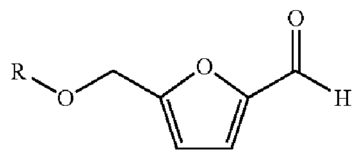
FUR 223b PC

Process for the preparation of a furfural derivative comprising neutralizing an acid reaction mixture

The present invention relates to a process for the preparation of a furfural derivative comprising neutralizing an acid reaction mixture.

The furfural derivative can be described as having the chemical formula (1):



(1),

wherein R represents hydrogen, an alkyl or an acyl group.

Furfural derivatives of chemical formula (1), including 5-hydroxymethylfurfural (HMF), 5-alkoxymethylfurfural (AlkMF) and 5-acyloxymethylfurfural (AcMF) are interesting chemicals. The furfural derivatives find application as precursor for e.g. furan dicarboxylic acid, an important monomer for polyesters, polyamides and polyurethanes. Alternatively, they can be used as fuel components. HMF has further antibacterial and anticorrosive properties. HMF, AlkMF and AcMF can be derived from sustainable sources. The furfural derivatives may be derived from a variety of carbohydrates, in particular from hexoses, such as fructose and glucose. Raw materials such as starch, cellulose, sucrose or inulin can be used as starting products for the manufacture of hexoses.

Since HMF, AlkMF and AcMF can be obtained from sustainable sources the interest in their production is growing. A process for their production is described in US 7317116. This US patent specification describes a process for the preparation of HMF wherein a fructose source, such as high fructose corn syrup, and an organic solvent are heated in the presence of an acid catalyst to achieve the acid-catalyzed dehydration reaction of fructose. The resulting product may then be neutralized to a pH of 7 to 9, e.g. by the gradual addition of sodium hydroxide. In examples the neutralization is carried out to pH values of at least 7.5. Subsequently, the thus neutralized product was subjected to distillation to remove the solvent.

In a different embodiment US 7317116 describes the preparation of an R’-oxymethyl furfural ether wherein R’ may represent alkyl, by combining a fructose source and an R’-OH solvent and by contacting the combination thus obtained with a solid acid catalyst bed in a chromatographic column. By heating the admixture in the chromatographic column fructose is dehydrated to form R’-oxymethylfurfural ether.

In US 8877950 a process is described wherein ethers of 5-hydroxymethylfurfural are manufactured by reacting a fructose-containing starting material with methanol, in the presence of a catalytic or sub-stoichiometric amount of a homogenous acid catalyst, wherein water is present as solvent in addition to the alcohol, wherein the ratio of alcohol/water-solvent is from 50:1 to 10:1, wherein the method is performed in a continuous flow process at a temperature of 175 to 225° C and at a residence time in the flow process from 1 minute to 10 minutes. Very suitably the homogenous acid catalyst is sulfuric acid.

Further, furfural derivatives of the chemical formula (1) wherein R represents an acyl group, have been described in US 8242293. Such derivatives can be prepared by reacting a fructose and/or glucose-containing starting material with a carboxylic acid in the presence of an acid catalyst in a continuous mode, wherein water is present in a small proportion. The carboxylic acid may be selected from e.g. C_1-C_6 carboxylic acids. The reaction yields esters of HMF, wherein the acyl moiety of the carboxylic acid is bound to the oxygen atom of the oxymethyl group at the 5-position, herein referred to as AcMF. As shown in an example, sulfuric acid may be used as the acid catalyst.

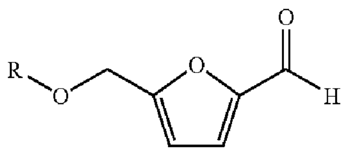
The product obtained in any of these processes includes by-products, in addition to HMF and/or AlkMF and/or AcMF. A competing side reaction is the polymerization of HMF, AlkMF or AcMF and/or the hexose to form humin polymers. Humin polymers or humins are the colored bodies which are believed to be polymers containing moieties from hydroxymethylfurfural, furfural, carbohydrate and levulinic acid. Humins are obtained as insoluble solid material. As shown in the examples of US 7317116, other by-products may include levulinic acid, levulinate esters and formic acid. Such by-products add to the acidity of the reaction mixture, which may already be acidic in view of the presence of the acid catalyst. It was found that under such acidic conditions degradation reactions occur which affect the yield of the desired products HMF, AlkMF and/or AcMF. Therefore, it has been proposed to neutralize the reaction mixture.

US 7317116 teaches in particular a process for the preparation of HMF by i) combining a fructose source, an organic solvent, and an acid catalyst to provide a reaction mixture; ii) heating said reaction mixture to a temperature and for a time sufficient to promote a dehydration reaction of fructose in said fructose source to form a first product mixture; iii) neutralizing the pH of the first product mixture to a pH of about 7 to 9; iv) distilling the first product mixture after neutralizing the pH to remove said organic solvent remaining in the first product mixture; and v) purifying said product mixture to provide a second product mixture comprising greater than 60% by weight of HMF. In one embodiment, the product is adjusted to a neutral pH after removing the ion-exchange resin from said product mixture, and before being subjected to a distillation to remove the organic solvent.

According to US 7317116 neutralization is desirable as it allows for product recovery by distillation without heat-catalyzed degradation or polymerization, leading to tarry degradation products and resinous solids, i.e. humins. The neutralization step also stated to allow for product recovery with a flowing agent without such degradation or polymerization.

It has now been found that under acidic conditions in the reaction mixture some reaction products, viz. HMF and/or AlkMF and/or AcMF, may undergo acetal formation. It has been found that in particular when the reaction mixture comprises an alcohol, which is especially the case when the reaction of the fructose or glucose source has been conducted with an alcohol, the furfural product obtained, i.e. an AlkMF, may react with the alcohol to form hemiacetals or acetals. Such reaction may take place under the influence of the presence of an acid catalyst. It has now surprisingly been found that when a part of the reaction mixture is removed before neutralization the eventual products no longer contain acetals.

Accordingly, the present invention provides a process for the preparation of a furfural derivative having the chemical formula (1)



(1)

wherein R represents hydrogen, an alkyl group or an acyl group, which process comprises

reacting a fructose- and/or glucose-containing starting material with a liquid hydroxyl group-containing compound of formula R-OH in the presence of an acid catalyst at a reaction temperature in the range of 150 to 300 °C to produce a primary acid reaction mixture comprising the furfural derivative of chemical formula (1);

separating at least part of the liquid hydroxyl group-containing compound from the primary acid reaction mixture to yield a secondary acid reaction mixture;

neutralizing the secondary acid reaction mixture to a pH-value of at least 3 to obtain a neutralized reaction mixture; and

purifying the neutralized reaction mixture to recover the furfural derivative of chemical formula (1).

It was found that the furfural derivative of chemical formula (1) that is recovered in the process according to the invention does not contain hemiacetal or acetal moieties. In US 7317116 it was not realized that the reversed order of first distillation of the excess solvent and subsequent neutralization had such advantageous results. The neutralizing of the pH of the secondary acid reaction mixture is suitably accomplished by the addition of a neutralizing agent. Preferably, the complete product of the separation of at least part of the liquid hydroxyl group-containing compound from the primary reaction mixture is recovered as the secondary acid reaction mixture. However, it will be understood by the skilled person that the secondary acid reaction mixture to be neutralized may be the major part of this separation product when a small part of the separation product is discharged and used for different purposes.

When R-OH represents water it may be beneficial to remove part of the water from the primary acid reaction mixture before neutralization. It may lead to the formation of a more concentrated solution of the furfural derivative and other products which may facilitate their subsequent recovery. Also when the group R in the compound R-OH represents an acyl group it may be feasible to separate a part of the compounds before neutralization. In that case, some of the R-OH compounds, which are acids, can be removed to facilitate the subsequent neutralization and reduce the required amount of neutralizing agent. However, the liquid hydroxyl group-containing compound is preferably a compound of formula R-OH, wherein R represents an alkyl group. When the primary acid reaction mixture comprises an alcohol, the formation of hemiacetal and acetal groups is most prominent. That implies that the advantage of the present invention is most apparent when the reaction of the fructose and/or glucose-containing starting material is carried out with a compound R-OH wherein R represents an alkyl group. Without wishing to be bound to any theory it is believed that the acetal formation from carbonyl groups with an alcohol is catalyzed by strongly acidic conditions, e.g. such that prevail in the primary acid reaction mixture. When hemiacetal and acetal compounds are present in a less acidic environment in the presence of water, it is believed that the acetal moieties decompose to again form the carbonyl groups. Therefore, it is particularly advantageous to remove alcohol before neutralization.

In WO2013/043131 it is described that secondary and tertiary alcohols form solely HMF and do not form AlkMF. Table 1 in this application suggests that also these alcohols form AlkMF (cf. entry 9*). This is confirmed by an earlier application, viz. WO2009/030506, wherein t-butyl alcohol or isopropanol are reacted with fructose at a temperature of at least 150 ^{o}C, whereby mixtures of HMF and the respective AlkMF are formed. Hence, the present invention is clearly of benefit to processes wherein R is a primary alcohol, but also a secondary or tertiary alcohol. WO 2013/043131 also teaches that certain 5-alkoxymethyl furfural compounds may form acetals. However, it does not provide a solution to the prevention of the formation thereof.

Suitably, at least part of any water, either formed during the conversion of the fructose and/or glucose-containing starting material or already supplied together with the alcohol, in the primary acid reaction mixture is left in the remaining part of the secondary acid reaction mixture. With water any acetal formed can decompose to again form the carbonyl group. Therefore the removal of the liquid hydroxyl group-containing compound, such as an alcohol, is suitably carried out such that most, such as at least 50 %wt, suitably at least 70 %wt, based on the amount of liquid hydroxyl group-containing compound in the primary acid reaction mixture, more preferably substantially all, of the liquid hydroxyl group-containing compound is removed from the primary acid reaction mixture before the remaining secondary acid reaction mixture is subjected to neutralization. A suitable method of removing the liquid hydroxyl group-containing compound is evaporation, flashing or distillation. The liquid hydroxyl group-containing compound that is recovered in this way may suitably be recycled to the reaction thereof with the fructose and/or glucose-containing starting material.

As apparent from the descriptions of US 8877950 and US 8242293 the furfural derivatives of chemical formula (1) are suitably manufactured in the presence of water. Therefore, the liquid hydroxyl-containing compound used in the present process preferably comprises water. Further, since the use of R-OH, wherein R represents hydrogen or alkyl, is preferred, suitably an alkanol, more preferably an alkanol with 1 to 6 carbon atoms is used, which alkanol optionally contains a proportion of water. The alkanol is preferably a primary alcohol. Such a proportion of water may suitably range from 0.5 to 20 %wt, based on the weight of the alkanol and water in the primary acid reaction mixture. Even more preferably, the liquid hydroxyl-containing compound comprises ethanol or methanol, most preferably methanol. As the presence of water is advantageous, the liquid hydroxyl-containing compound is suitably water, methanol or a mixture thereof.

The furfural derivative of chemical formula (1) is prepared from a reaction of a fructose- and/or glucose-containing starting material with R-OH in the presence of an acid catalyst. Suitable acid catalysts have been described in US 7317116, US 8242293 and US 8877950. Such suitable catalysts include inorganic acids, such as sulfuric acid, phosphoric acid, hydrochloric acid and nitric acid, and organic acids, such as oxalic acid, levulinic acid, trifluoroacetic acid, methane sulfonic acid or p-toluene sulfonic acid. Immobilized acid catalysts in the form of e.g. sulfonic acid on resins may also be used. Other acid ion exchange resins are feasible as well as acid zeolites. Lewis acids, such as boron trifuoride or etherate complexes thereof, are further suitable catalysts. Also metals, such as Zn, Al, Cr, Ti, Th, Zr, and V can be used as catalyst in the form of ions, salts, or complexes. It appears that a wide range of acid components can be used as catalysts. The present process is very suitably carried out with an acid catalyst being a Brønsted acid selected from the group consisting of mineral inorganic acids, organic acids and mixtures thereof. Suitable mineral acids are sulfuric acid, nitric acid, hydrochloric acid and phosphoric acid, wherein sulfuric acid is particularly preferred. The organic acids are suitably selected from strong acids. Examples thereof include trifluoroacetic acid, methane sulfonic acid and p-toluene sulfonic acid.

The use of the mineral acids and strong organic acids suitably results in that the primary and also the secondary acid reaction mixture typically have a pH value of smaller than 3, preferably smaller than 2. However, it is also possible to arrive at low pH values when acid heterogeneous catalysts are used, such as acid ion exchange resins or acid zeolites. As indicated above, products of the conversion of fructose and/or glucose-containing starting materials may also include various organic acids, such as levulinic acid and formic acid. The process of the present invention is therefore also suitable for embodiments wherein the reaction of the fructose and/or glucose-containing starting material is achieved with a heterogeneous, i.e. solid, catalyst.

The neutralization may be conducted to a pH value in the range of 7 and above, e.g. from 7 to 13, or to 7 to 9, as disclosed in US 7317116. Suitably the neutralization is conducted to a pH value in the range of 3 to 13, more suitably in the range of 3 to 9. However, it has been found that furfural derivatives of chemical formula (1) may undergo degradation reactions in the presence of alkaline agents. It is therefore not preferred to neutralize the secondary acid reaction mixture to a pH of above 9, or even 7 and above. In addition to the potential degradation of the furfural derivatives under the influence of alkaline media, there is a further disadvantage in that large amounts of alkaline neutralizing agents may be required to arrive at such high pH-values. It represents therefore an advantageous surprise that neutralization does not have to be done to a pH-value of 7 and above. It has surprisingly been found that already at a pH-value of at least 3 the degradation can be significantly avoided.

Advantageously, the neutralization is conducted to as low a pH as feasible. That implies that the pH is increased to such a low value that only a little amount of neutralizing agent is to be added to the secondary acid reaction mixture and at the same time that the degradation of valuable products such as the furfural derivative of chemical formula (1), but also degradation of compounds such as levulinic acid and esters thereof, does not take place. It has been found that very good results are obtained when the pH is brought into the range of 3 to 7, suitably 3 to 6.5, more suitably 3 to 6, preferably from 3 to 5, more preferably from 3 to 4.5.

Such neutralization can suitably be achieved by adding an aqueous solution of the neutralizing agent to the secondary acid reaction mixture. At the same time the skilled person will realize that it is advantageous when the neutralizing agent is added in a form as concentrated as feasible. When the neutralizing agent is added to the secondary acid reaction mixture in the form of a concentrated solution the secondary acid reaction solution is suitably agitated to accomplish a distribution of the neutralizing agent as quickly and as homogeneously as possible to avoid the occurrence of any side reaction between any of the products in the secondary acid reaction mixture and the neutralizing agent.

The neutralized reaction mixture comprises the furfural derivative of the chemical formula (1). The furfural derivative may be recovered from the neutralized reaction mixture by any feasible purification step. A suitable purification step may comprise an evaporation and/or distillation step.

Following the neutralization step the neutralized reaction mixture obtained may be used to recover the furfural derivative of chemical formula (1) by purification of the neutralized reaction mixture. Such recovery may be conducted on the neutralized reaction mixture obtained. However, suitably the neutralized reaction mixture is subjected to separation of at least part of the liquid hydroxyl-containing compound from the neutralized reaction mixture to yield a product mixture; and to recovery of the furfural derivative with the chemical formula (1) from the product mixture. In this way the reactants and the products may be obtained in a convenient way. Suitably at least part of liquid hydroxyl group-containing compounds is removed to yield the product mixture. Such removal may include the removal of any excess R-OH that was added at the start of the reaction, or that is formed during the decomposition of acetal groups, but also any water that has been formed during the reaction. In accordance with the present invention part of the compound R-OH has been removed from the primary acid reaction mixture before neutralization. So for the preparation of the product mixture any remaining R-OH and water formed may suitably be removed from the neutralized reaction mixture. Such a removal step may suitably be carried out in the form of evaporation, flashing or distillation. Subsequently the product mixture may be used to recover the furfural derivative of chemical formula (1). The furfural derivative may be obtained by evaporation or distillation of the product mixture. From such evaporation or distillation also other products, such as levulinic acid, levulinate esters and formic acid, may be recovered as fractions in the purification. The distillation may be carried out in one or more columns, as the skilled person will realize. The evaporation or distillation will result in a bottom residue. The bottom residue may comprise acid catalyst. It may further comprise salts that result from the neutralization of the secondary acid reaction mixture.

Any solids that are obtained in any of the process steps are suitably removed by filtration. The solids comprise in particular humins that are the result of side-reactions of the fructose and/or glucose-containing starting material. Other solids may comprise solid salts, e.g. the salts that result from the addition of the neutralizing agent to the secondary acid reaction mixture. Such salts may suitably comprise the alkali metal and/or alkaline earth metal salts of inorganic acids, such as sulfates, phosphates, chlorides or nitrates, when an inorganic acid is used as acid catalyst. Also such metal salts of organic acids are possible, such as alkali metal and/or alkaline earth metal salts of oxalic, p- toluene sulfonic, methane sulfonic, and trifluoroacetic acid, when such acids have been employed as acid catalyst. When a heterogeneous catalyst has been used, solid salts of products such as levulinic acid or formic acid may be formed. Solids removal is suitably carried out by filtration of the primary or secondary acid reaction mixture. In this way the filtrate of the primary or secondary acid reaction mixture is a homogenous liquid which facilitates handling, such as stirring during the neutralization. The filtered solids, such as humins, are suitably washed with water to remove acid catalyst, if any. Washing can suitably be done with water. In an alternative embodiment, the solids are separated by means of centrifugation.

Alternatively, the solids removal, e.g. filtration or centrifugation, is carried out after neutralization at the neutralized reaction mixture. Due to the neutralization, some solid salts may have been formed. Such salts are then suitably removed together with the humins fraction. If desired, the neutralized reaction mixture may be subjected to an evaporation step to remove at least some of the water and, optionally, alcohol or other volatile components, in order to concentrate the products and the solids so that most, if not all, of the salts formed are precipitated and removed together with the humins.

When the furfural derivative of chemical formula (1) is recovered by evaporation or distillation the bottom residue may comprise any acid catalyst and salts resulting from the neutralization. The bottom residue is suitably washed to remove as much acid catalyst and as much salt as possible so that the remaining washed residue can be either combusted or disposed in another environmentally-friendly way. Thereto, the bottom residue is preferably washed with an aqueous liquid. Thereby, water-soluble salts and acid are suitably removed from the residue.The fructose- or glucose-containing starting material may be selected from a variety of possible feedstocks. The starting material may comprise mono-, di-, oligo- or polysaccharides. The components of particular interest in biomass are those feedstocks that contain a monosaccharide. Examples of suitable monosaccharides include fructose and mixtures of fructose with other monosaccharides, such as other hexoses and/or pentoses. Suitable other hexoses include but are not limited to glucose, galactose, mannose, and their oxidized derivatives, e.g. aldonic acid, reduced derivatives, e.g. alditol, etherified, esterified and amidated derivatives. The di- and oligosaccharide carbohydrates containing more than one saccharide unit, are suitably hydrolysed in the alcohol, resulting in a mixture of dissolved di- and/or oligosaccharides, monomeric saccharide units and/or glycoside units. Examples of suitable disaccharides include maltose, lactose, trehalose, turanose and sucrose, sucrose being preferred. Sucrose is abundantly available and therefore very suitable. The disaccharides can easily be converted into the monomeric units. Examples of suitable oligosaccharide are fructo-oligosaccharides which are found in many vegetables. By oligosaccharides is understood a carbohydrate that is built up of 3 to 10 monosaccharide units. Polysaccharides have more than ten monosaccharide units. These are polymeric structures formed of repeating units joined together by glycosidic bonds. The number of monosaccharide units in a polysaccharide may vary widely, and may range from 10 to 3000. Suitable polysaccharides include fructan, i.e. a polymer of fructose moieties, and levan, which is composed of D- fructofuranosyl moieties. Mixtures may also be used. Hydrolysis process streams from enzymatic or catalytic hydrolysis of starch, cellulose and hemi-cellulose or from alcoholysis processes that already contain mono- and disaccharides can suitably be used as starting material for the present process. In view of the above, the preferred monosaccharide is fructose, glucose and mixtures thereof. A suitable starting material is HFCS, i.e. high fructose corn syrup, comprising a major amount of fructose and some glucose. The preferred disaccharide is sucrose.

The fructose and/or glucose starting material may further comprise glycosides as described in WO 2012/091570.

The conditions under which the present process can be carried out has been generally described in the prior art. Advantageously, the process is carried out as described in US 8877950. For the reaction between the fructose and/or glucose-containing starting material with the compound R-OH that includes preferably a temperature of 175 to 225° C and at a residence time in the flow process from 1 minute to 10 minutes. The pressure is preferably in the range of 5 to100 bar, more preferably from 10 to 40 bar. The process is preferably carried out as a continuous process. The conditions for the neutralization are not critical. The pressure has little influence on the reaction. Therefore, the pressure may vary between wide ranges at the discretion of the skilled person. Suitable pressures include those in the range of 0.1 to 40 bar. Also the temperature for the neutralization may be selected from a wide range and is suitably selected such that the at least part of the secondary acid reaction mixture is neutralized at a temperature in the range of 25 to 150 ºC.

The present invention will be illustrated by means of the following example.

EXAMPLE

To mimic the product stream of a fructose conversion reaction with methanol as described in US 8877950 a composition was prepared comprising methoxymethylfurfural (MMF), hydroxymethylfurfural (HMF), methyl levulinate (ML), water, methanol, sulfuric acid, levulinic acid and formic acid. Two portions of this composition were subjected to two experimental procedures.

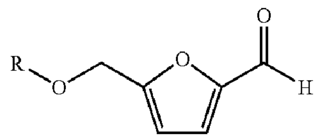
In the first procedure the first portion of the composition was neutralized to a pH of 6 by the addition of aqueous sodium hydroxide under vigorous stirring at 50 - 70ºC. The composition thus obtained was subjected to methanol removal by evaporation in a wiped film evaporator with the jacket set at 115 ºC at 200 mbar. Subsequently, water and other volatile compounds were removed by evaporation in a wiped film evaporator at with a jacket set at 120 ºC at 65 mbar. By further lowering the pressure to 15 mbar MMF and ML were recovered. The stream of MMF contained 3%wt of MMF-acetal.

In the second experimental procedure the second portion of the composition was subjected to methanol removal by evaporation in a wiped film evaporator with the jacket set at 115 ºC at 200 mbar, whereby the majority of the methanol was removed. The mixture thus obtained was neutralized to pH of 6 by the addition of aqueous sodium hydroxide under vigorous stirring at 70 – 100 ºC. Subsequently, water and other volatile compounds were removed by evaporation in a wiped film evaporator at with a jacket set at 120 ºC at 65 mbar. By further lowering the pressure to 15 mbar MMF and ML were recovered. The stream of MMF was analyzed for acetal presence. None was detected.

The result of these experiments show that by removing methanol after the reaction of the fructose-containing starting material but before neutralization, the product stream does not contain any acetal, whereas in a similar process wherein the neutralization was carried out before methanol was removed, the product stream contained a considerable amount of acetal.

CLAIMS

1. Process for the preparation of a furfural derivative having the chemical formula (1)



(1)

wherein R represents hydrogen, an alkyl group or an acyl group, which process comprises

reacting a fructose- and/or glucose-containing starting material with a liquid hydroxyl group-containing compound of formula R-OH in the presence of an acid catalyst at a reaction temperature in the range of 150 to 300 °C to produce a primary acid reaction mixture comprising the furfural derivative of chemical formula (1);

separating at least part of the liquid hydroxyl group-containing compound from the primary acid reaction mixture to yield a secondary acid reaction mixture;

neutralizing the secondary acid reaction mixture to a pH-value of at least 3 to obtain a neutralized reaction mixture; and

purifying the neutralized reaction mixture to recover the furfural derivative of chemical formula (1).

2. Process according to claim 1, wherein R represents an alkyl group.

3. Process according to claim 1 or 2, wherein the liquid hydroxyl group-containing compound is water or methanol or a mixture thereof.

4. Process according to any one of claims 1 to 3, wherein the acid catalyst is a Brønsted acid selected from the group consisting of mineral inorganic acids, organic acids and mixtures thereof.

5. Process according to any one of claims 1 to 4, wherein the secondary acid reaction mixture has a pH of smaller than 2.

6. Process according to any one of claims 1 to 5, wherein the secondary acid reaction mixture is neutralized by the addition of an alkali metal or alkaline earth metal hydroxide or alkoxide.

7. Process according to any one of claims 1 to 6, wherein the neutralized reaction mixture is subjected to separation of at least part of the liquid hydroxyl group-containing compound from the neutralized reaction mixture to yield a product mixture; and to recovery of the furfural derivative with the chemical formula (1) from the product mixture.

8. Process according to claim 7, wherein the neutralized reaction mixture is subjected to evaporation to yield the product mixture.

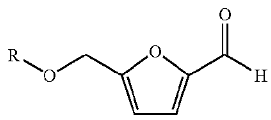
9. Process according to any one of claims 1 to 8, wherein the furfural derivative with the chemical formula (1) is recovered by evaporation or distillation to yield the furfural derivative with the chemical formula (1) and a bottom residue comprising acid catalyst.

10. Process according to claim 9, wherein the bottom residue is washed with an aqueous liquid.

11. Process according to any one of claims 1 to 10, wherein the secondary acid reaction mixture is neutralized at a temperature in the range of 25 to 150 ºC and a pressure of 0.1 to 40 bar.

ABSTRACT

A furfural derivative having the chemical formula (1)



(1)

wherein R represents hydrogen, an alkyl group or an acyl group, is prepared in a process, which process comprises

reacting a fructose- and/or glucose-containing starting material with a liquid hydroxyl group-containing compound of formula R-OH in the presence of an acid catalyst at a reaction temperature in the range of 150 to 300 °C to produce a primary acid reaction mixture comprising the furfural derivative of chemical formula (1);

separating part of the liquid hydroxyl group-containing compound from the primary acid reaction mixture to yield a secondary acid reaction mixture;

neutralizing the secondary acid reaction mixture to a pH-value of at least 3 to obtain a neutralized reaction mixture; and

purifying the neutralized reaction mixture to recover the furfural derivative of chemical formula (1).