CLAIMS

What is claimed is:

1. A method of increasing the level of soluble C_5 saccharides produced from lignocellulosic biomass, comprising:

providing a fractionated lignocellulosic biomass comprising:

a solid fraction comprising:

optionally, insoluble C_5 oligosaccharide;

cellulose; and

lignin; and

a first liquid fraction at a first temperature and a first pressure comprising:

soluble C_5 saccharides selected from the group consisting of C_5 oligosaccharides, xylose, arabinose, lyxose, ribose, and mixtures thereof;

wherein said soluble C_5 saccharides are present at a first level;

acidifying said fractionated lignocellulosic biomass to form a second liquid fraction comprising soluble C_5 saccharides at a second lever and at a second temperature;

wherein said second level is higher than said first level;

optionally, separating said solid fraction from said second liquid fraction; and

optionally, hydrolyzing said second liquid fraction to form C_5 saccharides selected from the group consisting of C_5 oligosaccharides having fewer mer units, xylose, arabinose, lyxose, ribose, and mixtures thereof.

2. A method of claim 1,

wherein said acidifying comprises adding to said fractionated lignocellulosic biomass at least one aqueous acid selected from the group consisting of an organic acid, an inorganic acid, and mixtures thereof.

3. A method of claim 2,

wherein said inorganic acid is sulfuric acid, hydrochloric acid, phosphoric acid, nitric acid, or a combination thereof.

4. A method of claim 3,

wherein said inorganic acid is present at a level of about 0.05%, by weight, to about 2.0%, by weight, based on the weight of said first liquid fraction.

5. A method of claim 1,

wherein said acidifying comprises contacting said fractionated lignocellulosic biomass with a gaseous compound that forms acid *in situ*.

6. A method of claim 5,

wherein said gaseous compound is SO_2, CO_2, NO_2, HX wherein X is Cl, Br, F, or I, or a combination thereof.

7. A method of claim 1,

wherein said acidifying comprises contacting said fractionated lignocellulosic biomass with a solid acid catalyst.

8. A method of claim 7,

wherein said solid acid catalyst is a zeolite, an anionic exchange resin, or a combination thereof.

9. A method of claim 1,

wherein said method is continuous.

10. A method of claim 1,

wherein said second level of said soluble C_5 saccharides is greater than said first level of said soluble C_5 saccharides.

11. A method of claim 1,

wherein said soluble C_5 saccharides in second liquid fraction have an average molecular weight that is lower than the average molecular weight of said soluble C_5 saccharides in said first liquid fraction.

12. A method of claim 1,

wherein said C_5 oligosaccharides in said first liquid fraction have about 2 mer units to about 25 mer units; and

wherein said C_5 oligosaccharides in said second liquid fraction have about 2 mer units to about 15 mer units.

13. A method of claim 1,

wherein said fractionated lignocellulosic biomass is formed by contacting said lignocellulosic biomass with a first reaction fluid comprising hot compressed water and, optionally, carbon dioxide;

wherein said first reaction fluid further comprises acid, when said lignocellulosic biomass comprises softwood;

wherein said first reaction fluid is at a temperature of at least 100°C under a pressure sufficient to maintain said first reaction fluid in liquid form.

14. A method of claim 1,

wherein said first temperature and said second temperature are substantially the same.

15. A method of claim 14,

wherein said first temperature and said second temperature are both no greater than about 240°C.

16. A method of claim 1,

wherein said second temperature is less than said first temperature.

17. A method of claim 16,

wherein said first temperature and said second temperature are both no greater than about 240°C.

18. A method of claim 1, further comprising:

reducing said first pressure of said first liquid fraction prior to separating said solid fraction from said second liquid fraction.

19. A method of claim 1,

wherein said hydrolyzing said second liquid fraction to form C_5 saccharides comprises contacting said C_5 oligosaccharides with dilute acid for a time sufficient to hydrolyze said C_5 oligosaccharides to C_5 oligosaccharides having fewer mer units, C_5 monosaccharides, and mixtures thereof.

20. A method of claim 1,

wherein said hydrolyzing said second liquid fraction to form C_5 saccharides comprises enzymatic hydrolysis.

21. A method of claim 1,

wherein the yield of said xylose is at least 68% of theoretical yield.

22. A method of claim 1,

wherein the yield of said soluble C_5 saccharides is at least 68% of theoretical yield.

23. A method of claim 1, further comprising:

producing at least one of said acetic acid and furfural from said soluble C_5 saccharides.

24. A product produced by the method of claim 1.

25. A composition, comprising:

soluble C_5 oligosaccharides capable of being membrane separated; and

water;

wherein said composition has a pH of about 1.5 to about 6.0; and

wherein said soluble C_5 oligosaccharides have about 2 mer units to about 15 mer units.

26. A method of increasing the level of soluble C_5 saccharides produced from lignocellulosic biomass, comprising:

providing a fractionated lignocellulosic biomass comprising:

a solid fraction comprising:

optionally, insoluble C_5 oligosaccharide;

cellulose; and

lignin; and

a first liquid fraction at a first temperature and a first pressure comprising:

soluble C_5 saccharides selected from the group consisting of C_5 oligosaccharides, xylose, arabinose, lyxose, ribose, and mixtures thereof;

wherein said soluble C_5 saccharides are present at a first level;

optionally, separating said solid fraction from said first liquid fraction at a second pressure;

wherein said first pressure and said second pressure are substantially the same;

contacting said first liquid fraction with a solid acid catalyst to form a second liquid fraction comprising said soluble C_5 saccharides at a second level and at a second temperature;

wherein said second level is greater than said first level; and

optionally, hydrolyzing said second liquid fraction to form C_5 saccharides selected from the group consisting of C_5 oligosaccharides having fewer mer units, xylose, arabinose, lyxose, ribose, and mixtures thereof.

27. A method of claim 26,

wherein said solid acid catalyst is a zeolite, an anionic exchange resin, or a combination thereof.

28. A method of claim 26,

wherein said method is continuous.

29. A method of claim 26,

wherein said second level of said soluble C_5 saccharides is greater than said first level of said soluble C_5 saccharides.

30. A method of claim 26,

wherein said soluble C_5 saccharides in second liquid fraction have an average molecular weight that is lower than the average molecular weight of said soluble C_5 saccharides in said first liquid fraction.

31. A method of claim 26,

wherein said C_5 oligosaccharides in said first liquid fraction have about 2 mer units to about 25 mer units; and

wherein said C_5 oligosaccharides in said second liquid fraction have about 2 mer units to about 15 mer units.

32. A method of claim 26,

wherein said fractionated lignocellulosic biomass is formed by contacting said lignocellulosic biomass with a first reaction fluid comprising hot compressed water and, optionally, carbon dioxide;

wherein said first reaction fluid further comprises acid, when said lignocellulosic biomass comprises softwood;

wherein said first reaction fluid is at a temperature of at least 100°C under a pressure sufficient to maintain said first reaction fluid in liquid form.

33. A method of claim 26,

wherein said first temperature and said second temperature are substantially the same.

34. A method of claim 33,

wherein said first temperature and said second temperature are both no greater than about 240°C.

35. A method of claim 26,

wherein said second temperature is less than said first temperature.

36. A method of claim 35,

wherein said first temperature and said second temperature are both no greater than about 240°C.

37. A method of claim 26, further comprising:

reducing said first pressure of said first liquid fraction prior to separating said solid fraction from said second liquid fraction.

38. A method of claim 26,

wherein said hydrolyzing said second liquid fraction to form C_5 saccharides comprises contacting said C_5 oligosaccharides with dilute acid for a time sufficient to hydrolyze said C_5 oligosaccharides to C_5 oligosaccharides having fewer mer units, xylose, arabinose, lyxose, ribose, and mixtures thereof.

39. A method of claim 26,

wherein said hydrolyzing said second liquid fraction to form C_5 saccharides comprises enzymatic hydrolysis.

40. A method of claim 26,

wherein the yield of said xylose is at least 68% of theoretical yield.

41. A method of claim 26,

wherein the yield of said soluble C_5 saccharides is at least 68% of theoretical yield.

42. A method of claim 26, further comprising:

producing at least one of said acetic acid and furfural from said soluble C_5 saccharides.

43. A product produced by the method of claim 26.

44. A method of increasing the level of fermentative or catalytic product produced from lignocellulosic biomass, comprising:

providing a fractionated lignocellulosic biomass comprising:

a solid fraction comprising:

optionally, insoluble C_5 oligosaccharide;

cellulose; and

lignin; and

a first liquid fraction at a first temperature and a first pressure comprising:

soluble C_5 saccharides selected from the group consisting of C_5 oligosaccharides, xylose, arabinose, lyxose, ribose, and mixtures thereof;

wherein said soluble C_5 saccharides are present at a first level;

acidifying said fractionated lignocellulosic biomass to form a second liquid fraction comprising said soluble C_5 saccharides at a second level and at a second temperature;

wherein said second level is greater than said first level;

optionally, separating said solid fraction from said second liquid fraction;

hydrolyzing said second liquid fraction to form C_5 saccharides selected from the group consisting of C_5 oligosaccharides having fewer mer units, xylose, arabinose, lyxose, ribose, and mixtures thereof; and

fermenting, catalyzing, or fermenting and catalyzing said C_5 saccharides to form said fermentation product, said catalytic product, or a combination thereof.

45. A method of claim 44,

wherein said fermentative product or said catalytic product is ethanol, butanol, or a combination thereof.

46. A method of claim 44,

wherein said acidifying comprises adding to said fractionated lignocellulosic biomass at least one aqueous acid selected from the group consisting of an organic acid, an inorganic acid, and mixtures thereof.

47. A method of claim 46,

wherein said inorganic acid is sulfuric acid, hydrochloric acid, phosphoric acid, nitric acid, or a combination thereof.

48. A method of claim 46,

wherein said inorganic acid is present at a level of about 0.05%, by weight, to about 2.0%, by weight, based on the weight of the second liquid fraction.

49. A method of claim 44,

wherein said acidifying comprises contacting said fractionated lignocellulosic biomass with a gaseous compound that forms acid *in situ*.

50. A method of claim 49,

wherein said gaseous compound is SO_2, CO_2, NO_2, HX wherein X is Cl, Br, F, or I, or a combination thereof.

51. A method of claim 44,

wherein said acidifying comprises contacting said fractionated lignocellulosic biomass with a solid acid catalyst.

52. A method of claim 51,

wherein said solid acid catalyst is a zeolite, an anionic exchange resin, or a combination thereof.

53. A method of claim 44,

wherein said method is continuous.

54. A method of claim 44,

wherein said second level of said soluble C_5 saccharides is greater than said first level of said soluble C_5 saccharides.

55. A method of claim 44,

wherein said soluble C_5 saccharides in second liquid fraction have an average molecular weight that is lower than the average molecular weight of said soluble C_5 saccharides in said first liquid fraction.

56. A method of claim 44,

wherein said C_5 oligosaccharides in said first liquid fraction have about 2 mer units to about 25 mer units; and

wherein said C_5 oligosaccharides in said second liquid fraction have about 2 mer units to about 15 mer units.

57. A method of claim 44,

wherein said fractionated lignocellulosic biomass is formed by contacting said lignocellulosic biomass with a first reaction fluid comprising hot compressed water and, optionally, carbon dioxide;

wherein said first reaction fluid further comprises acid, when said lignocellulosic biomass comprises softwood;

wherein said first reaction fluid is at a temperature of at least 100°C under a pressure sufficient to maintain said first reaction fluid in liquid form.

58. A method of claim 44,

wherein said first temperature and said second temperature are substantially the same.

59. A method of claim 58,

wherein said first temperature and said second temperature are both no greater than about 240°C.

60. A method of claim 44,

wherein said second temperature is less than said first temperature.

61. A method of claim 60,

wherein said first temperature and said second temperature are both no greater than about 240°C.

62. A method of claim 44, further comprising:

reducing said first pressure of said first liquid fraction prior to separating said solid fraction from said second liquid fraction.

63. A method of claim 44,

wherein said hydrolyzing said second liquid fraction to form C_5 saccharides comprises contacting said C_5 oligosaccharides with dilute acid for a time sufficient to hydrolyze said C_5 oligosaccharides to C_5 oligosaccharides having fewer mer units, xylose, arabinose, lyxose, ribose, and mixtures thereof.

64. A method of claim 44,

wherein said hydrolyzing said second liquid fraction to form C_5 saccharides comprises enzymatic hydrolysis.

CLAIMS

What is claimed is:

1. A method of producing soluble C_5 saccharides from lignocellulosic biomass, comprising:

providing a fractionated lignocellulosic biomass comprising:

a solid fraction comprising:

optionally, insoluble C_5 oligosaccharide;

cellulose; and

lignin; and

a first liquid fraction at a first temperature and a first pressure comprising:

soluble C_5 saccharides selected from the group consisting of C_5 oligosaccharides, xylose, arabinose, lyxose, ribose, and mixtures thereof;

wherein said soluble C_5 saccharides are present at a first level;

optionally, separating said solid fraction from said first liquid fraction at a second pressure;

contacting said first liquid fraction with a solid acid catalyst to form a second liquid fraction comprising said soluble C_5 saccharides at a second level and at a second temperature;

wherein said second level is greater than said first level; and

wherein said second temperature is less than said first temperature;

optionally, hydrolyzing said second liquid fraction to form C_5 saccharides selected from the group consisting of C_5 oligosaccharides having fewer mer units, xylose, arabinose, lyxose, ribose, and mixtures thereof;

wherein said fractionated lignocellulosic biomass is formed by contacting said lignocellulosic biomass with a first reaction fluid comprising hot compressed water and, optionally, carbon dioxide;

wherein said first reaction fluid further comprises acid, when said lignocellulosic biomass comprises softwood;

wherein said first reaction fluid is at a temperature of at least 100ºC under a pressure sufficient to maintain said first reaction fluid in liquid form.

2. The method of claim 1,

wherein said solid acid catalyst is a zeolite, a cation exchange resin, or a combination thereof.

3. The method of claim 2,

wherein said solid acid catalyst is a zeolite.

4. The method of claim 2,

wherein said solid acid catalyst is a cation exchange resin.

5. The method of claim 1,

wherein said method is continuous.

6 The method of claim 1,

wherein said soluble C_5 saccharides in said second liquid fraction have an average molecular weight that is lower than the average molecular weight of said soluble C_5 saccharides in said first liquid fraction.

7. The method of claim 1,

wherein said C_5 oligosaccharides in said first liquid fraction have about 2 mer units to about 25 mer units; and

wherein said C_5 oligosaccharides in said second liquid fraction have about 2 mer units to about 15 mer units.

8. The method of claim 1

wherein said first temperature and said second temperature are both no greater than about 240ºC.

9. The method of claim 1,

wherein said separating is performed; and

wherein said first pressure is reduced prior to said separating.

10. The method of claim 1,

wherein said hydrolyzing said second liquid fraction to form C_5 saccharides is performed and comprises contacting said C_5 oligosaccharides with dilute acid for a time sufficient to hydrolyze said C_5 oligosaccharides to C_5 oligosaccharides having fewer mer units, xylose, arabinose, lyxose, ribose, and mixtures thereof.

11. The method of claim 10,

wherein said dilute acid is sulfuric acid.

12. The method of claim 11,

wherein said sulfuric acid is present at a level of about 0.05 wt.% to about 2 wt.%, based on the total weight of the second liquid fraction.

13. The method of claim 1,

wherein said hydrolyzing said second liquid fraction to form C_5 saccharides is performed and comprises enzymatic hydrolysis.

14. The method of claim 1,

wherein the yield of said xylose is at least 68% of theoretical yield.

15. The method of claim 1,

wherein the yield of said soluble C_5 saccharides is at least 68% of theoretical yield.

16. The method of claim 1, further comprising:

producing at least one of acetic acid and furfural from said soluble C_5 saccharides.

17. The method of claim 16,

wherein furfural is produced from said soluble C_5 saccharides by dehydration with an inorganic acid.

18. The method of claim 17,

wherein said dehydration is performed at a temperature of about 240 °C to about 300 °C.

19. The method of claim 17,

wherein said inorganic acid is sulfuric acid.

20. The method of claim 1,

wherein said first pressure and said second pressure are substantially the same.

21. The method of claim 1,

wherein said method is carried out in a digester.

22. The method of claim 21,

wherein said digester is a vertical digester.

23. The method of claim 1,

wherein said method is carried out in a digester system comprising a digester and a steam explosion unit.

24. The method of claim 1,

wherein said contacting said first liquid fraction with a solid acid catalyst further comprises adding an aqueous acid.

25. The method of claim 24,

wherein said aqueous acid is selected from the group consisting of sulfuric acid, phosphoric acid, nitric acid, and hydrochloric acid.

26. The method of claim 24,

wherein said aqueous acid is sulfuric acid.

27. The method of claim 24,

wherein said aqueous acid is present in an amount from about 0.07 wt.% to about 2 wt.%.

28. The method of claim 24,

wherein said aqueous acid is present in an amount of less than about 1 wt.%.

29. The method of claim 24,

wherein said aqueous acid is present in an amount of less than about 0.5 wt.%.

30. The method of claim 1,

wherein said contacting said first liquid fraction with a solid acid catalyst further comprises adding a gaseous compound that forms acid *in situ*.

31. The method of claim 30,

wherein said gaseous compound is selected from the group consisting of SO2, CO_2, NO_2, HX, or a combination thereof; and

wherein X is Cl, Br, F, or I.

32. The method of claim 30,

wherein said gaseous compound is SO_2.

33. The method of claim 30,

wherein said gaseous compound is CO_2.

34. The method of claim 30,

wherein said gaseous compound is present in an amount of about 0.07 wt.% to about 2 wt.%.

35. The method of claim 30,

wherein said gaseous compound is present in an amount of less than about 1 wt.%.

36. The method of claim 30,

wherein said gaseous compound is present in an amount of less than about 0.5 wt.%.

37. The method of claim 1,

wherein said method is performed as a batch process.

38. The method of claim 1,

wherein said method is performed as a semi-batch process.

39. The method of claim 1,

wherein said hydrolyzing said second liquid fraction to form C_5 saccharides is performed and comprises addition of an acid selected from the group consisting of sulfonic acid, phosphoric acid, phosphonic acid, nitric acid, nitrous acid, hydrochloric acid, hydrobromic acid, hydroiodic acid, acetic acid, formic acid, benzoic acid, salicylic acid, oxalic acid, and adipic acid.

40. The method of claim 39,

wherein said acid is phosphoric acid.

41. The method of claim 39,

wherein said acid is nitric acid.

42. The method of claim 39,

wherein said acid is acetic acid.

43. The method of claim 39,

wherein said acid is oxalic acid.

44. The method of claim 1,

wherein said hydrolyzing said second liquid fraction to form C_5 saccharides is performed.

45. The method of claim 44,

further comprising separating said C_5 oligosaccharides having fewer mer units using membrane separation.

46. The method of claim 44,

further comprising separating said C_5 oligosaccharides having fewer mer units using nanofiltration.

47. The method of claim 1,

further comprising fermenting said soluble C_5 saccharides in said second liquid fraction to produce ethanol, butanol, or mixtures thereof.

48. The method of claim 47,

wherein said fermenting produces ethanol.

49. The method of claim 47,

wherein said fermenting produces butanol.

50. The method of claim 1,

further comprising separating C_5 oligosaccharides present in said second liquid fraction using membrane separation.

51. The method of claim 1,

further comprising separating C_5 oligosaccharides present in said second liquid fraction using nanofiltration.

52. The method of claim 1,

wherein said first reaction fluid further comprises carbon dioxide.

53. The method of claim 1,

wherein said lignocellulosic biomass comprises hardwood.

54. The method of claim 53,

wherein said fractionated lignocellulosic biomass is prepared without the addition of acid.

55. The method of claim 1,

wherein said lignocellulosic biomass comprises softwood.

56. The method of claim 55,

wherein said acid in said first reaction fluid comprises an inorganic acid.

57. The method of claim 56,

wherein said inorganic acid is selected from the group consisting of sulfuric acid, phosphoric acid, nitric acid, and hydrochloric acid.

58. The method of claim 56,

wherein said inorganic acid is sulfuric acid.

59. The method of claim 56,

wherein said inorganic acid is phosphoric acid.

60. The method of claim 55,

wherein said acid in said first reaction fluid comprises an organic acid.

61. The method of claim 60,

wherein said organic acid is selected from the group consisting of acetic acid, formic acid, benzoic acid, salicylic acid, oxalic acid, and an amino acid.

62. The method of claim 60,

wherein said organic acid is oxalic acid.

63 The method of claim 60,

wherein said organic acid is acetic acid.

64. The method of claim 60,

wherein said organic acid is formic acid.