NaOH Concentration – 17.5% | Percentage of CS₂ – 31 | CIV – 9.3  
|                    | Duration – 15 Minutes   | Temperature - 25°C     | SIV – 6          |
| 2                  |                      |                       | CIV – 9.3  
|                    |                      |                       | SIV – 6.5       |
| 3                  |                      |                       | CIV – 9.3  
|                    |                      |                       | SIV – 5.5       |
| 4                  |                      |                       | CIV – 9.3  
|                    |                      |                       | SIV – 7         |

The effect of the varying conditions on the properties of the resulting viscose solutions, presented in terms of their filtered weight shown on a 5 minute interval basis and cumulatively are provided as Figures 8a and 8b.

**Example 10 – Optimised Dissolution - Dissolution Temperature**

Viscose solutions were prepared in accordance with the following processing conditions. As can be seen, the only variable was the temperature at which dissolution occurred.

Weyerhauser Pearl pulp (a non-dissolving pulp) was steeped in a liquor having a concentration of 17.1% NaOH at 40°C for 15 minutes. This was then xanthated using 31% CS₂ by weight of cellulose present at a temperature of 25°C. The resulting product was then dissolved at 10°C, 17.3°C and 30°C to obtain a viscose solution having a Cellulose in Viscose (CIV) content of 9.3% and a Soda in Viscose (SIV) content of 6.5%
The Rv and TVW values of the resulting viscose solutions are provided in Figure 9. As can be seen, a dissolution temperature of around 17°C results in optimal Rv and TVW values for the viscose solutions tested.

**Summary of Examples 1 to 10**

Data reported in a number of the examples above is incorporated into the summary table provided as Figure 10. The best and worst Rv and VFW results obtained in those tests are shown on a test by test basis, and as can be seen, there is a clear improvement when the optimized processing conditions of the present invention are employed.

Importantly, in the first category (‘Overall’) a comparison is made between the best results obtained using the optimized processing conditions of the present invention and conventional processing conditions (as used with dissolving pulps) when used with non-dissolving pulp. There is clearly a significant improvement observed when the optimized processing conditions of the present invention are employed; an increase in Rv from 13.9 to 237.5 and an increase in TVW from 18.1 to 184.5. Thus, by employing the processing conditions of the present invention, it is advantageously now possible, for the first time, to prepare high quality viscose solutions suitable for forming cellulose shaped articles from non-dissolving pulps.

**Example 11 – Effect of Hemicellulose on Viscose Solution**
89.2g of Weyerhauser Pearl pulp (a non-dissolving pulp) was slurried with 1800mls of NaOH at varying temperatures, times and concentrations. The slurry was then pressed using a slurry press at 7 tonnes for 30 seconds. The excess NaOH was collected, weighed and tested for hemi-cellulose content.

The main variables studied in these tests were steeping temperature and the steeping caustic concentration and the results of this experimentation are provided as Figures 11a and 11b. As can be seen, steeping non-dissolving pulp at 40°C using a caustic solution having a concentration around 17% resulted in the lowest proportion of hemicellulose being extracted from the pulp.

Previously, it was thought that hemicellulose had a negative effect on the filterability of viscose solutions. However, from comparing the results shown in Figures 11a and 11b with the results shown in Figures 1 and 2, it can be seen that the steeping conditions which resulted in the removal of the lowest proportion of hemicellulose corresponded to the steeping conditions which resulted in viscose solutions exhibiting the best filterability.
CLAIMS

1. A process for producing cellulose shaped articles comprising the steps of:
   a. Steeping a non-dissolving cellulose-containing pulp in a basic liquid,
   b. Xanthating the steeped cellulose-containing pulp with carbon disulphide,
   c. Dissolving the xanthated cellulose-containing pulp in a basic liquid to form a viscose solution, and
   d. Casting a cellulose shaped article from the viscose solution,

wherein one or more of the following optimised processing conditions are employed:

- Steeping is carried out, at least partially, at a temperature of about 30°C to about 50°C,
- Steeping is carried out in a basic liquid having a concentration of base of about 16 to about 18% by weight,
- Xanthation is carried out using about 30 to about 40% carbon disulphide by weight of cellulose present,
- Xanthation is carried out, at least partially, at a temperature of about 20°C to about 30°C,
- Dissolution is carried out, at least partially, at a temperature of about 10°C to about 25°C,
- Dissolution results in a viscose solution having a base concentration of between about 5.5 and 7.5% by weight of the solution, and / or
- Dissolution results in a viscose solution having a cellulose concentration of between about 8.5 and about 9.5% by weight of the solution
2. The method of Claim 1, wherein steeping is carried out in a basic liquid having a concentration of base of about 16.5% to about 17.5% by weight.

3. The method of Claim 1 or Claim 2, wherein steeping is carried out, at least partially, at a temperature of about 40°C to about 48°C.

4. The method of any one of Claims 1 to 3, wherein steeping is carried out, at least partially, at a temperature of about 35°C to about 45°C.

5. The method of any one of Claims 1 to 4, wherein steeping is carried out in a slurry or solution which is substantially free of manganese.

6. The method of any one of Claims 1 to 5, wherein steeping is carried out in a slurry or solution which comprises a steeping additive.

7. The method of Claim 6, wherein the steeping additive is ethoxylated alcohol.

8. The method of Claim 6 or 7, wherein the steeping additive is Berol 388.

9. The method of any one of Claims 1 to 8, wherein prior to xanthation, the cellulose-containing pulp, or a slurry or solution prepared therefrom, is subjected to one or more of a drainage step, a shredding step and or an aging step.
10. The method of any one of Claims 1 to 9, wherein xanthation is carried out using about 32 to about 38% carbon disulphide by weight of cellulose present.

11. The method of any one of Claims 1 to 10, wherein the viscose solution prepared in step c) has a cellulose content of about 5% to about 10% by weight of the solution.

12. The method of any one of Claims 1 to 11, wherein dissolution is carried out, at least partially, at a temperature of about 15°C to about 20°C.

13. The method of any one of Claims 1 to 12, wherein upon completion of step a), the temperature of the slurry or solution is reduced to prevent mercerisation occurring.

14. The method of any one of Claims 1 to 13, wherein the viscose solution formed in step c) exhibits one or more of: an Rv of about 200 or higher, a TVW of about 100 or higher and / or a fibre count of about 100 fibres / gram or lower.

15. The method of any one of Claims 1 to 14, wherein the viscose solution formed in step c) exhibits one or more of: an Rv of about 500 or higher and / or a fibre count of 20 fibres / gram or lower.

16. The method of any one of Claims 1 to 15, wherein the viscose solution formed in step c) exhibits a K value of at least about 400.
17. The method of any one of Claims 1 to 16, wherein the viscose solution formed in step c) exhibits a K value of at least about 600.

18. The method of any one of Claims 1 to 17, wherein prior to step a), the pulp is subjected to one or more additional treatment steps selected from electron beam irradiation, fluffing, shredding, drying, delamination and / or reduced temperature treatment.

19. The method of Claim 18, wherein electron beam irradiation comprises exposing non-dissolving cellulose-containing pulp to electron beam radiation.

20. The method of Claim 19, wherein the electron beam radiation originates from a plurality of locations.

21. The method of Claim 19 or Claim 20, wherein electron beam radiation is emitted from two locations.

22. The method of any one of Claims 19 to 21, wherein electron beam radiation is emitted from locations above and below the pulp.

23. The method of any one of Claims 19 to 22, wherein the voltage of the electron beam radiation emitted at one or more of the plurality of the locations is about 1.5 meV or lower.
24. The method of any one of Claims 19 to 23, wherein the voltage of the electron beam radiation emitted at one or more of the plurality of the locations is about 500 keV or lower.

25. The method of any one of Claims 20 to 24, wherein the voltage of the electron beam radiation emitted from the plurality of locations varies by about 50 keV or lower.

26. The method of any one of Claims 19 to 25, wherein the dose of electron beam radiation to which the pulp is exposed is about 0.5 mRad to about 5.0 mRad.

27. The method of any one of Claims 19 to 26, wherein the dose of electron beam radiation to which the pulp is exposed is about 1.5 mRad to about 3.5 mRad.

28. The method of any one of Claims 19 to 27, wherein the pulp has a thickness of about 0.1mm to about 100mm.

29. The method of any one of Claims 19 to 28, wherein the pulp has a weight of about 200 g/m² to about 1200 g/m².

30. The method of any one of Claims 19 to 29, wherein, following exposure to electron beam radiation, the pulp exhibits an average degree of polymerisation of about 500 or lower.
31. The method of any one of Claims 19 to 30, wherein, following exposure to electron beam radiation, the pulp exhibits an average degree of polymerisation of about 400 or lower.

32. The method of any one of Claims 1 to 31, wherein at least about 15% of the cellulose steeped in step (a) and / or present in the solution formed in step (c) and / or present in the solution from which the cellulose shaped article is cast in step (d) will be obtained from a treated non-dissolving pulp.

33. The method of any one of Claims 1 to 32, wherein at least about 50% of the cellulose steeped in step (a) and / or present in the solution formed in step (c) and / or present in the solution from which the cellulose shaped article is cast in step (d) will be obtained from a treated non-dissolving pulp.

34. The method of any one of Claims 1 to 33, wherein at least about 90% of the cellulose steeped in step (a) and / or present in the solution formed in step (c) and / or present in the solution from which the cellulose shaped article is cast in step (d) will be obtained from a treated non-dissolving pulp.

35. The process of any one of Claims 1 to 34, wherein the shaped articles are fibres.

36. The process of any one of Claims 1 to 34, wherein the shaped articles are ropes, yarns, cloths or cigarette filters.
37. The process of Claim 35, further comprising the step of forming the cellulose fibres cast in step d) into an article.

38. The process of Claim 37, wherein the article is a rope, yarn, cloth or cigarette filter.

39. A cellulose shaped article obtained from the process of any one of Claims 1 to 38.
Patents Act 1977: Search Report under Section 17

Documents considered to be relevant:

<table>
<thead>
<tr>
<th>Category</th>
<th>Relevant to claims</th>
<th>Identity of document and passage or figure of particular relevance</th>
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<td>X</td>
<td>1 and 39 at least</td>
<td>GB 999587 A BRODY See whole document and especially example 1.</td>
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<td>X</td>
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<td>GB 367920 A LILIENFELD See whole document and especially example 6.</td>
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<td>GB 318255 A SOCIETY OF CHEMIICAL INDUSTRY INT BASLE See whole document and especially examples 2 &amp; 3.</td>
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<td>X</td>
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<td>US 3438969 A MEISTER See whole document and especially example 1.</td>
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Categories:

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<th>Document indicating technological background and/or state of the art</th>
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<tr>
<td>Y Document indicating lack of inventive step if combined with one or more other documents of same category</td>
<td>P Document published on or after the declared priority date but before the filing date of this invention.</td>
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<tr>
<td>&amp; Member of the same patent family</td>
<td>E Patent document published on or after, but with priority date earlier than, the filing date of this application.</td>
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Field of Search:
Search of GB, EP, WO & US patent documents classified in the following areas of the UKC^X: 
Worldwide search of patent documents classified in the following areas of the IPC

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The following online and other databases have been used in the preparation of this search report:

WPI, EPODOC