



(11) **EP 2 712 936 A1**

(12) **EUROPEAN PATENT APPLICATION**

(43) Date of publication:  
**02.04.2014 Bulletin 2014/14**

(51) Int Cl.:  
**C13K 1/02 (2006.01) C12P 19/02 (2006.01)**

(21) Application number: **13186469.6**

(22) Date of filing: **27.09.2013**

(84) Designated Contracting States:  
**AL AT BE BG CH CY CZ DE DK EE ES FI FR GB  
GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO  
PL PT RO RS SE SI SK SM TR**  
Designated Extension States:  
**BA ME**

(30) Priority: **28.09.2012 US 201261707576 P**  
**22.08.2013 US 201313973072**  
**23.09.2013 CN 201310435004**

(71) Applicant: **Industrial Technology Research  
Institute  
Hsinchu 31040 (TW)**

(72) Inventors:  
• **Shih, Ruey-Fu**  
**241 New Taipei City (TW)**

- **Chen, Jia-Yuan**  
**300 Hsinchu City (TW)**
- **Lin, Hui-Tsung**  
**241 New Taipei City (TW)**
- **Lee, Hom-Ti**  
**Zhunei City 302 Hsinchu County (TW)**
- **Wan, Hou-Peng**  
**333 Taoyuan County (TW)**
- **Hung, Wei-Chun**  
**238 New Taipei City (TW)**

(74) Representative: **Rieck, Markus  
Fuchs  
Patentanwälte  
Westhafenplatz 1  
60327 Frankfurt am Main (DE)**

(54) **Sugar products and fabrication method thereof**

(57) In an embodiment of the present disclosure, a sugar product and method for fabricating the same is provided. The method includes mixing an acid compound and lithium chloride, magnesium chloride, calcium chloride, zinc chloride or iron chloride or lithium bromide, magnesium bromide, calcium bromide, zinc bromide or iron bromide or heteropoly acid to form a mixing solution,

adding a cellulosic biomass to the mixing solution for a dissolution reaction, and adding water to the mixing solution for a hydrolysis reaction to obtain a sugar product. The present disclosure also provides a sugar product fabricated from the method.

**EP 2 712 936 A1**

**Description****CROSS REFERENCE TO RELATED APPLICATIONS**

5 [0001] This Application claims priority of China Patent Application No. 2013104350048, filed on Sep 23, 2013. This application claims the benefit of U.S. Application No. 13/973,072, filed on Aug 22, 2013, which claims the benefit of provisional Application No. 61/707,576, filed on Sep 28, 2012, the entireties of which are incorporated by reference herein.

**TECHNICAL FIELD**

10

[0002] The technical field relates to a sugar product and fabricating method thereof.

**BACKGROUND**

15 [0003] The world is facing problems such as the gradual extraction and depletion of petroleum reserves, and changes to the earth's atmosphere due to the greenhouse effect. In order to ensure the sustainability of human life, it has become a world trend to gradually decrease the use of petrochemical energy and petroleum feedstock and to develop new sources of renewable energy and materials.

20 [0004] Lignocellulose is the main ingredient of biomass, which is the most abundant organic substance in the world. Lignocellulose mainly consists of 38-50% cellulose, 23-32% hemicellulose and 15-25% lignin. Cellulose generates glucose through hydrolysis. However, it is difficult for chemicals to enter the interior of cellulose molecules for depolymerization due to strong intermolecular and intramolecular hydrogen bonding and Van de Waal forces and the complex aggregate structure of cellulose with high-degree crystallinity. The main methods of hydrolyzing cellulose are enzyme hydrolysis and acid hydrolysis. However, there is significant imperfection in these two technologies, therefore, it is difficult

25

[0005] Generally speaking, enzyme hydrolysis can be carried out at room temperature, which is an environmentally friendly method due to the rarity of byproducts, no production of anti-sugar fermentation substances, and integration with the fermentation process. However, a complicated pretreatment process is required, hydrolytic activity is low, the reaction rate is slow, and cellulose hydrolysis enzyme is expensive.

30

[0006] Dilute acid hydrolysis generally uses comparatively cheap sulfuric acid as a catalyst, but it must operate in a corrosion-resistant pressure vessel at more than 200°C, requiring high-level equipment; simultaneously, the temperature of the dilute acid hydrolysis is high, the byproduct thereof is plentiful, and the sugar yield is low. Concentrated acid hydrolysis can operate at lower temperature and normal pressure. However, there are problems of strong corrosivity of concentrated acid, complications in the post-treatment process of the hydrolyzed solution, large consumption of acid, and difficulties with recycling, among other drawbacks.

35

**SUMMARY**

40 [0007] One embodiment of the disclosure provides a sugar product, comprising: a sugar mixture comprising glucose, xylose, mannose, arabinose and oligosaccharides thereof with a weight ratio of 2-15wt%; an acid compound with a weight ratio of 48-97wt%; and a salt compound with a weight ratio of 1-50wt%.

[0008] One embodiment of the disclosure provides a method for fabricating a sugar product, comprising: mixing formic acid or acetic acid and lithium chloride, magnesium chloride, calcium chloride, zinc chloride, iron chloride, lithium bromide, magnesium bromide, calcium bromide, zinc bromide, iron bromide, or heteropoly acid to form a mixing solution; adding

45

[0009] A detailed description is given in the following embodiments.

**DETAILED DESCRIPTION**

50

[0010] In the following detailed description, for purposes of explanation, numerous specific details are set forth in order to provide a thorough understanding of the disclosed embodiments. It will be apparent, however, that one or more embodiments may be practiced without these specific details. In other instances, well-known structures and devices are schematically shown in order to simplify the drawing.

55

[0011] In one embodiment of the disclosure, a sugar product is provided. The sugar product comprises a sugar mixture, an acid compound, and a salt compound. The sugar mixture comprises glucose, xylose, mannose, arabinose and oligosaccharides thereof with a weight ratio of about 2-15wt% in the sugar product. The acid compound may comprise formic acid or acetic acid with a weight ratio of about 48-97wt% in the sugar product. The salt compound may comprise

lithium chloride, magnesium chloride, calcium chloride, zinc chloride, iron chloride, lithium bromide, magnesium bromide, calcium bromide, zinc bromide, or iron bromide with a weight ratio of about 1-5wt% in the sugar product.

**[0012]** In one embodiment of the disclosure, a method for fabricating a sugar product is provided, comprising the following steps. First, formic acid or acetic acid and lithium chloride, magnesium chloride, calcium chloride, zinc chloride, iron chloride, lithium bromide, magnesium bromide, calcium bromide, zinc bromide, iron bromide, or heteropoly acid are mixed to form a mixing solution. A cellulosic biomass is added to the mixing solution for a dissolution reaction. Water is added to the mixing solution for a hydrolysis reaction to obtain a sugar product.

**[0013]** The formic acid has a weight ratio of about 50-97wt% in the mixing solution.

**[0014]** The lithium chloride or lithium bromide has a weight ratio of about 5-20wt% or 10-20wt% in the mixing solution.

**[0015]** The magnesium chloride or magnesium bromide has a weight ratio of about 10-30wt% or 15-20wt% in the mixing solution.

**[0016]** The calcium chloride or calcium bromide has a weight ratio of about 12-40wt% or 12-30wt% in the mixing solution.

**[0017]** The zinc chloride or zinc bromide has a weight ratio of about 5-45wt% or 20-30wt% in the mixing solution.

**[0018]** The iron chloride or iron bromide has a weight ratio of about 1-50wt% or 5-10wt% in the mixing solution.

**[0019]** The heteropoly acid may comprise  $H_3PW_{12}O_{40}$ ,  $H_4SiW_{12}O_{40}$ ,  $H_3PMo_{12}O_{40}$  or  $H_4SiMo_{12}O_{40}$  with a weight ratio of about 1-5wt% or 2-5wt% in the mixing solution.

**[0020]** The cellulosic biomass may be derived from wood, grass, leaves, algae, waste paper, corn stalks, corn cobs, rice straw, rice husk, wheat straw, bagasse, bamboo, or crop stems. The cellulosic biomass may comprise cellulose, hemicellulose, or lignin with a weight ratio of about 1-20wt% or 5-15wt% in the mixing solution.

**[0021]** The dissolution reaction has a reaction temperature of about 40-90 or 50-70 and a reaction time of about 20-360 minutes or 30-120 minutes.

**[0022]** In the hydrolysis reaction, the amount of water added is larger than the total molar equivalent of monosaccharides hydrolyzed from the cellulosic biomass.

**[0023]** The hydrolysis reaction has a reaction temperature of about 50-150°C or 60-105 °C and a reaction time of about 30-180 minutes or 30-120 minutes.

**[0024]** The sugar product fabricated by the method may comprise a sugar mixture, an acid compound, and a salt compound. The sugar mixture may comprise glucose, xylose, mannose, arabinose and oligosaccharides thereof with a weight ratio of about 2-15wt% in the sugar product. The acid compound may comprise formic acid or acetic acid with a weight ratio of about 48-97wt% in the sugar product. The salt compound may comprise lithium chloride, magnesium chloride, calcium chloride, zinc chloride, iron chloride, lithium bromide, magnesium bromide, calcium bromide, zinc bromide, or iron bromide with a weight ratio of about 1-50wt% in the sugar product.

**[0025]** In one embodiment, the method further comprises adding inorganic acid to the mixing solution before, during or after the dissolution reaction. The inorganic acid may comprise sulfuric acid or hydrochloric acid. The inorganic acid has a weight ratio of about 1-2wt% in the mixing solution. When the inorganic acid is added, the adding amount of the chloride salt or the bromide salt may be reduced, for example, the weight ratio of the magnesium chloride, the magnesium bromide, the calcium chloride or the calcium bromide in the mixing solution may be reduced to about 1-10wt%, and the weight ratio of the lithium chloride, the lithium bromide, the zinc chloride, the zinc bromide, the iron chloride or the iron bromide in the mixing solution may be reduced to about 1-5wt%.

**[0026]** In the disclosure, formic acid or acetic acid (weak acid) is mixed with lithium chloride, magnesium chloride, calcium chloride, zinc chloride, iron chloride, lithium bromide, magnesium bromide, calcium bromide, zinc bromide, or iron bromide to be utilized as a solvent with the characteristic of dissolving cellulose under low temperature (<90°C) and rapid reaction time (<6 hours) to generate a homogeneous liquid. In the disclosed method, cellulose is dissolved in the solvent formed by chloride salt or bromide salt and formic acid or acetic acid to generate a homogeneous liquid at 40-150°C, and a sugar product is further obtained through hydrolysis. This method achieves the technical goals of low temperature, normal pressure, rapid reaction time and high sugar yield and without use of a strong acid corrosion-resistant reactor.

**[0027]** Examples

**[0028]** Example 1-1

**[0029]** Formic acid and zinc chloride ( $ZnCl_2$ ) were mixed and heated to form a mixing solution (60wt% of formic acid, 40wt% of zinc chloride). Avicel®cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (15wt% of Avicel®cellulose) for a dissolution reaction (50 , 20 minutes) to form a yellow, homogeneous, and transparent liquid, as recorded in Table 1.

**[0030]** Example 1-2

**[0031]** Formic acid and zinc chloride ( $ZnCl_2$ ) were mixed and heated to form a mixing solution (60wt% of formic acid, 40wt% of zinc chloride).  $\alpha$ -cellulose (Sigma Corporation, C8002) was added to the mixing solution (15wt% of  $\alpha$ -cellulose) for a dissolution reaction (50°C, 20 minutes) to form an amber, homogeneous, and transparent liquid, as recorded in Table 1.

**[0032]** Example 1-3

## EP 2 712 936 A1

**[0033]** Formic acid and calcium chloride (CaCl<sub>2</sub>) were mixed and heated to form a mixing solution (75wt% of formic acid, 25wt% of calcium chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (6wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (65 °C , 90 minutes) to form a yellow, homogeneous, and transparent liquid, as recorded in Table 1.

**[0034]** Example 1-4

**[0035]** Formic acid and calcium chloride (CaCl<sub>2</sub>) were mixed and heated to form a mixing solution (75wt% of formic acid, 25wt% of calcium chloride).  $\alpha$ -cellulose (Sigma Corporation, C8002) was added to the mixing solution (6wt% of  $\alpha$ -cellulose) for a dissolution reaction (65°C, 90 minutes) to form an amber, homogeneous, and transparent liquid, as recorded in Table 1.

**[0036]** Example 1-5

**[0037]** Formic acid and magnesium chloride (MgCl<sub>2</sub>) were mixed and heated to form a mixing solution (80wt% of formic acid, 20wt% of magnesium chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (65 °C , 120 minutes) to form an amber, homogeneous, and transparent liquid, as recorded in Table 1.

**[0038]** Example 1-6

**[0039]** Formic acid and magnesium chloride (MgCl<sub>2</sub>) were mixed and heated to form a mixing solution (80wt% of formic acid, 20wt% of magnesium chloride).  $\alpha$ -cellulose (Sigma Corporation, C8002) was added to the mixing solution (5wt% of  $\alpha$ -cellulose) for a dissolution reaction (65°C, 120 minutes) to form an amber, homogeneous, and transparent liquid, as recorded in Table 1.

Table 1

Examples	Salt (wt%)	Cellulose (wt%)	Dissolution temp. (°C)	Dissolution time (min)	Solution appearance
1-1	zinc chloride (40)	Avicel <sup>®</sup> cellulose (15)	50	20	yellow, homogeneous and transparent liquid
1-2	zinc chloride (40)	$\alpha$ -cellulose (15)	50	20	amber, homogeneous and transparent liquid
1-3	calcium chloride (25)	Avicel <sup>®</sup> cellulose (6)	65	90	yellow, homogeneous and transparent liquid
1-4	calcium chloride (25)	$\alpha$ -cellulose (6)	65	90	amber, homogeneous and transparent liquid
1-5	magnesium chloride (20)	Avicel <sup>®</sup> cellulose (5)	65	120	amber, homogeneous and transparent liquid
1-6	magnesium chloride (20)	$\alpha$ -cellulose (5)	65	120	amber, homogeneous and transparent liquid

**[0040]** Example 2-1

**[0041]** Formic acid and lithium chloride (LiCl) were mixed and heated to form a mixing solution (90wt% of formic acid, 10wt% of lithium chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 6 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

**[0042]** Example 2-2

**[0043]** Formic acid and lithium chloride (LiCl) were mixed and heated to form a mixing solution (95wt% of formic acid, 5wt% of lithium chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 12 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

**[0044]** Example 2-3

**[0045]** Formic acid and sodium chloride (NaCl) were mixed and heated to form a mixing solution (90wt% of formic acid, 10wt% of sodium chloride (saturated solution)). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 19 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

**[0046]** Example 2-4

**[0047]** Formic acid and lithium bromide (LiBr) were mixed and heated to form a mixing solution (90wt% of formic acid, 10wt% of lithium bromide). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution

(5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 0.5 hour). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

**[0048]** Example 2-5

**[0049]** Formic acid and sodium bromide (NaBr) were mixed and heated to form a mixing solution (82wt% of formic acid, 18wt% of sodium bromide). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 9 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

**[0050]** Example 2-6

**[0051]** Formic acid and calcium bromide (CaBr<sub>2</sub>) were mixed and heated to form a mixing solution (88wt% of formic acid, 12wt% of calcium bromide). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 6 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

**[0052]** Example 2-7

**[0053]** Formic acid and barium bromide (BaBr<sub>2</sub>) were mixed and heated to form a mixing solution (80wt% of formic acid, 20wt% of barium bromide). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 6 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

**[0054]** Example 2-8

**[0055]** Formic acid and magnesium chloride (MgCl<sub>2</sub>) were mixed and heated to form a mixing solution (80wt% of formic acid, 20wt% of magnesium chloride (saturated solution)). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (65 °C , 2 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

**[0056]** Example 2-9

**[0057]** Formic acid and magnesium chloride (MgCl<sub>2</sub>) were mixed and heated to form a mixing solution (90wt% of formic acid, 10wt% of magnesium chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 12 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

**[0058]** Example 2-10

**[0059]** Formic acid and calcium chloride (CaCl<sub>2</sub>) were mixed and heated to form a mixing solution (75wt% of formic acid, 25wt% of calcium chloride (saturated solution)). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (65°C, 1.5 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

**[0060]** Example 2-11

**[0061]** Formic acid and calcium chloride (CaCl<sub>2</sub>) were mixed and heated to form a mixing solution (82.5wt% of formic acid, 17.5wt% of calcium chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 2 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

**[0062]** Example 2-12

**[0063]** Formic acid and calcium chloride (CaCl<sub>2</sub>) were mixed and heated to form a mixing solution (88wt% of formic acid, 12wt% of calcium chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 6 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

**[0064]** Example 2-13

**[0065]** Formic acid and calcium chloride (CaCl<sub>2</sub>) were mixed and heated to form a mixing solution (90wt% of formic acid, 10wt% of calcium chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 12 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

**[0066]** Example 2-14

**[0067]** Formic acid and barium chloride (BaCl<sub>2</sub>) were mixed and heated to form a mixing solution (85wt% of formic acid, 15wt% of barium chloride (saturated solution)). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, >6 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

**[0068]** Example 2-15

**[0069]** Formic acid and zinc chloride (ZnCl<sub>2</sub>) were mixed and heated to form a mixing solution (60wt% of formic acid, 40wt% of zinc chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (50°C, 0.25 hour). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

**[0070]** Example 2-16

**[0071]** Formic acid and zinc chloride ( $\text{ZnCl}_2$ ) were mixed and heated to form a mixing solution (80wt% of formic acid, 20wt% of zinc chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (65°C, 0.25 hour). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

5 **[0072]** Example 2-17

**[0073]** Formic acid and zinc chloride ( $\text{ZnCl}_2$ ) were mixed and heated to form a mixing solution (95wt% of formic acid, 5wt% of zinc chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 6 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

10 **[0074]** Example 2-18

**[0075]** Formic acid and zinc chloride ( $\text{ZnCl}_2$ ) were mixed and heated to form a mixing solution (98wt% of formic acid, 2wt% of zinc chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, >6 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

15 **[0076]** Example 2-19

**[0077]** Formic acid and iron chloride ( $\text{FeCl}_3$ ) were mixed and heated to form a mixing solution (95wt% of formic acid, 5wt% of iron chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 1 hour). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

20 **[0078]** Example 2-20

**[0079]** Formic acid and iron chloride ( $\text{FeCl}_3$ ) were mixed and heated to form a mixing solution (98wt% of formic acid, 2wt% of iron chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 3 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

25 **[0080]** Example 2-21

**[0081]** Formic acid and iron chloride ( $\text{FeCl}_3$ ) were mixed and heated to form a mixing solution (99wt% of formic acid, 1wt% of iron chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 6 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

30 **[0082]** Example 2-22

**[0083]** Formic acid and ammonium chloride ( $\text{NH}_4\text{Cl}$ ) were mixed and heated to form a mixing solution (90wt% of formic acid, 10wt% of ammonium chloride (saturated solution)). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, >12 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

35 **[0084]** Example 2-23

**[0085]** Formic acid and aluminum chloride ( $\text{AlCl}_3$ ) were mixed and heated to form a mixing solution (98wt% of formic acid, 2wt% of aluminum chloride (saturated solution)). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70 °C, 6 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

40 **[0086]** Example 2-24

**[0087]** Formic acid and tin chloride ( $\text{SnCl}_2$ ) were mixed and heated to form a mixing solution (95wt% of formic acid, 5wt% of tin chloride (saturated solution)). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70 °C, 6 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

45 **[0088]** Example 2-25

**[0089]** Formic acid and calcium sulfate ( $\text{CaSO}_4$ ) were mixed and heated to form a mixing solution (80wt% of formic acid, 20wt% of calcium sulfate). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 6 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

50 **[0090]** Example 2-26

**[0091]** Formic acid and heteropoly acid ( $\text{H}_3\text{PW}_{12}\text{O}_{40}$ ) were mixed and heated to form a mixing solution (99wt% of formic acid, 1wt% of heteropoly acid). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (70°C, 6 hours). The dissolution of cellulose was observed using a polarizing microscope, as recorded in Table 2.

55

EP 2 712 936 A1

Table 2

Examples	Salt	wt%	Dissolution temp. (°C)	Dissolution time (hour)	Dissolution of cellulose	
5	2-1	lithium chloride	10	70	6	complete dissolution
	2-2		5	70	12	no dissolution
10	2-3	sodium chloride	10, saturated	70	19	no dissolution
	2-4	lithium bromide	10	70	0.5	complete dissolution
	2-5	sodium bromide	18	70	9	no dissolution
15	2-6	calcium	12	70	6	complete
		bromide				dissolution
	2-7	barium bromide	20	70	6	no dissolution
20	2-8	magnesium chloride	20, saturated	65	2	complete dissolution
	2-9		10	70	12	no dissolution
	2-10	calcium chloride	25, saturated	65	1.5	complete dissolution
25	2-11		17.5	70	2	complete dissolution
	2-12		12	70	6	complete dissolution
30	2-13		10	70	12	no dissolution
	2-14	barium chloride	15, saturated	70	>6	no dissolution
35	2-15	zinc chloride	40	50	0.25	complete dissolution
	2-16		20	65	0.25	complete dissolution
40	2-17		5	70	6	complete dissolution
	2-18		2	70	>6	no dissolution
	2-19	iron chloride	5	70	1	complete dissolution
45	2-20		2	70	3	complete dissolution
	2-21		1	70	6	complete dissolution
50	2-22	ammonium chloride	10, saturated	70	>12	no dissolution
	2-23	aluminum chloride	2, saturated	70	6	no dissolution
55	2-24	tin chloride	5, saturated	70	6	no dissolution

(continued)

Examples	Salt	wt%	Dissolution temp. (°C)	Dissolution time (hour)	Dissolution of cellulose
2-25	calcium sulfate	20	70	6	no dissolution
2-26	heteropoly acid (H <sub>3</sub> PW <sub>12</sub> O <sub>40</sub> )	1	70	6	complete dissolution

**[0092]** Example 3-1

**[0093]** Formic acid and magnesium chloride (MgCl<sub>2</sub>) were mixed by stirring and heated to 70°C under 1 atm to form a mixing solution (80wt% of formic acid, 20wt% of magnesium chloride). Avicel®cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel®cellulose) for a dissolution reaction (70°C, 2 hours). After the complete dissolution of the cellulose, water was added to the mixing solution (50wt% of water) and the mixing solution was heated to 100°C for a hydrolysis reaction (120 minutes). Next, saturated sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) aqueous solution was added to neutralize the mixing solution. Magnesium carbonate (MgCO<sub>3</sub>) precipitate was then removed from the mixing solution. Next, the total weight of the reducing sugar was measured using 3,5-dinitro-salicylic acid (DNS) method. The yield of the reducing sugar was then calculated. The reducing sugar comprised glucose, xylose, mannose, arabinose and oligosaccharides thereof. The yield of the reducing sugar is the ratio of the total weight of the reducing sugar and the weight of the cellulose. The result is shown in Table 3.

**[0094]** Example 3-2

**[0095]** Formic acid and magnesium chloride (MgCl<sub>2</sub>) were mixed by stirring and heated to 70°C under 1 atm to form a mixing solution (90wt% of formic acid, 10wt% of magnesium chloride). Avicel®cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel®cellulose) for a dissolution reaction (70°C, 6 hours). After the complete dissolution of the cellulose, water was added to the mixing solution (50wt% of water) and the mixing solution was heated to 100°C for a hydrolysis reaction (120 minutes). Next, saturated sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) aqueous solution was added to neutralize the mixing solution. Magnesium carbonate (MgCO<sub>3</sub>) precipitate was then removed from the mixing solution. Next, the total weight of the reducing sugar was measured using 3,5-dinitro-salicylic acid (DNS) method. The yield of the reducing sugar was then calculated. The reducing sugar comprised glucose, xylose, mannose, arabinose and oligosaccharides thereof. The yield of the reducing sugar is the ratio of the total weight of the reducing sugar and the weight of the cellulose. The result is shown in Table 3.

Table 3

Examples	Cellulose (wt%)	Mixing solution (magnesium chloride: formic acid) (wt%)	Dissolution temp. (°C)	Dissolution time (hour)	Hydrolysis temp. (°C)	Hydrolysis time (min)	Yield of reducing sugar (%)
3-1	5	20: 80	70	2	100	120	97.9
3-2	5	10: 90	70	6	100	120	75.3

**[0096]** Example 4-1

**[0097]** Formic acid and calcium chloride (CaCl<sub>2</sub>) were mixed by stirring and heated to 50°C under 1 atm to form a mixing solution (85wt% of formic acid, 15wt% of calcium chloride). Avicel®cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel®cellulose) for a dissolution reaction (50°C, 4 hours). After the complete dissolution of the cellulose, water was added to the mixing solution (50wt% of water) and the mixing solution was heated to 100°C for a hydrolysis reaction (60 minutes). Next, saturated sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) aqueous solution was added to neutralize the mixing solution. Calcium carbonate (CaCO<sub>3</sub>) precipitate was then removed from the mixing solution. Next, the total weight of the reducing sugar was measured using 3,5-dinitro-salicylic acid (DNS) method. The yield of the reducing sugar was then calculated. The reducing sugar comprised glucose, xylose, mannose, arabinose and oligosaccharides thereof. The yield of the reducing sugar is the ratio of the total weight of the reducing sugar and the weight of the cellulose. The result is shown in Table 4.

**[0098]** Example 4-2

**[0099]** Formic acid and calcium chloride (CaCl<sub>2</sub>) were mixed by stirring and heated to 70°C under 1 atm to form a mixing solution (88wt% of formic acid, 12wt% of calcium chloride). Avicel®cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel®cellulose) for a dissolution reaction (70°C, 4 hours). After the complete dissolution of the cellulose, water was added to the mixing solution (50wt% of water) and the mixing solution

## EP 2 712 936 A1

was heated to 100°C for a hydrolysis reaction (60 minutes). Next, saturated sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) aqueous solution was added to neutralize the mixing solution. Calcium carbonate (CaCO<sub>3</sub>) precipitate was then removed from the mixing solution. Next, the total weight of the reducing sugar was measured using 3,5-dinitro-salicylic acid (DNS) method. The yield of the reducing sugar was then calculated. The reducing sugar comprised glucose, xylose, mannose, arabinose and oligosaccharides thereof. The yield of the reducing sugar is the ratio of the total weight of the reducing sugar and the weight of the cellulose. The result is shown in Table 4.

**[0100]** Example 4-3

**[0101]** Formic acid and calcium chloride (CaCl<sub>2</sub>) were mixed by stirring and heated to 90°C under 1 atm to form a mixing solution (90wt% of formic acid, 10wt% of calcium chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (90°C, 4 hours). After the complete dissolution of the cellulose, water was added to the mixing solution (50wt% of water) and the mixing solution was heated to 100°C for a hydrolysis reaction (60 minutes). Next, saturated sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) aqueous solution was added to neutralize the mixing solution. Calcium carbonate (CaCO<sub>3</sub>) precipitate was then removed from the mixing solution. Next, the total weight of the reducing sugar was measured using 3,5-dinitro-salicylic acid (DNS) method. The yield of the reducing sugar was then calculated. The reducing sugar comprised glucose, xylose, mannose, arabinose and oligosaccharides thereof. The yield of the reducing sugar is the ratio of the total weight of the reducing sugar and the weight of the cellulose. The result is shown in Table 4.

Table 4

Examples	Cellulose (wt%)	Mixing solution (calcium chloride: formic acid) (wt%)	Dissolution temp. (°C)	Dissolution time (hour)	Hydrolysis temp. (°C)	Hydrolysis time (min)	Yield of reducing sugar (%)
4-1	5	15: 85	50	4	100	60	78.4
4-2	5	12: 88	70	4	100	60	70.6
4-3	5	10: 90	90	4	100	60	67.3

**[0102]** Example 5-1

**[0103]** Formic acid and zinc chloride (ZnCl<sub>2</sub>) were mixed by stirring and heated to 50°C under 1 atm to form a mixing solution (60wt% of formic acid, 40wt% of zinc chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (50°C). After the complete dissolution of the cellulose, water was added to the mixing solution (50wt% of water) and the mixing solution was heated to 100°C for a hydrolysis reaction (30 minutes). Next, saturated sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) aqueous solution was added to neutralize the mixing solution. Zinc carbonate (ZnCO<sub>3</sub>) precipitate was then removed from the mixing solution. Next, the total weight of the reducing sugar was measured using 3,5-dinitro-salicylic acid (DNS) method. The yield of the reducing sugar was then calculated. The reducing sugar comprised glucose, xylose, mannose, arabinose and oligosaccharides thereof. The yield of the reducing sugar is the ratio of the total weight of the reducing sugar and the weight of the cellulose. The result is shown in Table 5.

**[0104]** Example 5-2

**[0105]** Formic acid and zinc chloride (ZnCl<sub>2</sub>) were mixed by stirring and heated to 50°C under 1 atm to form a mixing solution (60wt% of formic acid, 40wt% of zinc chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (50°C). After the complete dissolution of the cellulose, water was added to the mixing solution (50wt% of water) and the mixing solution was heated to 100°C for a hydrolysis reaction (45 minutes). Next, saturated sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) aqueous solution was added to neutralize the mixing solution. Zinc carbonate (ZnCO<sub>3</sub>) precipitate was then removed from the mixing solution. Next, the total weight of the reducing sugar was measured using 3,5-dinitro-salicylic acid (DNS) method. The yield of the reducing sugar was then calculated. The reducing sugar comprised glucose, xylose, mannose, arabinose and oligosaccharides thereof. The yield of the reducing sugar is the ratio of the total weight of the reducing sugar and the weight of the cellulose. The result is shown in Table 5.

Table 5

Examples	Cellulose (wt%)	Adding amount of water (wt%)	Hydrolysis time (min)	Yield of reducing sugar (%)
5-1	5	50	30	65
5-2	5	50	45	89

**[0106]** Example 6

**[0107]** Formic acid and zinc chloride ( $\text{ZnCl}_2$ ) were mixed by stirring and heated to 55 °C under 1 atm to form a mixing solution (60wt% of formic acid, 40wt% of zinc chloride). Dried bagasse (comprising 43.58wt% of glucan, 24.02wt% of xylan, 12.45wt% of acid-soluble lignin, 18.12wt% of acid-insoluble lignin and 1.71wt% of ash) was added to the mixing solution (5wt% of bagasse) for a dissolution reaction (55°C). After the dissolution of the bagasse, water was added to the mixing solution (50wt% of water) and the mixing solution was heated to 100°C for a hydrolysis reaction (120 minutes). Next, saturated sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) aqueous solution was added to neutralize the mixing solution. Zinc carbonate ( $\text{ZnCO}_3$ ) precipitate was then removed from the mixing solution. Next, the yields of glucose and xylose were analyzed using high performance liquid chromatography (HPLC) and the total weight of the reducing sugar was measured using 3,5-dinitro-salicylic acid (DNS) method. The yield of the reducing sugar was then calculated. The reducing sugar comprised glucose, xylose, mannose, arabinose and oligosaccharides thereof. The yield of the glucose is the ratio of the moles of the produced glucose and the moles of the glucose monomers contained in the cellulose in the bagasse. The yield of the xylose is the ratio of the moles of the produced xylose and the moles of the xylose monomers contained in the hemicellulose in the bagasse. The yield of the reducing sugar is the ratio of the total weight of the reducing sugar and the total weight of the cellulose and hemicellulose in the bagasse. The result is shown in Table 6. After the hydrolysis reaction, a hydrolyzed solution comprising 25.3wt% of zinc chloride, 33.2wt% of water, 38.2wt% of formic acid, 2.3wt% of reducing sugar (comprising 43.2wt% of glucose and 30.4wt% of xylose), 0.4wt% of acid-soluble lignin and 0.6wt% of acid-insoluble lignin was formed.

Table 6

Examples	Bagasse (wt%)	Amount of water added (wt%)	Hydrolysis time (min)	Yield of glucose (%)	Yield of xylose (%)	Yield of reducing sugar (%)
6-1	5	50	30	36.3	88.5	93.3
6-2	5	50	60	53.3	94.2	97.9
6-3	5	50	120	70.4	89.9	105.2

**[0108]** Example 7

**[0109]** Formic acid and magnesium chloride ( $\text{MgCl}_2$ ) were mixed by stirring and heated to 50°C under 1 atm to form a mixing solution (80wt% of formic acid, 20wt% of magnesium chloride). Avicel®cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel®cellulose) for a dissolution reaction (50°C, 2.5 hours). After the dissolution of the cellulose, water was added to the mixing solution (50wt% of water) and the mixing solution was heated to 100°C for a hydrolysis reaction (90 minutes). Next, saturated sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) aqueous solution was added to neutralize the mixing solution. Magnesium carbonate ( $\text{MgCO}_3$ ) precipitate was then removed from the mixing solution. Next, the total weight of the reducing sugar was measured using 3,5-dinitro-salicylic acid (DNS) method. The yield of the reducing sugar was then calculated. The reducing sugar comprised glucose, xylose, mannose, arabinose and oligosaccharides thereof. The yield of the reducing sugar is the ratio of the total weight of the reducing sugar and the weight of the cellulose. The result is shown in Table 7.

Table 7

Examples	Cellulose (wt%)	Mixing solution (magnesium chloride: formic acid) (wt%)	Dissolution temp. (°C)	Dissolution time (hour)	Hydrolysis temp. (°C)	Hydrolysis time (min)	Yield of reducing sugar (%)
7	5	20: 80	50	2.5	100	0th	46
					100	90th	89

**[0110]** Example 8

**[0111]** Formic acid and zinc chloride ( $\text{ZnCl}_2$ ) were mixed by stirring and heated to 55 °C under 1 atm to form a mixing solution (60wt% of formic acid, 40wt% of zinc chloride). Dried corn stalks (comprising 44.5wt% of glucan, 12.4wt% of xylan, 4.6wt% of acid-soluble lignin, 24.4wt% of acid-insoluble lignin, 2.7wt% of water and 3.8wt% of ash) was added to the mixing solution (5wt% of corn stalks) for a dissolution reaction (55°C). After the dissolution of the corn stalks, water was added to the mixing solution (50wt% of water) and the mixing solution was heated to 100°C for a hydrolysis reaction (90 minutes). Next, saturated sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) aqueous solution was added to neutralize the mixing solution. Zinc carbonate ( $\text{ZnCO}_3$ ) precipitate was then removed from the mixing solution. Next, the yields of glucose and

xylose were analyzed using high performance liquid chromatography (HPLC) and the total weight of the reducing sugar was measured using 3,5-dinitro-salicylic acid (DNS) method. The yield of the glucose is the ratio of the moles of the produced glucose and the moles of the glucose monomers contained in the cellulose in the corn stalks. The yield of the reducing sugar was then calculated. The reducing sugar comprised glucose, xylose, mannose, arabinose and oligosaccharides thereof. The yield of the reducing sugar is the ratio of the total weight of the reducing sugar and the total weight of the cellulose and hemicellulose in the corn stalks. The result is shown in Table 8.

Table 8

Examples	Corn stalks (wt%)	Amount of water added (wt%)	Hydrolysis time (min)	Yield of glucose (%)	Yield of reducing sugar (%)
8	5	50	90	85	96

**[0112]** Example 9-1

**[0113]** 37wt% of HCl, zinc chloride ( $ZnCl_2$ ) and formic acid were mixed by stirring and heated to 55°C under 1 atm to form a mixing solution (1wt% of HCl, 5wt% of zinc chloride, 94wt% of formic acid). Dried bagasse (comprising 40.7wt% of glucan, 20.5wt% of xylan, 2.9wt% of Arab polysaccharides, 27.4wt% of lignin, 3.3wt% of ash and 5.2wt% of other ingredients) was added to the mixing solution (10wt% of bagasse) for a dissolution reaction (65°C). After the dissolution of the bagasse, water was added to the mixing solution (50wt% of water) and the mixing solution was heated to 100°C for a hydrolysis reaction. Next, saturated sodium carbonate ( $Na_2CO_3$ ) aqueous solution was added to neutralize the mixing solution. Zinc carbonate ( $ZnCO_3$ ) precipitate was then removed from the mixing solution. Next, the yields of glucose and xylose were analyzed using high performance liquid chromatography (HPLC) and the total weight of the reducing sugar was measured using 3,5-dinitro-salicylic acid (DNS) method. The yield of the reducing sugar was then calculated. The reducing sugar comprised glucose, xylose, mannose, arabinose and oligosaccharides thereof. The yield of the glucose is the ratio of the moles of the produced glucose and the moles of the glucose monomers contained in the cellulose in the bagasse. The yield of the xylose is the ratio of the moles of the produced xylose and the moles of the xylose monomers contained in the hemicellulose in the bagasse. The yield of the reducing sugar is the ratio of the total weight of the reducing sugar and the total weight of the cellulose and hemicellulose in the bagasse. The result is shown in Table 9.

**[0114]** Example 9-2

**[0115]** 37wt% of HCl, iron chloride ( $FeCl_3$ ) and formic acid were mixed by stirring and heated to 55°C under 1 atm to form a mixing solution (1wt% of HCl, 2wt% of iron chloride, 97wt% of formic acid). Dried bagasse (comprising 40.7wt% of glucan, 20.5wt% of xylan, 2.9wt% of Arab polysaccharides, 27.4wt% of lignin, 3.3wt% of ash and 5.2wt% of other ingredients) was added to the mixing solution (10wt% of bagasse) for a dissolution reaction (65°C). After the dissolution of the bagasse, water was added to the mixing solution (50wt% of water) and the mixing solution was heated to 100°C for a hydrolysis reaction. Next, saturated sodium carbonate ( $Na_2CO_3$ ) aqueous solution was added to neutralize the mixing solution. Iron carbonate ( $Fe_2(CO_3)_3$ ) precipitate was then removed from the mixing solution. Next, the yields of glucose and xylose were analyzed using high performance liquid chromatography (HPLC) and the total weight of the reducing sugar was measured using 3,5-dinitro-salicylic acid (DNS) method. The yield of the reducing sugar was then calculated. The reducing sugar comprised glucose, xylose, mannose, arabinose and oligosaccharides thereof. The yield of the glucose is the ratio of the moles of the produced glucose and the moles of the glucose monomers contained in the cellulose in the bagasse. The yield of the xylose is the ratio of the moles of the produced xylose and the moles of the xylose monomers contained in the hemicellulose in the bagasse. The yield of the reducing sugar is the ratio of the total weight of the reducing sugar and the total weight of the cellulose and hemicellulose in the bagasse. The result is shown in Table 9.

**[0116]** Example 9-3

**[0117]** 98wt% of  $H_2SO_4$ , iron chloride ( $FeCl_3$ ) and formic acid were mixed by stirring and heated to 55°C under 1 atm to form a mixing solution (1wt% of  $H_2SO_4$ , 2wt% of iron chloride, 97wt% of formic acid). Dried bagasse (comprising 40.7wt% of glucan, 20.5wt% of xylan, 2.9wt% of Arab polysaccharides, 27.4wt% of lignin, 3.3wt% of ash and 5.2wt% of other ingredients) was added to the mixing solution (10wt% of bagasse) for a dissolution reaction (65°C). After the dissolution of the bagasse, water was added to the mixing solution (50wt% of water) and the mixing solution was heated to 100°C for a hydrolysis reaction. Next, saturated sodium carbonate ( $Na_2CO_3$ ) aqueous solution was added to neutralize the mixing solution. Iron carbonate ( $Fe_2(CO_3)_3$ ) precipitate was then removed from the mixing solution. Next, the yields of glucose and xylose were analyzed using high performance liquid chromatography (HPLC) and the total weight of the reducing sugar was measured using 3,5-dinitro-salicylic acid (DNS) method. The yield of the reducing sugar was then calculated. The reducing sugar comprised glucose, xylose, mannose, arabinose and oligosaccharides thereof. The yield of the glucose is the ratio of the moles of the produced glucose and the moles of the glucose monomers contained in

the cellulose in the bagasse. The yield of the xylose is the ratio of the moles of the produced xylose and the moles of the xylose monomers contained in the hemicellulose in the bagasse. The yield of the reducing sugar is the ratio of the total weight of the reducing sugar and the total weight of the cellulose and hemicellulose in the bagasse. The result is shown in Table 9.

Table 9

Examples	Hydrolysis time (min)	Yield of glucose (%)	Yield of xylose (%)	Yield of reducing sugar (%)
9-1	90	67.5	82.7	94.5
9-2	90	57.5	78.3	76.6
9-3	90	50.5	85.3	75.1

**[0118]** Example 10-1

**[0119]** Formic acid, acetic acid and zinc chloride ( $ZnCl_2$ ) were mixed and heated to form a mixing solution (54wt% of formic acid, 6wt% of acetic acid and 40wt% of zinc chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (60°C, 60 minutes), forming an amber transparent liquid with a uniform phase. The dissolution of cellulose was observed using a polarizing microscope. The cellulose was completely dissolved.

**[0120]** Example 10-2

**[0121]** Formic acid, acetic acid and calcium chloride ( $CaCl_2$ ) were mixed and heated to form a mixing solution (72wt% of formic acid, 8wt% of acetic acid and 20wt% of calcium chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (60°C, 180 minutes), forming an amber transparent liquid with a uniform phase. The dissolution of cellulose was observed using a polarizing microscope. The cellulose was completely dissolved.

**[0122]** Example 10-3

**[0123]** Formic acid, acetic acid and zinc chloride ( $ZnCl_2$ ) were mixed and heated to form a mixing solution (50wt% of formic acid, 10wt% of acetic acid and 40wt% of zinc chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (65°C, 60 minutes), forming an amber transparent liquid with a uniform phase. The dissolution of cellulose was observed using a polarizing microscope. The cellulose was completely dissolved.

**[0124]** Example 10-4

**[0125]** Formic acid, acetic acid and zinc chloride ( $ZnCl_2$ ) were mixed and heated to form a mixing solution (40wt% of formic acid, 20wt% of acetic acid and 40wt% of zinc chloride). Avicel<sup>®</sup>cellulose (Sigma Corporation, Avicel-pH-105-27NI) was added to the mixing solution (5wt% of Avicel<sup>®</sup>cellulose) for a dissolution reaction (65°C, 60 minutes), forming an amber transparent liquid with a uniform phase. The dissolution of cellulose was observed using a polarizing microscope. The cellulose was completely dissolved.

**[0126]** It will be apparent to those skilled in the art that various modifications and variations can be made to the disclosed embodiments. It is intended that the specification and examples be considered as exemplary only, with the true scope of the disclosure being indicated by the following claims and their equivalents.

## Claims

1. A sugar product, comprising:

a sugar mixture comprising glucose, xylose, mannose, arabinose and oligosaccharides thereof with a weight ratio of 2-15wt%;  
 an acid compound with a weight ratio of 48-97wt%; and  
 a salt compound with a weight ratio of 1-50wt%.

2. The sugar product as claimed in claim 1, wherein the acid compound comprises organic acid compounds or inorganic acid compounds.

3. The sugar product as claimed in claim 1 or 2, wherein acid compound comprises formic acid, acetic acid or a mixture thereof.

## EP 2 712 936 A1

4. The sugar product as claimed in one of the preceding claims, wherein the salt compound comprises lithium chloride, magnesium chloride, calcium chloride, zinc chloride, iron chloride, lithium bromide, magnesium bromide, calcium bromide, zinc bromide or iron bromide.
5. A method for fabricating a sugar product, comprising:
- mixing an acid compound and lithium chloride, magnesium chloride, calcium chloride, zinc chloride, iron chloride, lithium bromide, magnesium bromide, calcium bromide, zinc bromide, iron bromide or heteropoly acid to form a mixing solution;
  - adding a cellulosic biomass to the mixing solution for a dissolution reaction;
  - and
  - adding water to the mixing solution for a hydrolysis reaction to obtain a sugar product.
6. The method for fabricating a sugar product as claimed in claim 5, wherein the acid compound comprises formic acid, acetic acid or a mixture thereof.
7. The method for fabricating a sugar product as claimed in claims 5 or 6, wherein the formic acid or acetic acid has a weight ratio of 50-97wt% in the mixing solution.
8. The method for fabricating a sugar product as claimed in one or more of claims 5 to 7, wherein the lithium chloride or lithium bromide has a weight ratio of 5-20wt% in the mixing solution.
9. The method for fabricating a sugar product as claimed in one or more of claims 5 to 8, wherein the magnesium chloride or magnesium bromide has a weight ratio of 10-30wt% in the mixing solution.
10. The method for fabricating a sugar product as claimed in one or more of claims 5 to 9, wherein the calcium chloride or calcium bromide has a weight ratio of 12-40wt% in the mixing solution.
11. The method for fabricating a sugar product as claimed in one or more of claims 5 to 10, wherein the zinc chloride or zinc bromide has a weight ratio of 5-45wt% in the mixing solution.
12. The method for fabricating a sugar product as claimed in one or more of claims 5 to 11, wherein the iron chloride or iron bromide has a weight ratio of 1-50wt% in the mixing solution.
13. The method for fabricating a sugar product as claimed in one or more of claims 5 to 12, wherein the heteropoly acid comprises  $H_3PW_{12}O_{40}$ ,  $H_4SiW_{12}O_{40}$ ,  $H_3PMo_{12}O_{40}$  or  $H_4SiMo_{12}O_{40}$ .
14. The method for fabricating a sugar product as claimed in one or more of claims 5 to 13, wherein the heteropoly acid has a weight ratio of 1-5wt% in the mixing solution.
15. The method for fabricating a sugar product as claimed in one or more of claims 5 to 14, wherein the cellulosic biomass comprises cellulose, hemicellulose or lignin.
16. The method for fabricating a sugar product as claimed in one or more of claims 5 to 15, wherein the cellulosic biomass is derived from wood, grass, leaves, algae, waste paper, corn stalks, corn cobs, rice straw, rice husk, wheat straw, bagasse, bamboo or crop stems.
17. The method for fabricating a sugar product as claimed in one or more of claims 5 to 16, wherein the dissolution reaction has a reaction temperature of 40-90°C.
18. The method for fabricating a sugar product as claimed in one or more of claims 5 to 17, wherein the dissolution reaction has a reaction time of 20-360 minutes.
19. The method for fabricating a sugar product as claimed in one or more of claims 5 to 18, wherein the amount of water added is larger than the total molar equivalent of monosaccharides hydrolyzed from the cellulosic biomass.
20. The method for fabricating a sugar product as claimed in one or more of claims 5 to 19, wherein the hydrolysis

reaction has a reaction temperature of 50-150°C.

- 5
21. The method for fabricating a sugar product as claimed in one or more of claims 5 to 20, wherein the hydrolysis reaction has a reaction time of 30-180 minutes.
- 10
22. The method for fabricating a sugar product as claimed in one or more of claims 5 to 21, wherein the sugar product comprises a sugar mixture, an acid compound and a salt compound.
- 15
23. The method for fabricating a sugar product as claimed in claim 22, wherein the sugar mixture comprises glucose, xylose, mannose, arabinose and oligosaccharides thereof.
- 20
24. The method for fabricating a sugar product as claimed in claim 22 or 23, wherein the sugar mixture has a weight ratio of 2-15wt% in the sugar product.
- 25
25. The method for fabricating a sugar product as claimed in one or more of claims 22 to 24, wherein the salt compound comprises lithium chloride, magnesium chloride, calcium chloride, zinc chloride, iron chloride, lithium bromide, magnesium bromide, calcium bromide, zinc bromide or iron bromide.
- 30
26. The method for fabricating a sugar product as claimed in one or more of claims 22 to 25, wherein the salt compound has a weight ratio of 1-50wt% in the sugar product.
- 35
27. The method for fabricating a sugar product as claimed in one or more of claims 5 to 26, further comprising adding inorganic acid to the mixing solution.
- 40
28. The method for fabricating a sugar product as claimed in claim 27, wherein the inorganic acid comprises sulfuric acid or hydrochloric acid.
- 45
29. The method for fabricating a sugar product as claimed in claim 27 or 28, wherein the inorganic acid has a weight ratio of 1-2wt% in the mixing solution.
- 50
30. The method for fabricating a sugar product as claimed in one or more of claims 27 to 29, wherein the magnesium chloride, the magnesium bromide, the calcium chloride or the calcium bromide has a weight ratio of 1-10wt% in the mixing solution.
- 55
31. The method for fabricating a sugar product as claimed in one or more of claims 27 to 30, wherein the lithium chloride, lithium bromide, the zinc chloride, the zinc bromide, the iron chloride or iron bromide has a weight ratio of 1-5wt% in the mixing solution.



EUROPEAN SEARCH REPORT

Application Number  
EP 13 18 6469

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	WO 2006/007691 A1 (IOGEN ENERGY CORP [CA]; FOODY BRIAN [CA]; TOLAN JEFFREY S [CA]) 26 January 2006 (2006-01-26) * claim 1 *	1-4	INV. C13K1/02 C12P19/02
A	WO 2010/104371 A1 (UNIV MALAYA [MY]; ZAINUDIN BIN ARIFIN [MY]; TEOH TEOW CHONG [MY]) 16 September 2010 (2010-09-16) * the whole document *	1-31	
A	US 2008/102502 A1 (FOODY BRIAN [CA] ET AL) 1 May 2008 (2008-05-01) * the whole document *	1-31	
A	US 4 452 640 A (CHEN LI FU [US] ET AL) 5 June 1984 (1984-06-05) * the whole document *	1-31	
			TECHNICAL FIELDS SEARCHED (IPC)
			C13K C12P
The present search report has been drawn up for all claims			
Place of search The Hague		Date of completion of the search 27 January 2014	Examiner Picout, David
CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ..... & : member of the same patent family, corresponding document	
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			

EPO FORM 1503 03/82 (P04C01)

ANNEX TO THE EUROPEAN SEARCH REPORT  
ON EUROPEAN PATENT APPLICATION NO.

EP 13 18 6469

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on  
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

27-01-2014

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 2006007691 A1	26-01-2006	AU 2005263133 A1	26-01-2006
		BR PI0513398 A	06-05-2008
		CA 2572502 A1	26-01-2006
		CN 101023179 A	22-08-2007
		EP 1778853 A1	02-05-2007
		JP 2008506370 A	06-03-2008
		US 2009023187 A1	22-01-2009
		WO 2006007691 A1	26-01-2006
WO 2010104371 A1	16-09-2010	CN 102348812 A	08-02-2012
		US 2011281317 A1	17-11-2011
		WO 2010104371 A1	16-09-2010
US 2008102502 A1	01-05-2008	NONE	
US 4452640 A	05-06-1984	NONE	

EPO FORM P0459

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

**REFERENCES CITED IN THE DESCRIPTION**

*This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.*

**Patent documents cited in the description**

- CN 2013104350048 [0001]
- US 97307213 A [0001]
- US 61707576 B [0001]