DESCRIPTION

Field of the invention

The invention relates to biodegradable super-absorbent polymers (SAPs), their manufacture and their use in absorbent products for bodily fluids such as baby diapers, adult incontinence articles, feminine-hygiene pads and wound dressings.

Background of the invention

Commercial SAPs for hygiene products are almost exclusively cross-linked poly-acrylates. They are efficient, but they are neither biodegradable nor perceived as soft in the finished article. Document D2 reports that cores based on poly-acrylate SAPs and cellulose fibres score only 10 - 20 on a 100-point softness scale, whereas higher scores are preferred by consumers . Biodegradable SAPs based on sodium-carboxymethyl-cellulose (CMCNa) and hydroxy-ethyl-cellulose (HEC) cross-linked with agents such as epichlorohydrin, formaldehyde or carbodiimides are known from Document D1, but they are aimed at soil-conditioning, not at hygiene products, and their absorption capacity ($\leq$ 39 g H_2O / g polymer) is too low for modern diapers .

Object of the invention

It is an object of the invention to provide a biodegradable SAP that

- matches or exceeds the liquid-handling performance of leading poly-acrylates,
- delivers higher perceived softness in the article, and
- can be produced economically on an industrial scale.

Summary of the invention

The object is achieved by a cross-linked CMCNa/HEC polymer whose **CMCNa : HEC weight ratio is 2.0 – 3.2** and whose **absorption capacity is at least 50 g H_2O / g polymer**.

Key process features are:

1. cross-linking in aqueous solution with **5 – 15 wt % of a carbodiimide** (preferably 7 wt % 1-ethyl-3-(3-dimethyl-aminopropyl)-carbodiimide, EDC) at **pH 3 – 6** (preferably 3.5 – 4.5) in the presence of an acid catalyst such as citric acid;
2. washing the resulting gel with **de-ionised water**;
3. **phase-inversion drying** (e.g. in acetone) to granules;
4. a resulting **degree of cross-linking of 2 – 10 %** (Italian Std. ITNA0011, 1990).

The polymer is blended with cellulosic fibres (preferably 10 – 50 wt % polymer / balance fibres) to form an absorbent core which is then laminated between a liquid-impermeable back-sheet and a non-woven top-sheet. The finished article shows a perceived softness $\geq$ 50, typically 70 – 85, clearly above articles containing commercial poly-acrylates.

Detailed description

Preparation of the SAP

An aqueous solution containing 5 wt % total CMCNa + HEC is adjusted to the desired CMCNa : HEC ratio (see Table 1). EDC (7 wt %) and citric acid are added, giving pH 4.0. After gel formation the material is washed with de-ionised water and poured into acetone until white granules precipitate. Dry granules are sieved to 100 – 850 µm.

Table 1 – Properties of representative polymers

Sample	CMCNa : HEC	Absorption (g/g)	Water retention (%)	Shear modulus (MPa)
4	1.2	39	65	3.0
8	**2.0**	**60**	**70**	3.0
10	**2.4**	**69**	**72**	2.8
12	**2.8**	**70**	**69**	3.2

Samples with a ratio ≥ 2.0 meet or exceed the performance of commercial poly-acrylate Superwet A1000 (45 g/g; 65 % water retention) while being fully biodegradable .

Manufacture of the absorbent core and article

Cotton, flax or rayon fibres are conveyed in hot air onto a rotating vacuum drum. SAP granules (sample 8 or 12) are injected into the fibre stream, giving a core containing 30 wt % SAP. The sheet is laminated between an impermeable back-sheet and a spun-bond non-woven top-sheet directly at drum exit.

Finished diapers were tested by a 40-person panel. Articles with the inventive SAP scored 72 – 80 on the softness scale versus 31 for articles containing Superwet A1000, while matching or surpassing them in absorbency and retention .

Advantages

- Performance (absorption & retention) comparable to or better than leading poly-acrylates.
- Perceived softness far superior, enabling premium positioning.
- Fully biodegradable chemistry, easing disposal concerns and regulatory pressure.
- Process window (specific carbodiimide level + phase-inversion drying) gives robust, mechanically strong granules.

CLAIMS

1. **Absorbent product** comprising
   a liquid-impermeable first layer,
   a second layer of non-woven material, and
   an absorbent core arranged between the layers,
   the core comprising (i) cellulosic fibres and (ii) granules of a biodegradable super-absorbent polymer (SAP),

wherein the SAP is a cross-linked mixture of sodium carboxy-methyl-cellulose (CMCNa) and hydroxy-ethyl-cellulose (HEC)

- the weight ratio CMCNa : HEC being 2.0 – 3.2,

- the polymer having an absorption capacity of at least 50 g water per g polymer and a water-retention value of at least 60 %, and

- the cross-linking having been carried out with 5 – 15 wt % of a carbodiimide.

2. Product according to claim 1, wherein the carbodiimide is 1-ethyl-3-(3-dimethyl-aminopropyl)-carbodiimide (EDC).
3. Product according to any of claims 1-2, wherein the degree of cross-linking of the SAP is 2 – 10 %.
4. Product according to any preceding claim, wherein the absorbent core contains 10 – 50 wt % of the SAP and 50 – 90 wt % cellulosic fibres selected from cotton, flax and rayon.
5. Product according to any preceding claim, exhibiting a perceived softness of at least 50 on a 1-100 scale determined by a panel test.
6. **Biodegradable super-absorbent polymer** consisting of a cross-linked mixture of CMCNa and HEC,

   wherein

   a) the CMCNa : HEC weight ratio is 2.0 – 3.2,

   b) the absorption capacity is ≥ 50 g water per g polymer,

   c) the water retention is ≥ 60 %, and

   d) the degree of cross-linking is 2 – 10 %.
7. Polymer according to claim 6, wherein cross-linking has been performed with 5 – 10 wt % EDC at pH 3.5 – 4.5 in the presence of citric acid.
8. Polymer according to claim 6 or 7, obtainable by the method of claim 11.
9. **Absorbent core** comprising 10 – 50 wt % of the polymer of any of claims 6-8 and 50 – 90 wt % cellulosic fibres.
10. Use of the polymer of any of claims 6-8 for producing an absorbent product for absorbing bodily fluids.
11. **Method for producing the polymer** of claim 6, comprising the sequential steps of

    (i) reacting an aqueous solution containing 3 – 10 wt % (CMCNa + HEC) whose CMCNa : HEC weight ratio is 2.0 – 3.2 with 5 – 15 wt % of a carbodiimide cross-linking agent at pH 3 – 6 in the presence of an acid catalyst to obtain a gel,

    (ii) washing the gel with de-ionised water, and

    (iii) drying the gel by phase inversion in a liquid that is a non-solvent for the polymer and a solvent for water, to obtain granules.

12. Method according to claim 11, wherein the phase-inversion liquid is acetone.
13. The polymer produced by the method of claim 11 or 12.
14. **Use** of an absorbent core according to claim 9 in a diaper, adult incontinence article, sanitary napkin or wound dressing.
15. **Absorbent product** obtainable by laminating a core according to claim 9 between a liquid-impermeable back-sheet and a non-woven top-sheet.