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(54) Titre : **PIECE D'HABILLEMENT TECHNIQUE EN SOIE ET PROCEDES DE PREPARATION**  
 (54) Title: **SILK PERFORMANCE APPAREL AND PRODUCTS AND METHODS OF PREPARING THE SAME**

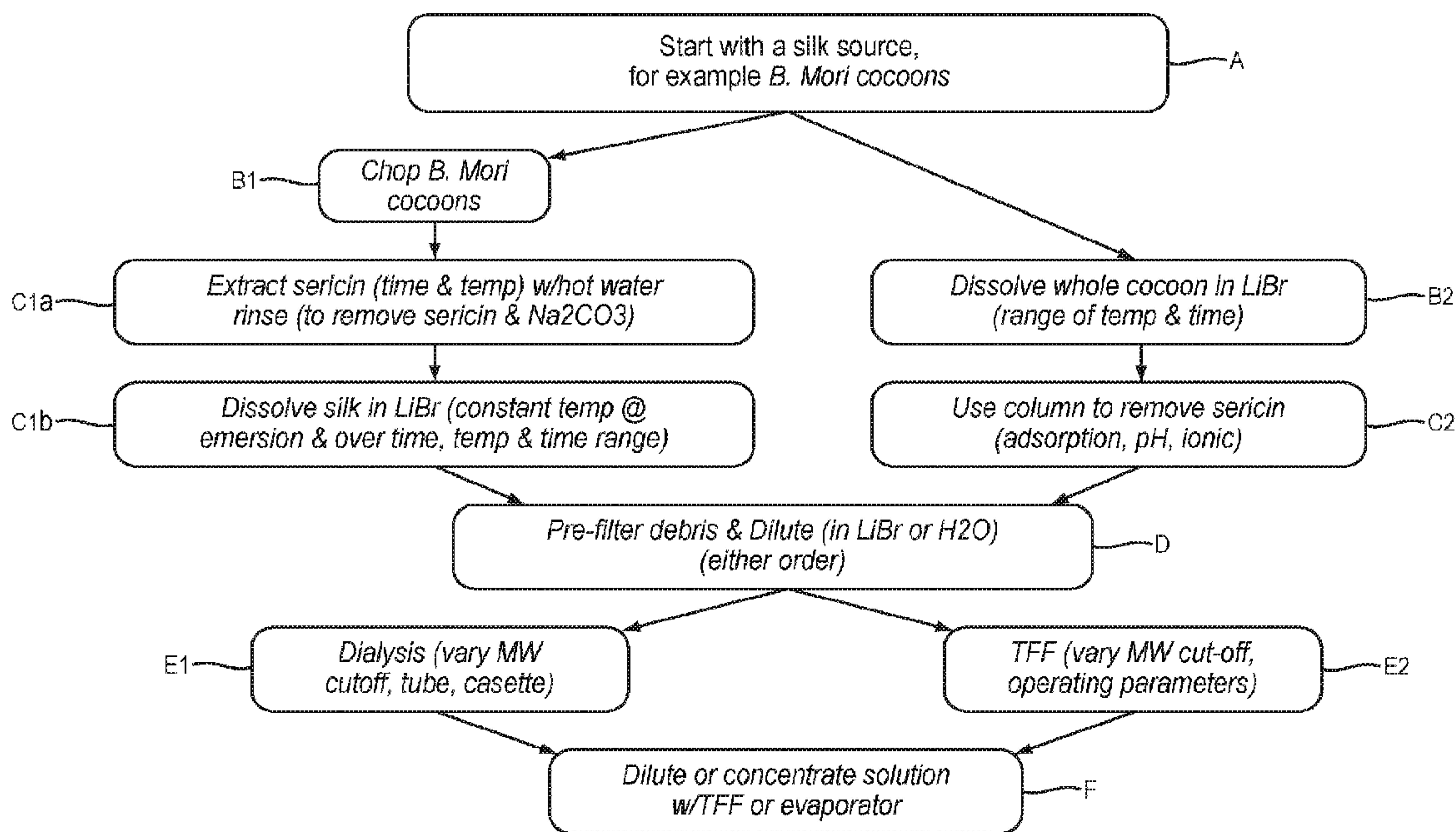


FIG. 1

(57) **Abrégé/Abstract:**

Silk infused performance apparel and methods of preparing the same are disclosed herein. In some embodiments, silk performance apparel includes textiles, fabrics, consumer products, leather, and other materials that are coated with aqueous solutions of pure silk fibroin-based protein fragments. In some embodiments, coated apparel products, textiles, and upholstery, as well as other materials, exhibit surprisingly improved moisture management properties, resistance to microbial growth, increased abrasion resistance, and flame resistance.

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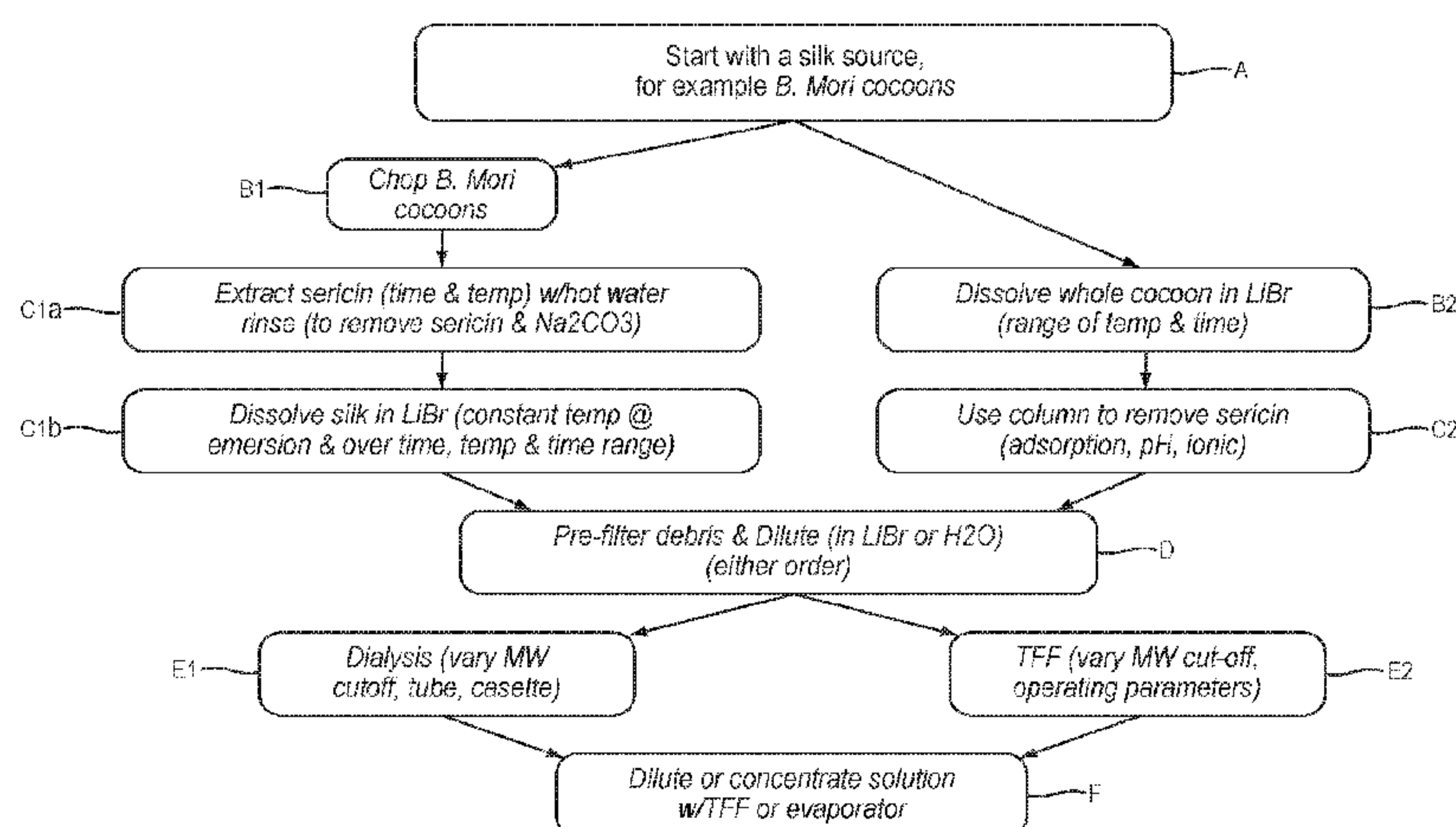


FIG. 1

(57) Abstract: Silk infused performance apparel and methods of preparing the same are disclosed herein. In some embodiments, silk performance apparel includes textiles, fabrics, consumer products, leather, and other materials that are coated with aqueous solutions of pure silk fibroin-based protein fragments. In some embodiments, coated apparel products, textiles, and upholstery, as well as other materials, exhibit surprisingly improved moisture management properties, resistance to microbial growth, increased abrasion resistance, and flame resistance.

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## CLAIMS

We claim:

1. A method of coating a material with silk fibroin that comprises silk-based proteins or fragments thereof to provide a silk fibroin coated material, wherein the silk fibroin coated upon the silk fibroin coated material is heat resistant to a selected temperature, the method comprising:
  - (a) preparing a silk fibroin solution comprising a concentration of one or more of low molecular weight silk fibroin, medium molecular weight silk fibroin, and high molecular weight silk fibroin at less than about 1% by volume (v/v);
  - (b) coating a surface of the material with the silk fibroin solution; and
  - (c) drying the surface of the material that has been coated with the silk fibroin solution to provide the silk fibroin coated material, wherein drying the surface of the material comprises heating the surface of the material without substantially modifying silk fibroin coating performance.
2. The method of claim 1, wherein the silk fibroin solution comprises low molecular weight silk fibroin.
3. The method of any one of the preceding claims, wherein the silk fibroin solution comprises medium molecular weight silk fibroin.
4. The method of any one of the preceding claims, wherein the step of preparing the silk fibroin solution comprises adding a chemical fabric softener to the silk fibroin solution.

5. The method of any one of the preceding claims, wherein the silk fibroin solution comprises a Bronsted acid.

6. The method of any one of the preceding claims, wherein the silk fibroin solution  
5 comprises one or more of citric acid and acetic acid.

7. The method of any one of the preceding claims, wherein the step of coating a surface of the material comprises one or more of a roller application process, a saturation and removal process, and a topical application process.

10

8. The method of any one of the preceding claims, wherein the step of coating a surface of the material comprises one or more of a bath coating process, a kiss rolling process, spray coating, and a two-sided rolling process.

15 9. The method of any one of the preceding claims, wherein the step of coating a surface of the material comprises coating one surface of the material.

10. The method of any one of the preceding claims, wherein the step of coating a surface of the material comprises coating two surfaces of the material.

20

11. The method of any one of the preceding claims, further comprising the step of dyeing the surface of the material prior to coating a surface of the material with the silk fibroin solution.

25 12. The method of any one of claims 1-10, further comprising the step of dyeing the surface of the material after coating a surface of the material with the silk fibroin solution.

13. The method of any one of the preceding claims wherein the material comprises one or more of a woven material, a non-woven material, a knit material, and a crochet material.

5

14. The method of any one of the preceding claims, wherein the material comprises fabric, thread, yarn, or a combination thereof.

10 15. The method of any one of the preceding claims, wherein the material comprises one or more of polyester, polyamide, polyaramid, polytetrafluorethylene, polyethylene, polypropylene, polyurethane, silicone, mixtures of polyurethane and polyethyleneglycol, ultrahigh molecular weight polyethylene, high-performance polyethylene, nylon, and LYCRA.

15 16. A silk fibroin coated material manufactured by the method of any one of claims 1-15.

17. A method of coating a textile with a silk fibroin solution that includes silk-based proteins or fragments thereof to provide a silk fibroin coated article, wherein the silk  
20 fibroin coated upon the silk fibroin coated article is heat resistant to a selected temperature, the method comprising the steps of:

- (a) preparing the silk fibroin solution with one or more of low molecular weight silk fibroin, medium molecular weight silk fibroin, and high molecular weight silk fibroin;
- 25 (b) acidically adjusting the pH of the silk fibroin solution with an acidic agent;
- (c) coating a surface of the textile with the silk fibroin solution; and



- (d) drying the surface of the textile that has been coated with the silk fibroin solution to provide the silk fibroin coated article, wherein drying the surface of the textile comprises heating the surface of the textile without substantially modifying silk fibroin coating performance.

5

18. The method of claim 17, wherein the silk fibroin solution comprises low molecular weight silk fibroin.

19. The method of any one of claims 17 and 18, wherein the silk fibroin solution  
10 comprises medium molecular weight silk fibroin.

20. The method of any one of claims 17-19, wherein the step of preparing the silk fibroin solution comprises adding a chemical fabric softener to the silk fibroin solution.

15 21. The method of any one of claims 17-20, wherein the acidic agent comprises a Bronsted acid.

22. The method of any one of claims 17-21, wherein the acidic agent comprises one or more of citric acid and acetic acid.

20

23. The method of any one of claims 17-22, wherein the step of coating a surface of the textile comprises one or more of a roller application process, a saturation and removal process, and a topical application process.

25 24. The method of any one of claims 17-23, wherein the step of coating a surface of the textile comprises one or more of a bath coating process, a kiss rolling process, spray coating, and a two-sided rolling process.

25. The method of any one of claims 17-24, wherein the step of coating a surface of the textile comprises coating one surface of the textile.

5 26. The method of any one of claims 17-25, wherein the step of coating a surface of the textile comprises coating two surfaces of the textile.

27. The method of any one of claims 17-26, further comprising the step of dyeing the surface of the textile prior to coating a surface of the textile with the silk fibroin solution.

10

28. The method of any one of claims 17-26, further comprising the step of dyeing the surface of the textile after coating a surface of the textile with the silk fibroin solution.

15

29. The method of any one of claims 17-28, wherein the textile comprises one or more of a woven material, a non-woven material, a knit material, and a crochet material.

30. The method of any one of claims 17-29, wherein the textile comprises fabric, thread, yarn, or a combination thereof.

20

31. The method of any one of claims 17-30, wherein the textile comprises one or more of polyester, polyamide, polyaramid, polytetrafluorethylene, polyethylene, polypropylene, polyurethane, silicone, mixtures of polyurethane and polyethyleneglycol, ultrahigh molecular weight polyethylene, high-performance polyethylene, nylon, and LYCRA.

25

32. A silk fibroin coated article manufactured by the method of any one of claims 17-31.

33. A method of manufacturing a silk fibroin coated textile having selected fabric properties, the method comprising the steps of:

5 (a) admixing silk-based proteins or fragments thereof with one or more chemical agents to provide a coating solution, wherein the one or more chemical agents are selected to modify one or more of a first selected property and second selected property of the silk fibroin coated textile;

10 (b) providing the coating solution to a textile to be coated with one or more of a bath coating process, a kiss rolling process, spray coating, and a two-sided rolling process;

(c) removing excess coating solution from the silk fibroin coated textile; and

(d) heating the silk fibroin coated textile to modify a third selected property of the silk fibroin coated textile.

15 34. The method of claim 33, wherein the first selected property comprises one or more of an antimicrobial property, a water repellent property, an oil repellent property, a flame retardant property, a coloring property, a fabric softening property, a stain repellent property, a pH adjusting property, an anticrocking property, an antipilling property, and an antifelting property.

20 35. The method of claim 33 or 34, wherein the second selected property comprises one or more of wetting time, absorption rate, spreading speed, accumulative one-way transport, and overall moisture management capability.

25 36. The method of any one of claims 33-35, wherein the third selected property comprises one or more of fabric hand, fabric stretch, and drapability.



37. The method of any one of claims 33-36, wherein the coating solution comprises low molecular weight silk fibroin.

38. The method of any one of claims 33-37, wherein the coating solution comprises  
5 medium molecular weight silk fibroin.

39. The method of any one of claims 33-38, wherein the chemical agent comprises a chemical fabric softener.

10 40. The method of any one of claims 33-39, wherein the chemical agent comprises an acidic agent.

41. The method of any one of claims 33-40, wherein the textile comprises one or more of a woven material, a non-woven material, a knit material, and a crochet material.

15

42. The method of any one of claims 33-41, wherein the textile comprises fabric, thread, yarn, or a combination thereof.

43. The method of any one of claims 33-42, wherein the textile comprises one or  
20 more of polyester, polyamide, polyaramid, polytetrafluorethylene, polyethylene, polypropylene, polyurethane, silicone, mixtures of polyurethane and polyethyleneglycol, ultrahigh molecular weight polyethylene, high-performance polyethylene, nylon, and LYCRA.

25 44. A silk fibroin coated textile manufactured by the method of any one of claims 33-43.

45. An article comprising a fabric, wherein the fabric is coated with a coating, wherein the coating comprises silk fibroin, and wherein the silk fibroin, before coating, is a solution comprising silk fibroin at a concentration of 0.001% to 1% by weight.

5 46. The article of Claim 45, wherein the solution is an aqueous solution.

47. The article of Claim 45, wherein the solution is an organic solution.

48. The article of any one of Claims 45 to 47, wherein the fabric is selected from the  
10 group consisting of cotton, alpaca fleece, alpaca wool, lama fleece, lama wool, cotton, cashmere, sheep fleece, sheep wool, polyester, nylon, polyester-polyurethane copolymer, and combinations thereof.

49. The article of any one of Claims 45 to 47, wherein the fabric comprises a  
15 polyester-polyurethane copolymer.

50. The article of Claim 45 or 46, wherein a portion of the silk fibroin is partially dissolved in a surface of the polyester-polyurethane copolymer.

20 51. The article of Claim 45 or 46, wherein the silk fibroin is crosslinked with the polyester-polyurethane copolymer.

52. The article of any one of Claims 45 to 51, wherein the silk fibroin coating contains no sites for bacterial or fungal adhesion.

53. The article of any one of Claims 45 to 51, wherein the silk fibroin coating  
5 comprises low molecular weight silk fibroin.

54. The article of any one of Claims 45 to 51, wherein the silk fibroin coating comprises medium molecular weight silk fibroin.

10 55. A method of coating a material with silk-based proteins or fragments thereof to provide a coated material, the method comprising:

- 15
- (a) preparing a coating solution comprising a concentration of silk-based proteins or fragments thereof that comprises one or more of low molecular weight silk fibroin, medium molecular weight silk fibroin, and high molecular weight silk fibroin at less than about 1% by volume (v/v);
  - (b) adding silicone to the coating solution; and
  - (c) coating a surface of the material with the coating solution to provide a coated material;

20 wherein the coated material comprises improved water transport as compared to a material coated with silicone without silk-based proteins or fragments thereof.

56. A method of coating a material with silk-based proteins or fragments thereof to provide a coated material, the method comprising:

- 25
- (a) preparing a coating solution comprising a concentration of silk-based proteins or fragments thereof that comprises one or more of low molecular



weight silk fibroin, medium molecular weight silk fibroin, and high molecular weight silk fibroin at less than about 1% by volume (v/v);

- (b) coating a surface of the material with the coating solution; and
- (c) coating the surface of the material with silicone to provide a coated material;

wherein the coated material comprises improved water transport as compared to a material coated with silicone without silk-based proteins or fragments thereof.

57. A method of coating a material with silk-based proteins or fragments thereof to provide a coated material, the method comprising:

- (a) coating a surface of the material with silicone;
- (b) preparing a coating solution comprising a concentration of silk-based proteins or fragments thereof that comprises one or more of low molecular weight silk fibroin, medium molecular weight silk fibroin, and high molecular weight silk fibroin at less than about 1% by volume (v/v); and
- (c) coating the surface of the material with the coating solution to provide a coated material;

wherein the coated material comprises improved water transport as compared to a material coated with silicone without silk-based proteins or fragments thereof.

58. The method of any one of claims 55-57, wherein the coating solution comprises low molecular weight silk fibroin.

59. The method of any one of claims 55-58, wherein the coating solution comprises medium molecular weight silk fibroin.

60. The method of any one of claims 55-59, wherein the step of preparing the coating solution comprises adding a chemical fabric softener to the coating solution.

61. The method of any one of claims 55-60, wherein the coating solution comprises a  
5 comprises a Bronsted acid.

62. The method of any one of claims 55-61, wherein the coating solution comprises one or more of citric acid and acetic acid.

10 63. The method of any one of claims 55-62, further comprising the step of dyeing the surface of the material prior to coating the surface of the material with the coating solution.

15 64. The method of any one of claims 55-63, further comprising the step of dyeing the surface of the material after coating the surface of the material with the coating solution.

65. The method of any one of claims 55-64, wherein the material comprises one or more of a woven material, a non-woven material, a knit material, and a crochet material.

20 66. The method of any one of claims 55-65, wherein the material comprises fabric, thread, yarn, or a combination thereof.

25 67. The method of any one of claims 55-66, wherein the material comprises one or more of polyester, polyamide, polyaramid, polytetrafluorethylene, polyethylene, polypropylene, polyurethane, silicone, mixtures of polyurethane and polyethyleneglycol, ultrahigh molecular weight polyethylene, high-performance polyethylene, nylon, and LYCRA.

68. An antimicrobial-coated material prepared according to the method of any one of claims 55-67.

5 69. The antimicrobial-coated material of claim 68, wherein the antimicrobial coated material comprises one or more of an antibacterial coating and an antifungal coating.

70. A method of coating a material with silk-based proteins or fragments thereof to provide a silk protein coated material, wherein the silk protein coated upon the silk protein coated material comprises one or more coating properties, the method  
10 comprising:

- 15 (a) preparing a coating solution comprising a concentration of one or more of low molecular weight silk fibroin, medium molecular weight silk fibroin, and high molecular weight silk fibroin at less than about 1% by volume (v/v);
- (b) coating a surface of the material with the coating solution; and
- (c) drying the surface of the material that has been coated with the coating solution to provide the silk protein coated material, wherein the one or more coating properties subsist after the step of drying the surface of the  
20 silk protein coated material.

71. The method of claim 70, wherein the coating solution comprises low molecular weight silk fibroin.

25 72. The method of any one of claims 70 and 71, wherein the coating solution comprises medium molecular weight silk fibroin.



73. The method of any one of claims 70-72, wherein the step of preparing the coating solution comprises adding a chemical agent to the coating solution.

74. The method of claim 73, wherein the chemical agent is selected from the group consisting of an antimicrobial agent, a softener, a water repellent agent, an oil repellent agent, a dye, a flame retardant, a fabric softener, a pH adjusting agent, an anticrocking agent, an antipilling agent, an antifelting agent, and combinations thereof.

75. The method of claim 73, wherein the chemical agent is selected from the group consisting of a chemical fabric softener, a dye, an acidic agent, and combinations thereof.

76. The method of claim 75, wherein the acidic agent comprises one or more of citric acid and acetic acid.

77. The method of any one of claims 70-76, wherein the step of drying the surface of the material comprises heating the material.

78. The method of any one of claims 70-77, wherein the one or more coating properties are selected from the group consisting of an antimicrobial property, a water repellent property, an oil repellent property, a flame retardant property, a coloring property, a fabric softening property, a stain repellent property, a pH adjusting property, an anticrocking property, an antipilling property, and an antifelting property.

79. The method of any one of claims 70-77, wherein the one or more coating properties are selected from the group consisting of wetting time, absorption rate, spreading speed, accumulative one-way transport, and overall moisture management capability.

80. The method of any one of claims 70-79, wherein the material comprises one or more of polyester, polyamide, polyaramid, polytetrafluorethylene, polyethylene, polypropylene, polyurethane, silicone, mixtures of polyurethane and polyethyleneglycol, ultrahigh molecular weight polyethylene, high-performance polyethylene, nylon, and LYCRA.
81. The method of any one of claims 70-80, wherein the silk protein coated material is a biocompatible silk protein coated material.
82. A silk protein coated article manufactured by the method of any one of claims 70-81.
83. A method of manufacturing a coated textile comprising a silk-based coating having selected fabric properties, the method comprising the steps of:
- (a) admixing silk-based proteins or fragments thereof with one or more chemical agents to provide a coating solution, wherein the one or more chemical agents are selected to modify one or more of a first selected property and second selected property of the coated textile;
  - (b) providing the coating solution to a textile to be coated with one or more of a bath coating process, a kiss rolling process, spray coating, and a two-sided rolling process; and
  - (c) removing excess coating solution from the coated textile.
84. The method of claim 83, wherein the first selected property comprises one or more of an antimicrobial property, a water repellent property, an oil repellent property, a flame retardant property, a coloring property, a fabric softening property, a stain repellent

property, a pH adjusting property, an anticrocking property, an antipilling property, and an antifelting property.

85. The method of claim 83 or 84, wherein the second selected property comprises  
5 one or more of wetting time, absorption rate, spreading speed, accumulative one-way transport, and overall moisture management capability.

86. The method of any one of claims 83-85, further comprising the step of heating the  
10 coated textile to modify a third selected property of the coated textile, wherein the third selected property comprises one or more of fabric hand, fabric stretch, and drapability.

87. The method of any one of claims 83-86, wherein the coating solution comprises low molecular weight silk fibroin.

15 88. The method of any one of claims 83-87, wherein the coating solution comprises medium molecular weight silk fibroin.

89. The method of any one of claims 83-88, wherein the chemical agent comprises a chemical fabric softener.

20

90. The method of any one of claims 83-89, wherein the chemical agent comprises an acidic agent.

25 91. The method of any one of claims 83-90, wherein the textile comprises one or more of polyester, polyamide, polyaramid, polytetrafluorethylene, polyethylene, polypropylene, polyurethane, silicone, mixtures of polyurethane and polyethyleneglycol,



ultrahigh molecular weight polyethylene, high-performance polyethylene, nylon, and LYCRA.

92. A coated textile manufactured by the method of any one of claims 83-91.

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93. A coated polymer article comprising:

(a) a polymeric substrate comprising one or more of polyester, polyamide, polyaramid, polytetrafluorethylene, polyethylene, polypropylene, polyurethane, silicone, mixtures of polyurethane and polyethyleneglycol, ultrahigh molecular weight  
10 polyethylene, high-performance polyethylene, nylon, and LYCRA; and

(b) a silk-based coating infused to a surface of the polymeric substrate such that the silk-based coating is co-dissolved in the polymeric substrate to a depth of at least about 1 nm, forming a dispersion.

15 94. The coated polymer article of claim 93, wherein the silk-based coating comprises silk-fibroin proteins or fragments thereof.

95. The coated polymer article of any one of claims 93 and 94, wherein the silk-based coating comprises a chemical agent.

20

96. The coated polymer article of any one of claims 94-95, wherein the silk based coating comprises one or more of an antimicrobial agent, a softener, a water repellent agent, an oil repellent agent, a dye, a flame retardant, a fabric softener, a pH adjusting agent, an anticrocking agent, an antipilling agent, and an antifelting agent.

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97. The coated polymer article of any one of claims 94-96, wherein the silk based coating comprises one or more of a chemical fabric softener, a dye, and an acidic agent.

98. The coated polymer article of any one of claims 94-97, wherein the polymeric substrate comprises one or more of a woven material, a non-woven material, a knit material, and a crochet material.

5 99. The coated polymer article of any one of claims 94-98, wherein the polymeric substrate comprises fabric, thread, yarn, or a combination thereof.

100. The coated polymer article of any one of claims 94-99, wherein the silk-based coating is a biocompatible silk-based coating.

10

101. The coated polymer article of any one of claims 94-100, wherein the dispersion is a molecular solid solution.

102. The coated polymer article of any one of claims 94-101, wherein the silk-based  
15 coating is co-dissolved in the polymeric substrate to a depth of at least about 10 nm.

103. The coated polymer article of any one of claims 94-102, wherein the silk-based coating is co-dissolved in the polymeric substrate to a depth of at least about 100 nm.

20 104. A method for preparing a coated polymer article comprising the steps of:

(a) providing a polymeric substrate comprising one or more of polyester, polyamide, polyaramid, polytetrafluorethylene, polyethylene, polypropylene, polyurethane, silicone, mixtures of polyurethane and polyethyleneglycol, ultrahigh molecular weight polyethylene, high-performance polyethylene, nylon, and LYCRA;

25 (b) applying a silk-based coating to the surface of the polymeric substance to create a coated surface;

(c) co-dissolving the silk-based protein at the coated surface with the polymeric substrate so that the silk-based protein is co-dissolved in the polymeric substrate to a depth of at least about 1 nm using a co-dissolution process.

5 105. The method of claim 104, wherein the silk-based coating comprises silk-fibroin proteins or fragments thereof.

106. The method of claim 104, wherein the co-dissolution process is a thermal process.

10 107. The method of claim 106, wherein the thermal process comprises the step of heating the coated polymeric substrate and silk-based protein to a temperature selected from the group consisting of 100 °C, 125 °C, 150 °C, 175 °C, 200 °C, 225 °C, and 250 °C, for a time period selected from the group consisting of 1 minute, 2 minutes, 5 minutes, 10 minutes, 15 minutes, 20 minutes, and 30 minutes.

15

106. The method of claim 104, wherein the thermal process comprises the step of heating the coated polymeric substrate and silk-based protein to a temperature above the glass transition temperature of the coated polymeric substrate or the glass transition temperature of the silk-based protein, for a time period selected from the group consisting  
20 of 1 minute, 2 minutes, 5 minutes, 10 minutes, 15 minutes, 20 minutes, and 30 minutes.

107. The method of claim 104, further comprising the step of treating the coated surface with an organic solvent so that the silk-based protein is co-dissolved in the polymeric substrate.

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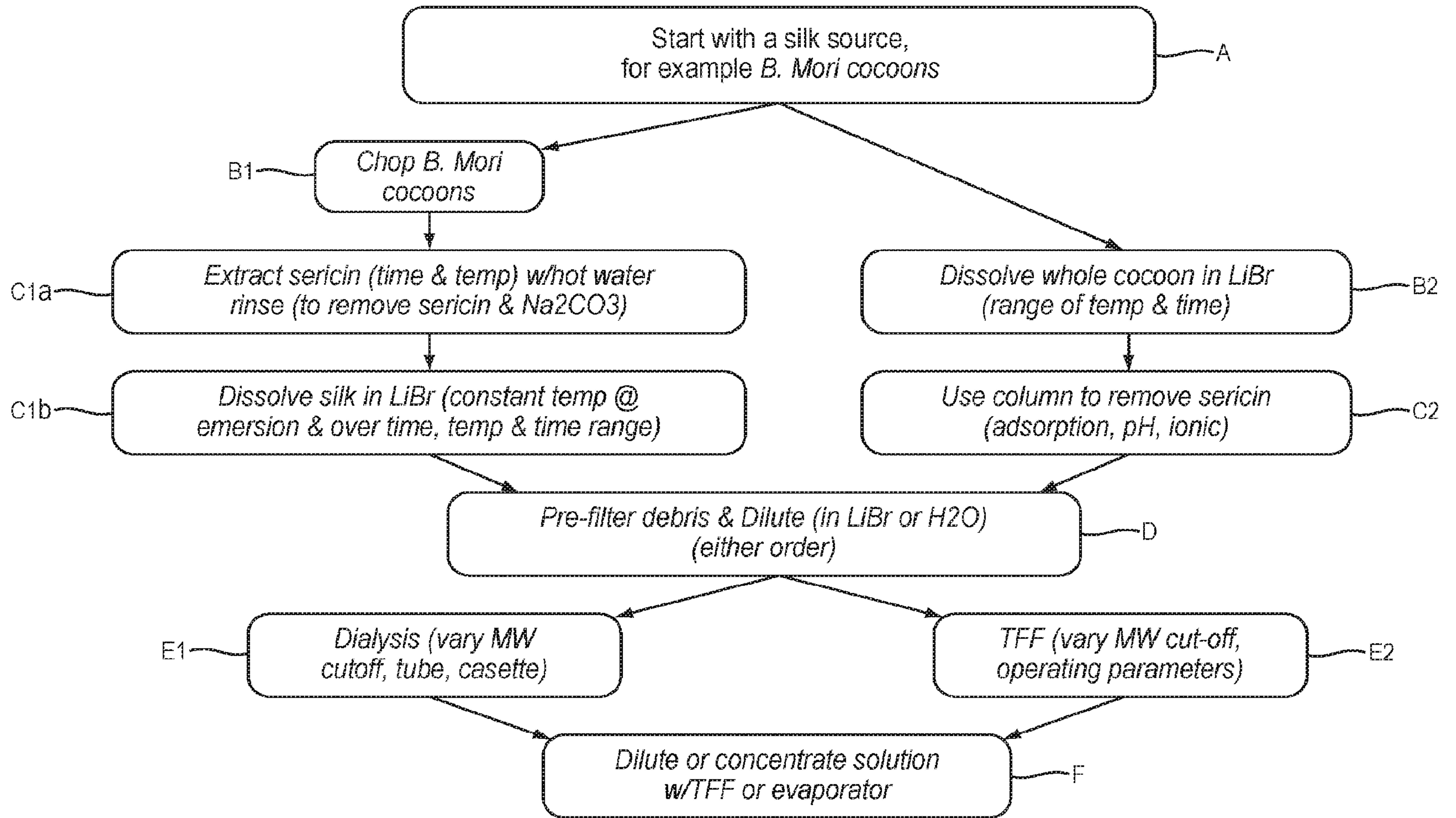


FIG. 1