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(54) Process for manufacturing fuel from ligno-cellulose material.

(57) The invention relates to a process for manufacturing fuel from ligno-cellulosic material such as wood, straw, nut hulls etc.. Such material can be converted to a cellulosic slurry which contains high sugar and saccarine values after elimination of most of its lignin which may be used as a heat source, or is dried and ground to a fine powder, and is then subjected to a catalytic cracking and reforming process whereby different hydrocarbons are produced which are separated in a separation column.

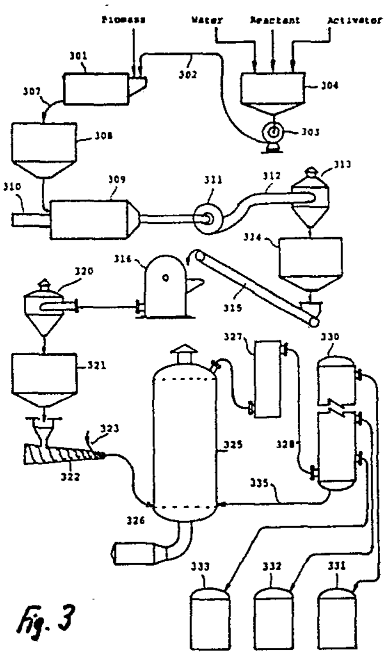

Fig. 3

Process for Manufacturing Fuel from Ligno-Cellulose Material

The invention relates to a process as stated in the prior art portion of claims 1 or 4.

A process comprising the steps of reacting the ligno-cellulose material with sulfur dioxide and water under heat and pressure by introducing steam; mixing the reacted material from the first step with an alkaline solution to dissolve the reacted lignin; and washing the material from the second step to remove the dissolved lignin from the rest of the mixture, is known from EP-B 105 937 by the applicant. It had been known for quite some time (US-A 4 053 645) to increase the digestability of cellulosic material by converting it to a sugar and saccarine compound which can be used for feeding animals. This compound, also, can be fermented and distilled, leading to alcohol which can be used as fuel. This can be carried out in a self-contained process. Fermentation, however, is a batch-process and usually requires 12 to 24 hours, depending upon the type of cellulose derived from the raw material. Thus, this fermentation step considerably limits the productivity of the prior art process. Further, the distillation for separating the different alcohol and ether compounds requires a lot of energy and causes high expenses.

Other attempts to gain fuel from biomass use pyrolyzis of the biomass and then in a next step the pyrolyzis products are chemically condensed, i.e. subjected to a chemical reaction involving union between molecules, possibly at the same time eliminating simple molecules, to form new more complex compounds of greater molecular weight which are intended to at least partially be used as fuel. The achievable yield, however, is rather modest while the energy consumption for keeping the process going is rather high.

The invention aims to enhance the productivity limits and to save costs, working time and energy consumption. This is achieved by the invention characterized in the claims. By the process of the invention, fermentation is eliminated and a high yield is gained, and it is also possible to eliminate distillation. According to one aspect of the invention, characterized in claim 1, the cellulosic slurry which contains the sugar and saccarine values after having removed approximately 95% of the original lignin is the result of a process similar to the known process of EP-B 105 937 (Jelks - Strouth) as described up to Col. 5, line 29 thereof. Instead of fermentation and distillation, the process of the invention uses the cracking procedure as conventionally used for the production of petroleum products. Cracking in its broadest sense has already been contemplated also for biomass products (BE-A 349 622 = DE-A 567 330) but without practical utilization. If in the invention the input material which according to the process of claim 1 is a slurry is to be further liquified for enabling the cracking process, part of the cracking product may be re-cycled. 5 to 10% of the cracked/reformed product will be a tar. This tar product is returned to the cracking chamber for recycling and re-appears with the cracked/reformed product without loss. Much of the heat consumed in the process can be recovered from the process itself. In some cases a combustible byproduct is produced which can be used for steam generation etc. Separation may be carried out by a separation column which allows to discriminate the different products. The waste heat of these partial processes can be again exploited for the process. What is required for a pilot plant having an input of 1 t per day of dry biomass which originally contained 35% moisture is approximately 250 kW for the digester heating and part of the heat needed for cracking. The plant as described can be run by three to two men and can produce approximately 380 l (85 imperial gallons) of fuel per day.

According to another aspect of the invention, cracking is carried out not only on the basis of the cellulose material but also on the basis of the lignin contained in the biomass, the process comprising the steps of pyrolyzing the ligno-cellulose material so as to crack it and obtain gaseous pyrolyzis products, and carrying out a catalytic condensation reaction of the pyrolyzis products to result in a series of compounds from which such usables as fuel are separated. No energy being gained from the lignin content of the biomass, it is desired to work with minimum energy consumption and maximum yield, which is obtained by the measure that before pyrolyzing, the ligno-cellulose material is pulverized, and that pyrolyzation and the catalytic reaction take place in one same high-temperature reaction zone, into which the pulverized material is transported to be mixed with catalyst powder. By having dry materials to be processed, the heating energy is much less, and by getting the places of pyrolization and catalytic reaction as close together as possible, a maximum yield is obtained.

The subjects of claims 5 and 6 serve to obtain a very suitable fine-grain powder as defined in claim 7, the particular digestion step leading to a very brittle wood material which can easily be ground. The chemical treatment of the biomass is preferably as described in other context in US-PS 4 053 645.

According to claims 8 and 9, the pulverized biomass is transported into contact with the catalyst by means of a fluid stream which preferably is superheated steam which proves the cheapest while at the same time heating the reaction zone.

The process can be carried out for increased energy saving in the manner as defined in claim 13, using immediately part of the product which part might be less suitable as fuel, for the system heating. Further, it is possible to re-cycle part of the product so as to support transport and reaction.

The drawing shows schematic arrangements of process steps and of apparatuses which may be employed in practising the invention.

Fig. 1 shows a schematic diagram illustrating the process wherein the material is supplied in slurry status to the reaction zone;

Fig. 2 shows a similar arrangement for cracking powdered biomass which for pre-processing has been separated into a lignin and into a cellulose path; and

Fig. 3 shows a schematic diagram for reacting pulverized biomass which had been pre-processed un-separated.

In Fig. 1, first and second globe rotary digesters 10 and 12 are shown. While only two digesters are shown, it is possible to use only a single digester or any number of digesters for the purposes of this invention. The use of more than one digester, however, is preferred since the reaction which takes place within each of the digesters is a batch process. By using a plurality of digesters the procedure can be carried out as a continuous process. Each of the globe rotary digesters 10, 12 is an apparatus which is used to subject the contents to heat and pressure while the contents is being mixed. Any device which accomplishes these results would be within the scope of the invention, whether carried out as a batch process or a continuous process.

A ligno-cellulose material cut into small pieces is deposited in digester 10 to substantially fill the digester, however leaving sufficient room so that the material will mix as the digester rotates. The chosen ligno-cellulose may be any type of wood, straw, bagasse, nut hulls, seed hulls, etc. The woody material is first chopped or ground into particles. The size of the particles should be not more than approximately 7 mm (1/4") but may be as fine as desired. The size of the particles is not important except that it is apparent that the smaller the particles the more expeditious will be the subsequent reaction. Thus the particle size is a matter of engineering trade-off between the cost of reducing the ligno-cellulose material to smaller sizes versus the time utilized in the steps of reaction.

After the ligno-cellulose material (hereinafter called wood) is deposited in digester 10, water and sulfur dioxide are added. The water is added at a ratio of approximately 40% by weight of water to the dry weight of the wood. Sulfur dioxide is added in form of a gas at a ratio by weight of about ½ to 6% of the dry weight of the wood. The quantity of sulfur dioxide is predicated upon the percentage of lignin compared to cellulose contained in the wood. The quantity of lignin is less when cottonwood species is used as a raw material, compared to greater percentage when oak or pine are used as raw material with the quantity of sulfur dioxide adjusted, accordingly. After the wood, sulfur dioxide and water have been added, the contents of the digester 10 are pressurized by the introduction of steam. Sufficient steam is introduced to bring the temperature of the reaction mixture to between 44°C and 200°C, a range between 49°C and 200°C being preferred, and at a pressure of between 2463 and 15750 hPa (35 and 225 psi). The mixture is cooked at this pressure and temperature for a period of about 40 minutes to 1 hour. However, the duration is determined by the particulate size of the wood raw material and the characteristics of the material.

After a cooking cycle is completed in digester 10, a valve 14 is opened to allow steam to escape to the atmosphere. After the digester has been depressurized, the loading door is opened and the contents discharge onto a digester pump pit 16. After the digester is emptied, it is refilled with wood type material, water, and sulfur dioxide and the process is repeated. By use of the second digester 12 it can be seen that one digester may be in the process of being emptied and refilled while the other digester is in the cooking cycle so that reacted raw material is deposited into the digester pump pit 16 on a more frequent basis. By the use of a greater number of digesters the input can be even more frequent so that the digester pump pit 16 always contains a quantity of reacted material for subsequent processing on a continuous basis.

The reacted material in digester pit 16 is in the form of a slurry and, if necessary, additional water may be added to the pit to maintain the slurry as a pumpable mixture. The digester pump pit includes stirring apparatus 18 to make sure the reacted material does settle out and is retained as a pumpable slurry.

By means of a pump 20 the slurry is conducted to a mixing box 22 where the mixture is mixed with an alkaline solution from line 24. The source of the alkaline solution which is conducted through line 24 will be described subsequently.

The reaction of the wood in digesters 10 and 12 is such that the sulfur dioxide in the presence of water reacts with the lignin content of the wood to enhance the solubility of the lignin content without destroying the carbohydrate content. Lignin is a fairly complex organic material, varying somewhat from one plant species to another and the exact reaction with sulfur dioxide is not precisely known;

however, it has been experimentally verified that the reaction of the sulfur dioxide and wood produces a solution wherein when mixed with an alkaline solution the lignin is dissolved.

The processed mixture and alkaline solution flows out of mixing box 22 through line 26 into a vacuum washer 28 for separating lignin and cellulose materials. This apparatus is a known type of equipment utilized in the chemical processing industry, and particularly in the paper making industry. Therefore, it is well understood by practitioners in the art and will not be described in detail. Suffice it to say that in a vacuum washer a cylinder is equipped which is rotated in the solution. The interior of the cylinder is impressed with a vacuum. Water is sprayed onto the cylinder from above the solution to wash away the dissolved matter, the cellulose material, being fibrous in nature, forms a mat on the cylinder. The fresh water utilized to wash the lignin content from the matted cellulose is supplied by line 30. The vacuum required by the washer 28 may be supplied by a vacuum pump or by means of, as illustrated, a barometric leg 32 which communicates with an overflow box 34. The dissolved lignin is carried out of the overflow box by line 36 while the cellulose is rolled off the vacuum forming cylinder into a receiving box 38. This matted material is still in a slurry form and is carried by a pump 40 into a second mixing box 42 where the cellulosic material is mixed with water from line 30. The washing procedure is repeated in a second vacuum washer 44 having a barometric leg 46 and overflow box 48 by which the overflow liquid content is carried away from the vacuum washer.

The matted cellulose content is rolled off the cylinder into a receiving box 50. The content of receiving box 50 is properly washed material which has more than 95% of the original lignin content removed. It is passed on via a line 52.

While the use of vacuum washers 28 and 44 has been illustrated herein, it is understood that alternate washing procedures can be used in practice of the invention and that the particular means of washing the reacted ligno-cellulose material mixed with an alkaline solution is not a critical element in the practice of the invention and that other known washing systems may be employed. The essence of the washing step is simply to separate the dissolved lignin content from the cellulose material.

The materials needed in the process include sulfur dioxide, a metal iron catalyst from that group of metal having a valence of 3, an alkaline solution, water, heat and steam, produced by the process itself.

The lignin solution from overflow box 48 is taken by line 90 to a dissolving tank 92, if separated. Melt from the melt pit 94 of a boiler 96 also passes into the dissolving tank 92, when used.

The lignin solution from overflow box 34 is carried by line 36 to a set of multi-effect evaporators 98, 100, and 102. The number of such evaporators may be varied. The purpose of the evaporators is to evaporate the water content from the dissolved liquid solution. To obtain the heat necessary for the evaporation step, steam is applied from line 104 to a heat exchanger 106 in evaporator 102. Steam from evaporator 102 is passed by line 108 to a heat exchanger 110 in evaporator 100. In like manner, steam from evaporator 100 is passed by line 112 to a heat exchanger 114 in evaporator 98. The condensate from evaporator 98 is passed by line 116 into evaporator 100. The condensate from evaporator 100 is passed by line 118 into evaporator 98. The condensate from evaporator 102 is passed by line 120 into a contact evaporator 122. The lignin content of the solution from the overflow box 34 will normally be of about 20% solids, more or less, and the balance liquid. This solution will be concentrated so that the ligning content passing from the multi-effective evaporators in line 120 will be about 55% more or less. In the contact evaporator 122 this solution is brought in contact with recovery boiler stack gases from boiler 96. The solution in the contact evaporator 122 is concentrated to about 65% solids which are carried into a burner 124. The organic solids burns in burner 124 and boiler 96 to produce significant quantities of heat. This heat is used to convert water in the boiler to steam which passes out through steam line 126. The inorganic portion of the burned material is primarily sodium and ends up as molten sodium carbonate in the melt pit 94 under boiler 96.

The molten sodium carbonate passes into the melt pit 94 and from there into dissolving tank 92. When mixed with water in the dissolving tank an alkaline solution forms which is conveyed by pump 128 and line 24 into the mixing box 22. Thus the burned lignin content of the wood raw material is used to produce the alkaline solution for the process. Obviously, if additional alkaline solution is required, it may be supplied by supplementary means.

Steam from boiler 96 passing out through steam line 126 is conveyed to a turbine generator 130 to produce electrical energy. The turbine is preferably of the bleeder type which generates electricity and provides process steam by bleeding it from an intermediate point in the turbine at line 132. This process steam is carried by line 134 to the globe rotary digesters 10 and 12 and by line 104 to a multi-effect evaporator 102. Spent steam from the multi-effect evaporators may be collected by apparatus not shown and condensed to provide the water which is reused for input into the globe digesters 10, 12, the mixing box 42, and to spray water on the washers 28 and 44.

The material supplied through line 52 is a cellulosic slurry which contains high sugar and saccarine values, approximately 95% of the original lignin having been eliminated. This slurry, however, still contains a lot of water part of which is vaporized in a heating unit 140. It is then passed through the heating unit 140 and from thence via a line 141 to a cracking reaction chamber 142. If for the further processing it is necessary to re-liquify it so as to have a pumpable mixture, this can be accomplished by recycling and adding approximately 5 to 10%, releating to the mixture weight, of the end product to the slurry, the respective end product being supplied into line 52 via a line 139. This material is subjected to the cracking process known for petrochemical materials which process is here used to crack the sugar and saccarine material with the aid of a catalyst into products useful as high octane fuel, the resultant materials comprising alcohol and ether compounds such as pheno glyserol, T-6 di methoxy phenol, di pheno ether, phenolic alcohol, 1, sodugeno, dugenol, di methol pheno guiacol, cresole, methyl xylene, proplene methol benzene, benzene prople xylene.

The cracking process uses a molecular sieve, a red-hot catalyst bed heated to 400° C. The catalyst used is commercially available and recoverable by ion exchange in the presence of steam of approximately 0.1 to 0.2 MPa (1 to 2 atm). The cracking reaction takes 1 to 10 milliseconds approximately in the reaction chamber 142, or about 5 to 7 minutes resident time. Exhaust heat from the cracking stage is used in the process, e.g. by additionally heating the steam in line 104 which can be taken from near the exhaust exit of the turbine 130.

The reaction products of the cracking process are transported via a line 143 to a separation column 145. The separation column breaks down the cracked materials into various fractions. The material enters as a mixture of hot vapours the column and therein rises through the bubble caps and condenses on different trays. The lighter fractions do not condense until they reach the cooler trays, i.e. the more up trays. The heavier fractions condense on the lower trays. Among the lighter fractions are those which can be used as fuel. If desired. some heavier fractions can be re-cycled via a line (not shown) to the heating unit 140 and hence into the cracking reaction chamber 142. Some residue which is unuseable is diverted via a line 147. 5 to 10% of the cracked/reformed product will be a tar. This tar product is returned to the cracking chamber for recycling and re-appears with the cracked/reformed product without loss.

The reaction chamber 142 needs steam which is taken from line 104.

The described method provides a substantially self-contained process for manufacturing fuel out of non-digestible organic ligno-cellulose material in a manner wherein a supplementary energy source for supplying heat is either not required or if supplemental energy is required such need is greatly minimized.

The drawings of Fig. 1 is separated by a chain line indicating the interface beyond which the arrangement of Fig. 2 is different. Accoring to the process of Fig. 2, the slurry of cellulose material from receiving box 50 is supplied by a pump 201 to a dryer 202 which also receives the concentrated lignin solution from line 120 and receives heating steam from the turbine 130 via line 132. In the dryer 202, the already more or less concentrated slurry material is fully dried and sent via a line 203 to a pulverizer 204 where it is pulverized to a very fine dry powder, sufficiently fine to pass a 100-mesh screen.

The pulverized material is transported to a cracker/reformer 205 wherein, in a same reaction zone, the powder is cracked by pyrolyzation and is reformed by condensation. In the cracker/reformer 205, the powdered biomass is introduced into a hot environment, i.e. in a catalyst bed being fluidized by a carrying gas which preferably is superheated steam which is cheapest to arrange, or is an inert gas which may be cold or warm up to 110 to 160° C, which is a temperature well below the temperature where pyrolyzis starts. The fluidized bed of the catalyst has a much higher temperature, the carrier gas thereof - of the same kind as the transporting carrier gas for the biomass - being revolved by a blower pump 206 and in this cycle being heated by a heater 207.

The biomass powder entering the reaction zone is heated and pyrolyzis takes place, resulting in fragmented molecules which enter the catalyst immediately and chemically condense in contact therewith to form mainly liquid materials satisfactory for use as gasoline fuel or to be blended with gasoline. The catalyst is a zeolyte type catalyst, i.e. SZM-5. At output line 143, the reaction products are supplied to the separation column 145 as before. The reaction also results in some waste gas and in the separation column 145, some non-condensable gas is formed. This waste gas from the cracker/reformer 205 is transported via a line 208 to a fuel gas storage also acting as a blender, as it further receives fuel gas from outside by a line 210. Part of this gas is recirculated via a line 211 to the cracker/reformer 205, and part of it as well as the non-condensable gas from the column 145 is transported via a line 212 or 213, respectively, to the boiler 96 which also has a supply 214 of combustion air, for heating and thus generating process steam. Further, as in Fig. 1, some tar products are recirculated via line 148 into the cracker/reformer.

By combining the pyrolyzis and the chemical condensation in the same reaction zone, a considerably high yield is obtained in this step, leading generally to methyl-benzenes and to a small decree to by-products such as coke, carbon oxides and water. If on the other hand the pyrolyzed biomass would have been allowed to cool before condensation, the resulting large molecules would also be reformed to the desired compounds, but would shed water and carbon to a higher decree which would lower the yield of the desired compounds.

Fig. 3 shows a third variant of the process. The biomass, i.e. saw dust, chips etc., is mixed in a mixer 301 with a reaction solution coming via a line 302 from a mix tank 304 which is supplied by water, reactant and activator as described somewhat later. The mixture of biomass and solution is supplied by a line 307 to a wet chip storage and cooking tank 308 where it is subjected to boiling temperature and from there to a dryer 309 which is combined with a furnace 310 for delivering heat for drying. After careful drying, the bone-dry, brittle biomass is transported, via a blower 311 and a separator 313, to a dry chip storage 314 and from there, via a feeder 315, to a mill 316 grinding the dry chips to a fine powder similarly to be specified as in the description of Fig. 2. The powder passes again a separator 320, a storage 321, a powder feeder 322 and then, transported by a carrier gas stream 323, enters cracker/reformer 325 combined with a catalyst bed heater 326. The output of the cracker/reformer 325 passes a cooler 327 and then is supplied via a line 328 to separation column 330 from which the different final products are extracted and stored in storage tanks, a tank 331 for light, a tank 332 for medium and a tank 333 for heavy hydrocarbons. Tar products are re-cycled to the cracker/reformer 325 via a line 335 for renewed processing.

Of some interest is the preparation of the biomass before cooking and drying. It is carried out in a manner similar to what has been desribed in US-A 4053645 (Jelks). The reactant and activator comprise an oxydizing acid like nitric acid (HNO_3), a non-oxydizing acid such as sulfuric, phosphoric, hydrochloric or acetic acid and a tri-valent metal such as iron which has the function of a catalyst. Nitric acid which serves to deliver some required oxygen is requested in an amount of approximately 2 to 30 grams per kg of dry weight of the biomass.The materials are cooked, if necessary under pressure, with the effect oc attacking the lignin-cellulose bond and downgrading and saturating the cellulose, and then are carefully dried resulting in a very brittle material which can easily be ground to the required fine powder, demanding only rather low grinding energy. Careful drying will help to circumvent the application of pressure during digestion in the cooking step. The moderate energy consumption for grinding and for the pyrolyzis of the dry material, together with the high yield of the locally combined pyrolyzis and condensation, or in other words, cracking and reforming steps, makes the present process very economic.

While the invention, as above indicated, has been described in certain detail, it is understood that the invention is not limited to the embodiments set forth herein but is to be limited by the scope of the attached claims, including the full range of equivalency to which each element thereof is entitled.

Claims

1. Process for manufacturing fuel from ligno-cellulose material, to be carried out by the following steps:

a) reacting the ligno-cellulose material with sulfor dioxide and water under heat and pressure by introducing steam (in 10, 12);

b) mixing the reacted material from step a) with an alkaline solution to dissolve the reacted lignin (in 22);

c) washing the material from step b) to remove the dissolved lignin from the rest of the mixture (in 28); characterized by the further steps of

d) cracking the rest of the mixture (in 142) from step c) in a reaction zone with the aid of a heated catalyst; and

e) separating the cracking products.

2. Process according to claim 1, characterized in that as catalyst a molecular sieve is used.

3. Process according to claims 1 or 2, characterized in that 5 to 10% of weight of the cracked product, i.e. tar fraction separated in a separation column, is recycled to the cracking reaction chamber.

4. Process for manufacturing fuel from ligno-cellulose material, to be carried out by the steps of pyrolyzing the ligno-cellulose material so as to crack it and obtain pyrolyzis products, and carrying out a catalytic condensation reaction of the pyrolyzis products to result in a series of compounds from which usable fuels are separated, characterized in that before pyrolyzing, the ligno-cellulose material is pulverized, and that pyrolyzation and the catalytic reaction take place in one same high-temperature reaction zone, into which

the pulverized material is transported to be mixed with the catalyst.

5. Process according to claim 4, characterized in that the ligno-cellulose material, before being pulverized, is subjected to digestion.

6. Process according to claim 4, characterized in that the ligno-cellulose material, for digestion and pulverization, is saturated by a treatment with an acid with a tri valent metal ion under heat and pressure so as to attack the lignin cellulose bond and downgrade the cellulose, and then is dried and ground.

7. Process according to claim 4, characterized in that the lignocellulose material, for digestion, is saturated by a treatment with an acid with a tri valent metal ion, the saturated cellulose material being dried under conditions using dry air at a temperature under 100°C, so as to attack the lignin cellulose bond and downgrade the cellulose for grinding.

8. Process according to any of claims 4 to 7, characterized in that the ligno-cellulose material is pulverized to a grain size corresponding to less than 100 mesh.

9. Process according to any of claims 4 to 8, characterized in that the pulverized material and the catalyst powder are transported into a reaction chamber by a fluidized stream.

10. Process according to any of claims 4 to 8, characterized in that the reaction zone is a fluid bed carrying the catalyst powder, the pulverzied material being blown to this zone by a fluidized stream.

11. Process according to claims 9 or 10, characterized in that the fluid stream consists of superheated steam.

12. Process according to claims 9 or 10, characterized in that the fluid stream consists of an inert gas having a temperature which is lower than that required to pyrolyze the biomass.

13. Process according to any of claims 1 to 12, characterized in that the lignin material and the cellulose material are separated for processing, and are re-combined at least before the cracking step.

14. Process according to claim 12, characterized in that a combustible fuel is made for burning in a boiler (96) to produce at least part of the necessary heat from the dissolved lignin, and that waste heat from the cracking reaction chamber is collected to produce hot steam.

15. Process according to claim 14, characterized in that ash resulting from burning is used to provide an alkaline solution to dissolve modified lignin.

16. Process according to any of claims 1 to 15, characterized in that separation is carried out by feeding the chemical product output into a separation column.

17. Process according to any of claims 1 to 16, characterized in that as the catalyst a zeolit is used, the temperature in the reaction zone being 250°C to 600° C.

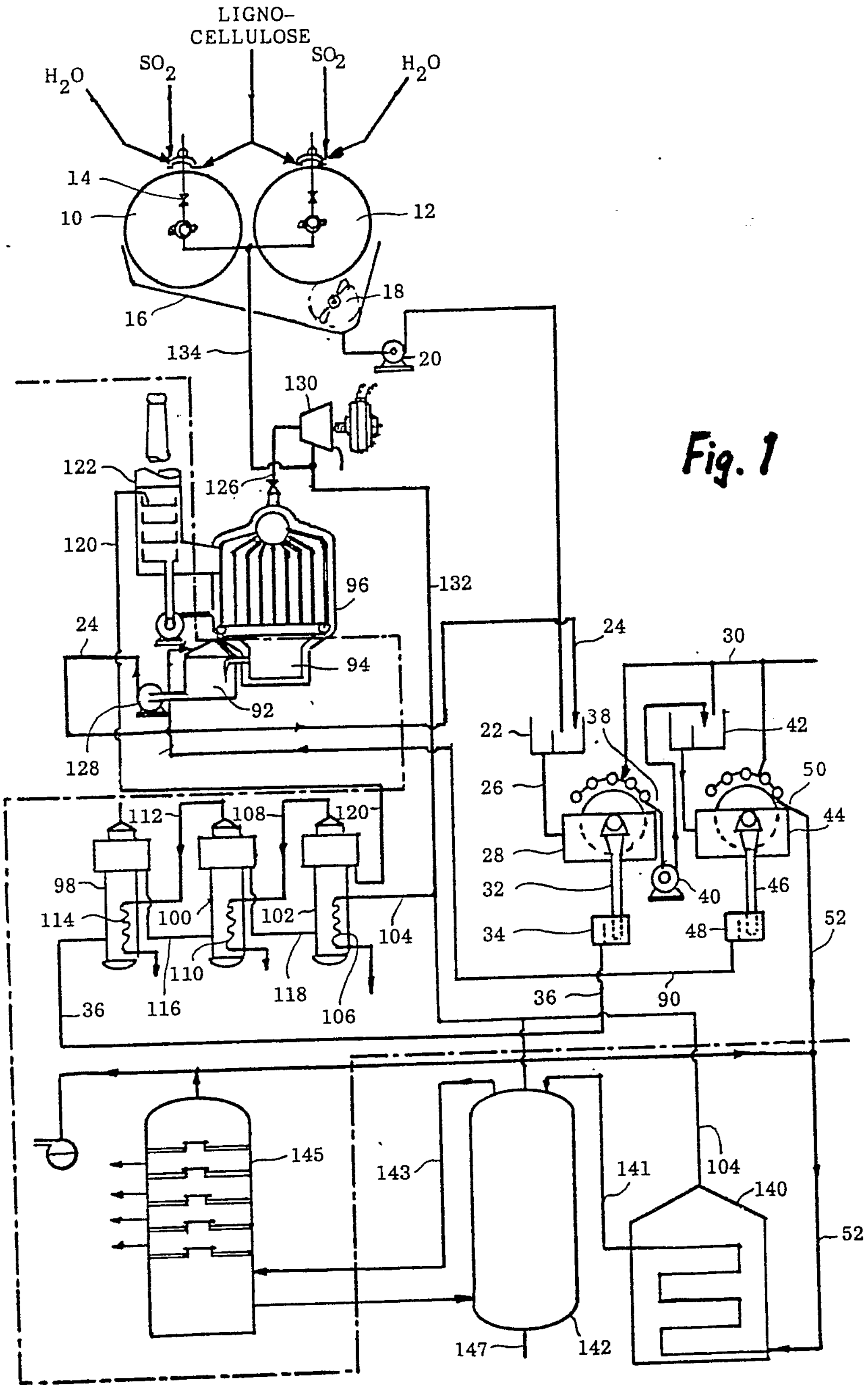
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Fig. 1

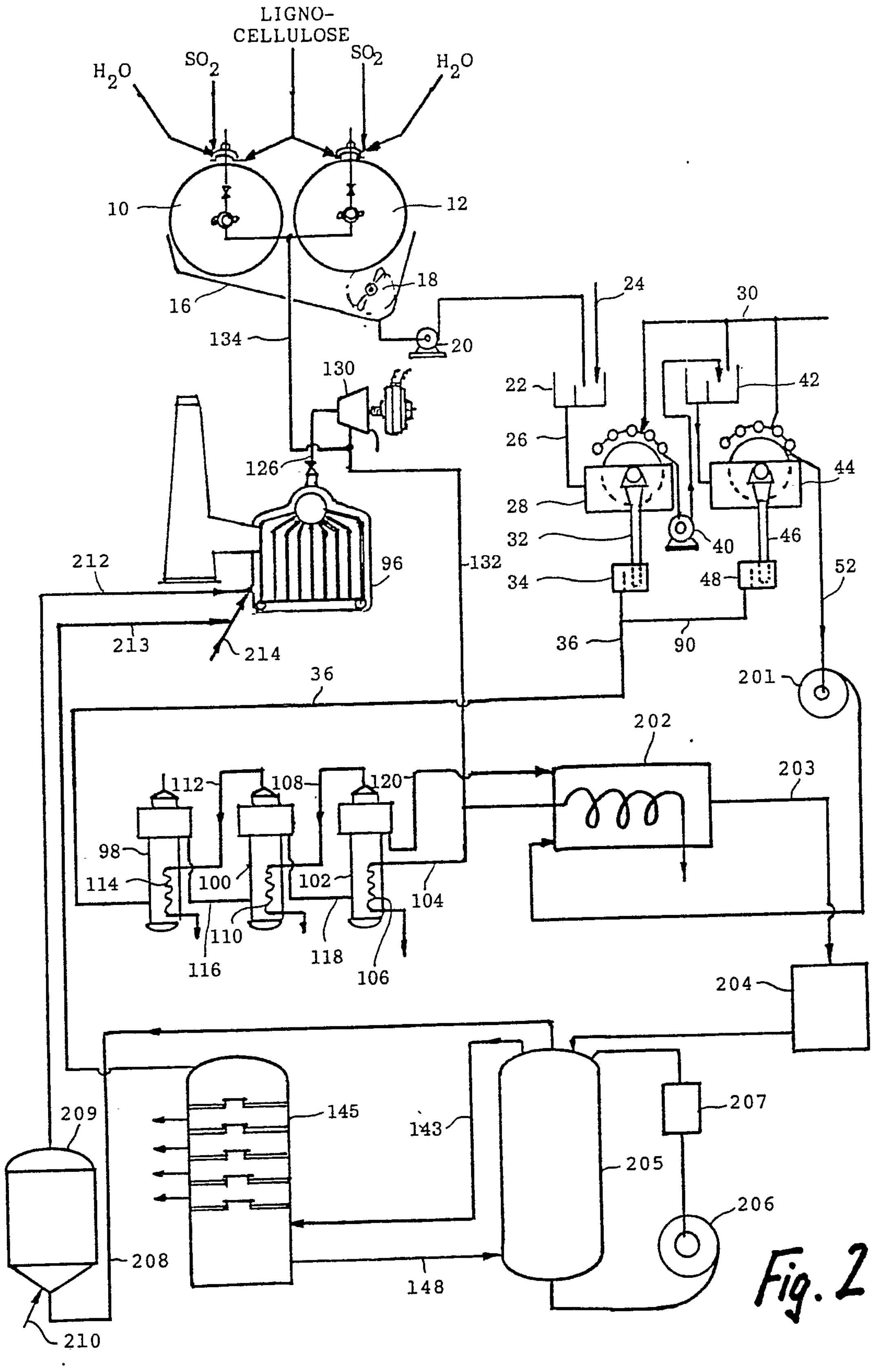
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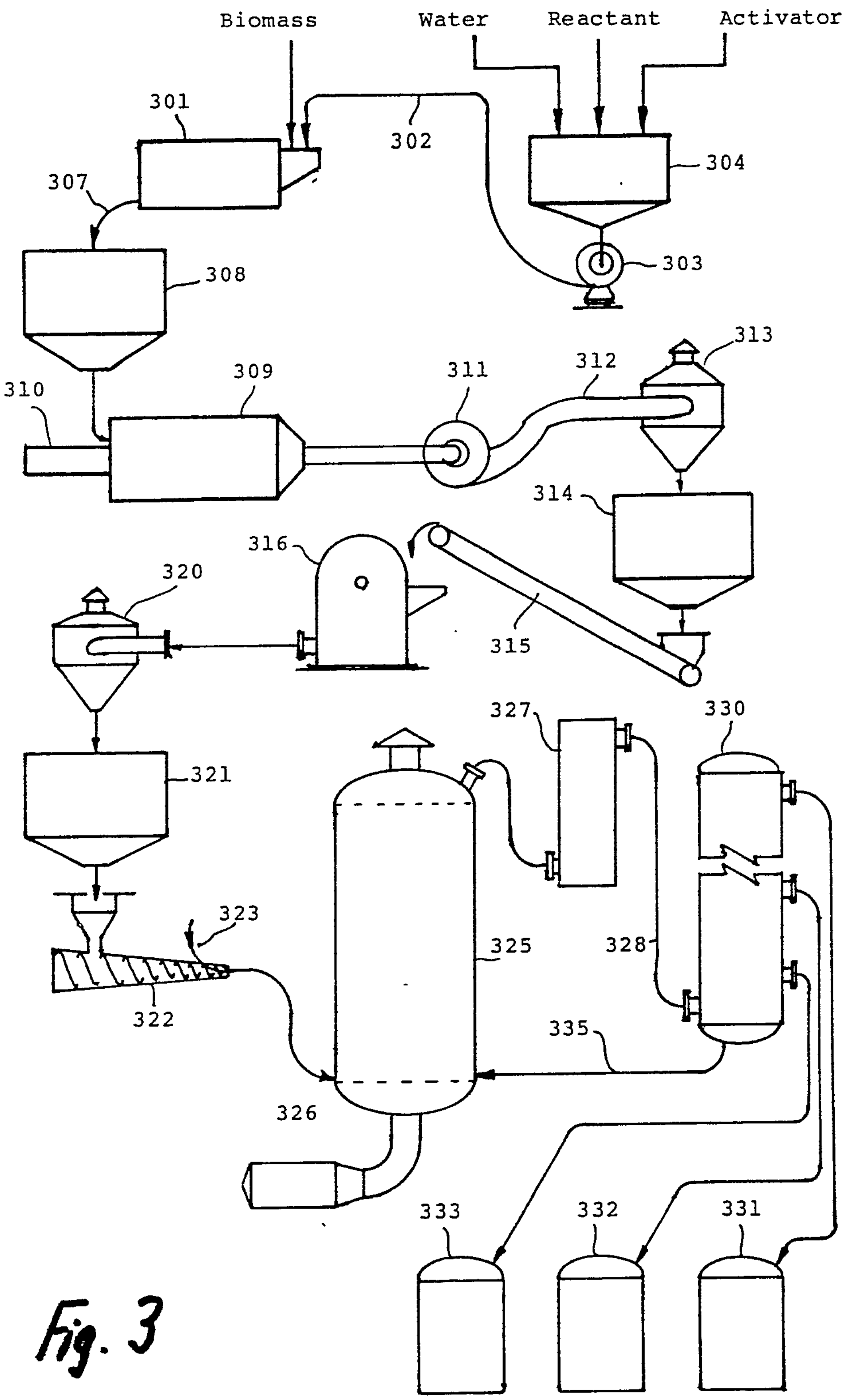
Fig. 2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Water
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Activator
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Fig. 3