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(71) Applicant: **TRUSTEES OF TUFTS COLLEGE** [US/US]; Ballou Hall, Medford, Massachusetts 02155 (US).

(72) Inventors: **OMENETTO, Fiorenzo G.**; Ballou Hall, Medford, Massachusetts 02155 (US). **LO PRESTI, Marco**; Ballou Hall, Medford, Massachusetts 02155 (US).

(74) Agent: **SCHLEICHER, James M.** et al.; GTC Law Group PC & Affiliates, One University Ave., Ste. 302B, Westwood, Massachusetts 02090 (US).

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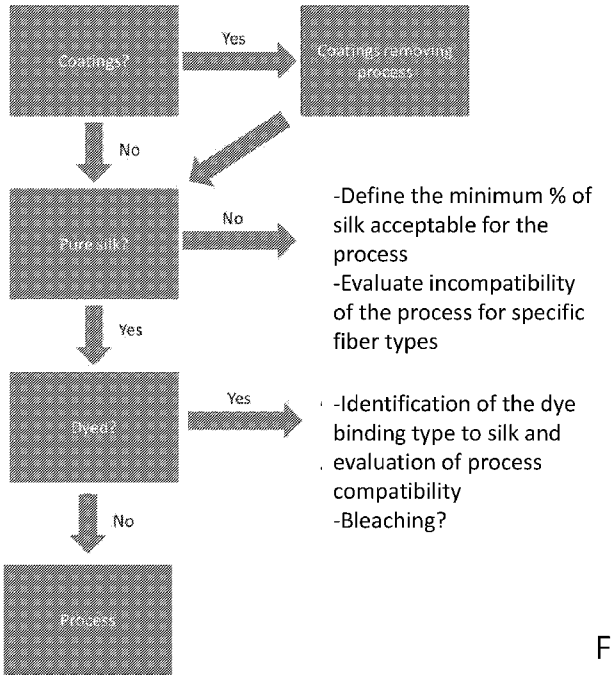


FIG. 1

(57) Abstract: Silk waste stream processing includes analyzing a finished textile to identify a dye-binding mode of a dye, the finished textile comprising silk and the dye, selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the identified dye-binding mode, and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

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SYSTEMS AND METHODS FOR SILK WASTE STREAM PROCESSING

CLAIM TO PRIORITY

[0001] This application relates to, incorporates by reference for all purposes, and claims priority to United States Application Serial Number 63/515,689, filed July 26, 2023.

BACKGROUND

[0002] Presently, the fashion and textile industry generate a staggering 1.23 billion tons of waste annually which is expected to reach 2.23 billion in 2025. This includes silk-based waste accounting for 11 million tons, much of which remains unrecycled. Silk textile waste-streams may include finished textiles/clothing that may be processed and/or colored/dyed.

[0003] A need exists for improved methods of recycling silk waste streams into useful compositions and articles.

SUMMARY

[0004] In some aspects, the techniques described herein relate to a method, including: analyzing a finished textile to identify a dye-binding mode of a dye, the finished textile including silk and the dye; selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the identified dye-binding mode; and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0005] In some aspects, the techniques described herein relate to a method, including: analyzing a finished textile spectroscopically to identify an aspect of a dye, the finished textile including silk and the dye; selecting one or more regeneration parameters of a silk fibroin regeneration process to be applied to the finished textile in response to the identified aspect of the dye; and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0006] In some aspects, the techniques described herein relate to a method, including: analyzing a finished textile to determine a proportion of silk in the finished textile; selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the determined proportion; and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0007] In some aspects, the techniques described herein relate to a method, including: analyzing a finished textile to determine a composition of the finished textile, the finished textile including silk; selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the determined composition; and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0008] In some aspects, the techniques described herein relate to a method, including: determining or estimating a molecular weight of a silk fibroin of silk in a finished textile; selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile further in

response to the determined or estimated molecular weight; and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0009] In some aspects, the techniques described herein relate to a method, including: imaging a finished textile to determine at least one of an age or a wear value of the finished textile, the finished textile including silk; selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the at least one determined age or determined wear value; and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0010] In some aspects, the techniques described herein relate to a method, including: determining at least one of an age or a wear value of a finished textile, the finished textile including silk, wherein determining includes at least one of imaging the finished textile, performing a microscopic analysis of the finished textile, or performing a mechanical analysis of the finished textile; selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the at least one determined age or determined wear value; and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0011] In some aspects, the techniques described herein relate to a method, including: identifying a presence of a coating of a finished textile, the finished textile including silk; selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the presence of the coating; and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0012] In some aspects, the techniques described herein relate to a method, including: identifying a presence or an absence of a coating of a finished textile, the finished textile including silk; sorting the finished textile in response to at least one of the presence or the absence of the coating; removing, in response to the presence, the coating from the finished textile; analyzing the finished textile to determine a proportion of silk in the finished textile; sorting the finished textile in response to the determined proportion; analyzing the finished textile to identify an aspect of a dye; sorting the finished textile in response to the identified aspect of the dye; selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to at least one of the determined proportion, the presence or the absence of the coating, or the aspect of the dye; and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0013] In some aspects, the techniques described herein relate to a method, including: identifying a presence or an absence of a coating of a finished textile, the finished textile including silk; removing, in response to the presence, the coating from the finished textile; analyzing the finished textile to determine a proportion of silk in the finished textile; analyzing the finished textile to identify an

aspect of a dye; selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to at least one of the determined proportion, the presence or the absence of the coating, or the aspect of the dye; and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0014] In some aspects, the techniques described herein relate to a composition including: a silk fibroin; and a regenerated silk fibroin made by a method of any one of the preceding claims.

[0015] In some aspects, the techniques described herein relate to a composition including: a silk fibroin; a regenerated silk fibroin made by a method of any one of the preceding claims; and a functionalizing agent.

[0016] In some aspects, a device may analyze a finished textile to identify a dye-binding mode of a dye, the finished textile comprising silk and the dye. A device may select one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the identified dye-binding mode. A device may regenerate a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0017] In some aspects, a textile waste stream processing system includes a spectroscopic imaging facility operationally coupled to a textile waste storage vessel, the spectroscopic imaging facility communicatively coupled to a data collector, the data collector structured to collect a plurality of detection values from the spectroscopic imaging facility upon imaging at least one waste textile in the textile waste storage vessel, a data storage structured to store a plurality of spectra patterns of textiles, the stored plurality of spectra patterns comprising a library of spectra patterns, a data acquisition circuit structured to interpret the plurality of detection values from the data collector, an expert system analysis circuit structured to analyze the plurality of detection values to determine if one of the plurality of detection values has a recognized spectra pattern corresponding to at least one of the stored plurality of spectra patterns from the library of spectra patterns, and a response circuit structured to provide an instruction in response to the one of the plurality of detection values having a stored spectra pattern.

[0018] These and other systems, methods, objects, features, and advantages of the present disclosure will be apparent to those skilled in the art from the following detailed description of the preferred embodiment and the drawings.

[0019] All documents mentioned herein are hereby incorporated in their entirety by reference. References to items in the singular should be understood to include items in the plural, and vice versa, unless explicitly stated otherwise or clear from the text. Grammatical conjunctions are intended to express any and all disjunctive and conjunctive combinations of conjoined clauses, sentences, words, and the like, unless otherwise stated or clear from the context.

BRIEF DESCRIPTION OF THE FIGURES

[0020] The disclosure and the following detailed description of certain embodiments thereof may be understood by reference to the following figures:

[0021] Fig. 1 depicts an example process for regenerating a silk fibroin solution according to an embodiment of the present disclosure.

[0022] Fig. 2 depicts a macroscopic image of a silk mono-material embroidery. It consists of silk fabric for the base, silk thread for embroidering, and silk sequins as embellishments.

[0023] Fig. 3 depicts fabrication processes of silk sequins from textile waste. Fig. 3A. Silk textile remnants are recycled into silk blend solution. This solution is dried into films that are laser cut into sequins for embellishments; Fig. 3B. Silk textile remnants are coated with silk fibroin solution. Once dried, these rigid textile sheets are laser cut into sequins for embellishments.

[0024] Fig.4 depicts an example fabrication and recycling process of silk mono-material embroidery.

[0025] Fig. 5 depicts the textile waste stream processing system.

DETAILED DESCRIPTION

[0026] Before the present disclosure is described in further detail, it is to be understood that the disclosure is not limited to the particular embodiments described. It is also understood that the terminology used herein is for the purpose of describing particular embodiments only, and is not intended to be limiting. The scope of the present disclosure will be limited only by the claims. As used herein, the singular forms "a", "an", and "the" include plural embodiments unless the context clearly dictates otherwise.

[0027] In this application, unless otherwise clear from context, (i) the term “a” may be understood to mean “at least one”; (ii) the term “or” may be understood to mean “and/or”; (iii) the terms “comprising” and “including” may be understood to encompass itemized components or steps whether presented by themselves or together with one or more additional components or steps; and (iv) the terms “about” and “approximately” are used as equivalents and may be understood to permit standard variation as would be understood by those of ordinary skill in the art; and (v) where ranges are provided, endpoints are included.

[0028] ***Approximately:*** as used herein, the term “approximately” or “about,” as applied to one or more values of interest, refers to a value that is similar to a stated reference value. In certain embodiments, the term “approximately” or “about” refers to a range of values that fall within 25%, 20%, 19%, 18%, 17%, 16%, 15%, 14%, 13%, 12%, 11%, 10%, 9%, 8%, 7%, 6%, 5%, 4%, 3%, 2%, 1%, or less in either direction (greater than or less than) of the stated reference value unless otherwise stated or otherwise evident from the context (except where such number would exceed 100% of a possible value).

[0029] ***Composition:*** as used herein, may be used to refer to a discrete physical entity that comprises one or more specified components. In general, unless otherwise specified, a composition may be of any form – e.g., gas, gel, liquid, solid, etc. In some embodiments, "composition" may refer to a combination of two or more entities for use in a single embodiment or as part of the same article. It is not required in all embodiments that the combination of entities result in physical admixture, that is, combination as separate co-entities of each of the components of the composition is possible; however many practitioners in the field may find it advantageous to prepare a composition that is an admixture of two or more of the ingredients in a pharmaceutically acceptable carrier, diluent, or excipient, making it possible to administer the component ingredients of the combination at the same time.

[0030] ***Improve, increase,* or *reduce:*** as used herein or grammatical equivalents thereof, indicate values that are relative to a baseline measurement, such as a measurement in a similar composition made according to previously known methods.

[0031] ***Mono-material embroidery:*** as used herein, the term "mono-material embroidery" refers to an embroidery where a single material makes up at least 50%, at least 75%, at least 90%, at least 95%, or substantially 100% of the underlying fabric, the thread/yarn that is used to decorate the fabric, and optional other embellishments such as sequins, beads, or other desirably-shaped embellishments that are added to the embroidery. In some cases, the single material makes up at least 50% of the underlying fabric, the thread/yarn, and the optional other embellishments. In some cases, the single material makes up at least 75% of the underlying fabric, the thread/yarn, and the optional other embellishments. In some cases, the single material makes up at least 90% of the underlying fabric, the thread/yarn, and the optional other embellishments. In some cases, the single material makes up at least 95% of the underlying fabric, the thread/yarn, and the optional other embellishments. In some cases, the single material makes up substantially 100% of the underlying fabric, the thread/yarn, and the optional other embellishments.

[0032] ***Substantially:*** as used herein, the term "substantially" refers to the qualitative condition of exhibiting total or near-total extent or degree of a characteristic or property of interest. One of ordinary skill in the biological arts will understand that biological and chemical phenomena rarely, if ever, go to completion and/or proceed to completeness or achieve or avoid an absolute result. The term "substantially" is therefore used herein to capture the potential lack of completeness inherent in many biological and chemical phenomena.

[0033] It should be apparent to those skilled in the art that many additional modifications beside those already described are possible without departing from the inventive concepts. In interpreting this disclosure, all terms should be interpreted in the broadest possible manner consistent with the context. Variations of the term "comprising" should be interpreted as referring to elements,

components, or steps in a non-exclusive manner, so the referenced elements, components, or steps may be combined with other elements, components, or steps that are not expressly referenced. Embodiments referenced as "comprising" certain elements are also contemplated as "consisting essentially of" and "consisting of" those elements. When two or more ranges for a particular value are recited, this disclosure contemplates all combinations of the upper and lower bounds of those ranges that are not explicitly recited. For example, recitation of a value of between 1 and 10 or between 2 and 9 also contemplates a value of between 1 and 9 or between 2 and 10.

[0034] As used herein, "silk fibroin" refers to silk fibroin protein whether produced by silkworm, spider, or other insect, or otherwise generated (Lucas et al., Adv. Protein Chem., 13: 107-242 (1958)). Any type of silk fibroin can be used in different embodiments described herein. Silk fibroin produced by silkworms, such as *Bombyx mori*, is the most common and represents an earth-friendly, renewable resource. For instance, silk fibroin used in a silk film may be attained by extracting sericin from the cocoons of *B. mori*. Organic silkworm cocoons are also commercially available. There are many different silks, however, including spider silk (e.g., obtained from *Nephila clavipes*), transgenic silks, genetically engineered silks, such as silks from bacteria, yeast, mammalian cells, transgenic animals, or transgenic plants, and variants thereof, that can be used. See, e.g., WO 97/08315 and U.S. Pat. No. 5,245,012, each of which is incorporated herein by reference in their entireties.

[0035] In response to the pressing issue of the waste produced by the fashion and textile industry, and particularly silk-based waste, the disclosure herein proposes a solution for recycling waste silk.

[0036] Silk textile waste-stream (which may include a finished textile) may be used as feedstock for silk solutions according to example embodiments of the disclosure. The finished textile of example embodiments of the disclosure may include, for example, a processed, colored, and/or used silk fabric (e.g., clothing) used as the starting material for example methods herein. The finished textile may have reached the end of what was heretofore considered its useful life.

[0037] Referring to Fig. 1, an example method according to an embodiment of the disclosure may include analyzing a finished textile to identify a dye-binding mode of a dye, where the finished textile may include silk and the dye. The analysis may include determining a presence or absence of a coating and optionally removing the coating. The analysis may include determining if the textile is pure silk. If not, the analysis may include defining a minimum % of silk acceptable for the process, or evaluating an incompatibility of the process for specific fiber types. The analysis may include determining if the textile is dyed. If it is, the analysis may include identification of the dye binding type to silk and evaluation of the process compatibility. Optionally, the process may include bleaching in response to the analysis. The analysis may culminate with processing the textile and/or

one or more parameters for processing the textile, or it may terminate at any point in the analysis of coatings, purity, and dyes.

[0038] The dye-binding mode may be a mode of the dye's binding to the silk. Further, the method may include selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the identified dye-binding mode and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0039] The example method may include identifying the dye-binding mode as reactive, and selecting the one or more regeneration parameters in response to the reactive dye-binding mode.

[0040] The example method may include identifying the dye-binding mode as direct, and selecting one or more regeneration parameters in response to the direct dye-binding mode.

[0041] The example method may include identifying the dye-binding mode as electrostatic, and selecting one or more regeneration parameters in response to the electrostatic dye-binding mode.

[0042] In the example method, the dye may be present at least one of throughout the finished textile or in a pattern on the finished textile.

[0043] Analyzing the finished textile according to the example method may include analyzing the finished textile by at least one of Fourier transform infrared ("FTIR") spectroscopy or Raman spectroscopy. In some examples, FTIR spectroscopy may provide information about the finished textile including at least one of a composition of the finished textile, the presence or absence of coatings, or a dye-binding mode. In some examples, Raman spectroscopy may provide information about the finished textile including a dye-binding mode or a potential dye.

[0044] Analyzing the finished textile according to the example method may include analyzing the finished textile by at least one of a chemical assay or an enzymatic assay.

[0045] The example method may include sorting the finished textile in response to the identified dye-binding mode. The sorting may include excluding undesirable finished textile from silk fibroin regeneration.

[0046] The example method may include analyzing the finished textile spectroscopically to determine a proportion of silk in the finished textile, sorting the finished textile in response to the determined proportion, and selecting one or more regeneration parameters of the silk fibroin regeneration further in response to the determined proportion. In some examples, the proportion of the silk in the finished textile (e.g. a purity) may indicate potential uses of the finished textile.

[0047] The example method may include determining or estimating a molecular weight of a silk fibroin of the silk in the finished textile. Furthermore, the example method may include selecting one or more regeneration parameters of the silk fibroin regeneration of the finished textile further in response to the determined or estimated molecular weight, and the example method may include sorting the finished textile in response to the determined or estimated molecular weight. In some

examples, the molecular weight of the silk fibroin may be an important indicator of the potential uses for the silk. For example, some technologies may not have particular molecular weight requirements, while others, such as silk bioplastic, may, in some examples, only be obtained from a molecular weight of 150kDa, or adhesives, which may, in some examples, show generally better performance at around 75kDa.

[0048] An example method according to an embodiment of the disclosure may include analyzing a finished textile spectroscopically to identify an aspect of a dye, where the finished textile may include silk and the dye. The example method may further include
selecting one or more regeneration parameters of a silk fibroin regeneration process to be applied to the finished textile in response to the identified aspect of the dye, and
regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters (as discussed with reference to example embodiments herein). For example,

[0049] The example embodiment may include sorting the finished textile in response to the identified aspect of the dye. Sorting may excluding undesirable finished textile from silk fibroin regeneration.

[0050] The aspect of the dye may be at least one of a color, an intensity, a substantivity, a chemical formula, a fluorescence, or a dye-binding type. The dye may be present at least one of throughout the finished textile or in a pattern on or of the finished textile.

[0051] The example method may include determining or estimating a molecular weight of a silk fibroin of the silk, and selecting one or more regeneration parameters of the silk fibroin regeneration of the finished textile further in response to the determined or estimated molecular weight.

[0052] The example method may include determining or estimating a molecular weight of a silk fibroin of the silk, and further sorting the finished textile in response to the determined or estimated molecular weight.

[0053] The example method may include identifying the dye is a blue dye, and selecting one or more regeneration parameters of the silk fibroin regeneration in response to the blue dye. The example method may further include identifying the dye is a red dye, and selecting one or more regeneration parameters of the silk fibroin regeneration in response to the red dye. The example method may further include identifying the dye is a black dye, and selecting one or more regeneration parameters of the silk fibroin regeneration in response to the black dye. The example method may further include identifying the dye is a green dye, and selecting one or more regeneration parameters of the silk fibroin regeneration in response to the green dye. The example method may further include identifying the dye is a yellow dye; and selecting one or more regeneration parameters of the silk fibroin regeneration in response to the yellow dye. The example method may further include identifying the dye is an orange dye, and selecting one or more

regeneration parameters of the silk fibroin regeneration in response to the orange dye. The example method may further include identifying the dye is a violet dye, and selecting one or more regeneration parameters of the silk fibroin regeneration response to the violet dye.

[0054] The example method may include identifying the dye-binding type is reactive, and selecting one or more regeneration parameters of the silk fibroin regeneration in response to the reactive dye-binding type.

[0055] The example method may include identifying the dye-binding type is direct, and selecting one or more regeneration parameters of the silk fibroin regeneration in response to the direct dye-binding type.

[0056] The example method may include identifying the dye-binding mode as electrostatic, and selecting one or more regeneration parameters in response to the electrostatic dye-binding mode.

[0057] An example method according to an embodiment of the present disclosure may include analyzing a finished textile to determine a proportion of silk in the finished textile, selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the determined proportion, and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0058] The example method may include sorting the finished textile in response to the determined proportion. Sorting may exclude undesirable finished textile from the silk fibroin regeneration in response to the determined proportion not meeting a threshold.

[0059] The example method may include restricting undesirable finished textile from the silk fibroin regeneration if the determined proportion does not meet a defined minimum proportion.

[0060] Analyzing the finished textile may include analyzing by FTIR spectroscopy. The method may include further analyzing the finished textile spectroscopically to identify a dye-binding mode, further sorting the finished textile in response to the identified dye-binding mode, and selecting one or more regeneration parameters of the silk fibroin regeneration further in response to the identified dye-binding mode.

[0061] The example method may further include determining or estimating a molecular weight of a silk fibroin of the silk, and selecting one or more regeneration parameters of the silk fibroin regeneration of the finished textile further in response to the determined or estimated molecular weight.

[0062] The example method may further include determining or estimating a molecular weight of a silk fibroin of the silk; and further sorting the finished textile in response to the determined or estimated molecular weight.

[0063] The proportion may be between 5% and 95% or between 10% and 80%, including but not limited to, at least 5%, at least 15%, at least 20%, at least 25%, at least 50%, at least 60%, at least

80%, or at least 90%, and at most 95%, at most 75%, at most 50%, at most 25%, at most 10%, or at most 5%.

[0064] An example method according to an embodiment of the present disclosure may include analyzing a finished textile to determine a composition of the finished textile, the finished textile comprising silk, selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the determined composition, and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0065] The example method may include sorting the finished textile in response to the determined composition. Sorting may exclude undesirable finished textile from the silk fibroin regeneration in response to the determined composition not meeting a threshold.

[0066] The example method may include restricting undesirable finished textile from the silk fibroin regeneration if the determined proportion does not meet a defined minimum composition.

[0067] Analyzing the finished textile may include analyzing by FTIR spectroscopy. The example method may include further analyzing the finished textile spectroscopically to identify a dye-binding mode, further sorting the finished textile in response to the identified dye-binding mode, and selecting one or more regeneration parameters of the silk fibroin regeneration further in response to the identified dye-binding mode.

[0068] Analyzing the finished textile may include analyzing by Raman spectroscopy. For example, the example method may identify a dye-binding mode or a potential dye through Raman analysis.

[0069] The example method may further include determining or estimating a molecular weight of a silk fibroin of the silk, and selecting one or more regeneration parameters of the silk fibroin regeneration of the finished textile further in response to the determined or estimated molecular weight.

[0070] The example method may further include determining or estimating a molecular weight of a silk fibroin of the silk, and further sorting the finished textile in response to the determined or estimated molecular weight.

[0071] An example method according to an embodiment of the present disclosure may include determining or estimating a molecular weight of a silk fibroin of silk in a finished textile, selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile further in response to the determined or estimated molecular weight, and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0072] The example method may exclude undesirable finished textile from silk fibroin regeneration in response to determining or estimating the molecular weight is below a threshold.

The example method may exclude undesirable finished textile from silk fibroin regeneration in response to determining or estimating the molecular weight is above a threshold.

[0073] An example method according to an embodiment of the present disclosure may include imaging a finished textile to determine at least one of an age or a wear value of the finished textile, the finished textile comprising silk, selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the at least one determined age or determined wear value, and

regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters. In some examples, it has been found that determining a wear value of the finished textile (e.g., how worn the fibers are, such as frayed) may provide an important indication of how difficult it may be to process the finished textile according to example methods described herein.

[0074] The example method may include sorting the finished textile in response to the at least one determined age or determined wear value. Sorting may exclude undesirable finished textile from silk fibroin regeneration.

[0075] The example method may include determining or estimating a molecular weight of a silk fibroin of the silk, and selecting one or more regeneration parameters of the silk fibroin regeneration of the finished textile further in response to the determined or estimated molecular weight.

[0076] The example method may include determining or estimating a molecular weight of a silk fibroin of the silk, and sorting the finished textile in response to the determined or estimated molecular weight.

[0077] An example method according to an embodiment of the present disclosure may include determining at least one of an age or a wear value of a finished textile, the finished textile comprising silk, where determining comprises at least one of imaging the finished textile, performing a microscopic analysis of the finished textile, or performing a mechanical analysis of the finished textile, selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the at least one determined age or determined wear value, and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0078] The example method may include sorting the finished textile in response to the at least one determined age or determined wear value. Sorting may exclude undesirable finished textile from silk fibroin regeneration.

[0079] The example method may include determining or estimating a molecular weight of a silk fibroin of the silk, and selecting one or more regeneration parameters of the silk fibroin regeneration of the finished textile further in response to the determined or estimated molecular weight.

[0080] The example method may include determining or estimating a molecular weight of a silk fibroin of the silk, and sorting the finished textile in response to the determined or estimated molecular weight.

[0081] An example method according to an embodiment of the present disclosure may include identifying a presence of a coating of a finished textile, the finished textile comprising silk, selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the presence of the coating, and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0082] The coating may be at least one of a waterproofing material, a synthetic plastic, an antibacterial material, a conductive material, or a flame-retardant material.

[0083] Sorting the finished textile may be in response to at least one of the presence or an absence of the coating. Sorting may exclude undesirable finished textile from silk fibroin regeneration.

[0084] Identifying may include at least one of spectroscopically analyzing the finished textile, imaging the finished textile, performing a chemical assay of the finished textile, or performing an enzymatic assay of the finished textile.

[0085] The example method may further include restricting undesirable finished textile from the silk fibroin regeneration in response to identifying the presence. The example method may further include removing, in response to identifying the presence, the coating from the finished textile prior to silk fibroin regeneration. In some examples, organic solvents may be used to remove a synthetic plastic, a waterproofing layer, and/or other coating from the finished textile.

[0086] An example method according to an embodiment of the present disclosure may include identifying a presence or an absence of a coating of a finished textile, the finished textile comprising silk, sorting the finished textile in response to at least one of the presence or the absence of the coating, removing, in response to the presence, the coating from the finished textile, analyzing the finished textile to determine a proportion of silk in the finished textile, sorting the finished textile in response to the determined proportion;

analyzing the finished textile to identify an aspect of a dye, sorting the finished textile in response to the identified aspect of the dye, selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to at least one of the determined proportion, the presence or the absence of the coating, or the aspect of the dye, and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0087] The example method may include restricting undesirable finished textile from the silk fibroin regeneration in response to the determined proportion not meeting a threshold.

[0088] Analyzing may include at least one of spectroscopically analyzing the finished textile, imaging the finished textile, performing a chemical assay of the finished textile, or performing an enzymatic assay of the finished textile.

[0089] The aspect of the dye may include at least one of a presence, a color, an intensity, a substantivity, a chemical formula, a fluorescence, or a binding type (e.g., direct, reactive, electrostatic).

[0090] The example method may further include bleaching the finished textile in response to the aspect of the dye.

[0091] An example method according to an embodiment of the present disclosure may include identifying a presence or an absence of a coating of a finished textile, the finished textile comprising silk, removing, in response to the presence, the coating from the finished textile, analyzing the finished textile to determine a proportion of silk in the finished textile, analyzing the finished textile to identify an aspect of a dye, selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to at least one of the determined proportion, the presence or the absence of the coating, or the aspect of the dye, and regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

[0092] In an example method according to an embodiment of the present disclosure, the finished textile may be a post-consumer waste product. In an example method according to an embodiment of the present disclosure, the finished textile may be at least one of a dyed textile, a coated textile, a waterproofed textile, a silkscreened textile, or a heterogenous product.

[0093] In an example method according to an embodiment of the present disclosure, the regenerated silk fibroin solution may include a color that is within 80% of a measured color of a cocoon-derived silk. In some examples, it has been found that the regenerated silk fibroin solution with a color that is within 80% of the measured color of the cocoon-derived silk may approach the performance of the cocoon-derived silk.

[0094] In an example method according to an embodiment of the present disclosure, the regenerated silk fibroin solution comprises a transparency that is within 80% of a measured transparency of a cocoon-derived silk. In some examples, it has been found that the regenerated silk fibroin solution with a transparency (e.g., a film/solution transparency) that is within 80% of the measured transparency of the cocoon-derived silk may approach the performance of the cocoon-derived silk.

[0095] In an example method according to an embodiment of the present disclosure, the regenerated silk fibroin solution may include a viscosity that is within 80% of a measured viscosity of a cocoon-derived silk. In some examples, it has been found that the regenerated silk fibroin

solution with a viscosity that is within 80% of the measured viscosity of the cocoon-derived silk may approach the performance of the cocoon-derived silk.

[0096] In an example method according to an embodiment of the present disclosure, the regenerated silk fibroin solution may include a secondary structure tuning (β-sheet induction after methanol treatment/water annealing) within 50% of a cocoon-derived silk. In some examples, it has been found that the regenerated silk fibroin solution with a secondary structure tuning that is within 50% of that of the cocoon-derived silk may approach the performance of the cocoon-derived silk.

[0097] An example method according to an embodiment of the present disclosure may include identifying at least one pattern of the finished textile, and selecting one or more regeneration parameters of the silk fibroin regeneration further in response to the at least one pattern.

[0098] In an example method according to an embodiment of the present disclosure, the at least one pattern is a woven pattern, a printed pattern, or a mixed media pattern.

[0099] An example method according to an embodiment of the present disclosure may include mechanically processing the finished textile. In an example method according to an embodiment of the present disclosure, mechanically processing may be at least one of shredding, flaking, cutting, milling, or grinding.

[0100] In an example method according to an embodiment of the present disclosure, silk fibroin regeneration may include the steps of dissolving a degummed, rinsed, and dried finished textile in a salt solution to form a silk fibroin solution, dialyzing the silk fibroin solution against distilled water to obtain a dialyzed silk fibroin solution in water, centrifuging the dialyzed silk fibroin solution twice to obtain a supernatant, and diluting the supernatant in deionized water to obtain a regenerated silk fibroin solution.

[0101] In the example method, the dialyzed silk fibroin solution may be a 5–8 % wt silk fibroin solution.

[0102] In the example method, dissolving may be in an oven at 60 °C for 4 hours.

[0103] In the example method, the silk fibroin solution may be stirred after an hour of elapsed dissolving time.

[0104] In the example method, the silk fibroin solution may be stirred after two hours of elapsed dissolving time.

[0105] In the example method, the salt solution may be a LiBr solution. The salt solution may be a 20 w/v% LiBr solution. A concentration of LiBr may be 9.3M.

[0106] In the example method, dialyzing may be in dialysis cassettes.

[0107] In the example method, dialyzing may be for 36 hours.

[0108] In the example method centrifuging may be at 8000 rpm.

[0109] The example method may include storing the regenerated silk fibroin solution at 4 °C

[0110] In an example method according to an embodiment of the present disclosure, the silk fibroin regeneration may include at least one organic solvent.

[0111] An example method according to an embodiment of the present disclosure may include collecting a precipitated material; and drying the precipitated material.

[0112] An example method according to an embodiment of the present disclosure may include collecting a precipitated material, and repeating the silk fibroin regeneration.

[0113] An example method according to an embodiment of the present disclosure may include collecting a precipitated material to be used as a moldable material.

[0114] An example method according to an embodiment of the present disclosure may include determining a presence of at least one of a non-silk fiber/component or a contaminant of the finished textile, and at least one of: sorting the finished textile in response to the presence, or selecting one or more regeneration parameters of the silk fibroin regeneration further in response to the presence. However, it has been found that in some examples, the example methods according to embodiments described herein may not be particularly sensitive to the presence of the contaminant in the finished textile. As the source of the finished textile may, in some embodiments, be inherently variable, this may provide advantages in requiring less sorting than expected.

[0115] In an example method according to an embodiment of the present disclosure, selecting one or more regeneration parameters may include selecting one or more dissolution operational parameters.

[0116] In an example method according to an embodiment of the present disclosure, the one or more dissolution operational parameters may include a dissolution temperature, a dissolution length of time, or a combination thereof.

[0117] In an example method according to an embodiment of the present disclosure, selecting one or more regeneration parameters of a silk fibroin regeneration process may include selecting one or more chemical composition parameters. Furthermore, the one or more chemical composition parameters may include a dissolution chemical composition, a desalinated chemical composition, a concentrated chemical composition, a dilute chemical composition, or a combination thereof.

[0118] In an example method according to an embodiment of the present disclosure, the molecular weight may be between 5 kDa to 400 kDa, between 3.5 kDa and 120 kDa, or between 20 kDa and 125 kDa, including but not limited to, at least 5 kDa, at least 20 kDa, at least 30 kDa, at least 40 kDa, at least 50 kDa, at least 60 kDa, at least 100 kDa, at least 200 kDa, at least 300 kDa, at least 400 kDa, at most 400 kDa, at most 375 kDa, at most 350 kDa, at most 325 kDa, at most 300 kDa, at most 200 kDa, at most 100 kDa, at most 50 kDa, at most 20 kDa, or at most 5 kDa.

[0119] An example method according to an embodiment of the present disclosure may include transforming a finished textile (e.g., silk waste) into a liquid. For example, the liquid may be a

regenerated silk fibroin solution. The example method may further include powderizing the liquid (e.g., the regenerated silk fibroin solution) and using the powderized liquid as an ink toner. Such an ink toner may be "circularly processed" and sustainable. A color of the ink toner may be determined by a coloration of a finished textile from which the liquid, such as the regenerated silk fibroin solution, is made (e.g., regenerated), as described by example with reference to example embodiments herein. For example, the color of the ink toner may be a same color as the finished textile, or an alteration of the color of the finished textile.

[0120] An example method according to an embodiment of the present disclosure may include pressing and/or forming a finished textile (e.g., a silk waste) at a high pressure to yield solid material formats and/or silk composites. In some examples, the finished textile may be raw, mechanically processed (e.g., shredded, flaked, cut, milled, and/or ground), mixed with silk fibroin (such as a regenerated silk fibroin solution), or sprinkled with a powderized silk fibroin (e.g., a powderized generated silk fibroin solution) prior to the pressing and/or forming. In some examples, the finished textile may thereby be impregnated with silk fibroin to yield the solid material formats and/or silk composites.

[0121] An example method according to an embodiment of the present disclosure may include thermal processing of a finished textile (e.g., a silk waste) together with one or more other biomaterials, such as lignin or cellulose, or other textiles, such as cotton. In some examples, the finished textile may be raw, mechanically processed (e.g., shredded, flaked, cut, milled, and/or ground), mixed with silk fibroin (such as a regenerated silk fibroin solution), or sprinkled with a powderized silk fibroin (e.g., a powderized generated silk fibroin solution) prior to thermal processing together with the one or more other biomaterials. In some examples, the finished textile and one or more other biomaterials may thereby yield solid material formats and/or silk composites.

[0122] An example composition according to an embodiment of the present disclosure may be made by an example method according to any one or more embodiments of the present disclosure described herein.

[0123] An example composition according to an embodiment of the present disclosure may include a silk fibroin, and a regenerated silk fibroin made by a method of the present disclosure.

[0124] An example composition according to an embodiment of the present disclosure may include a regenerated silk fibroin made by an example method according to any one or more embodiments of the present disclosure described herein and a functionalizing agent.

[0125] An example composition according to an embodiment of the present disclosure may include a silk fibroin, a regenerated silk fibroin made by a method disclosed herein, and a functionalizing agent.

[0126] In an example composition according to an embodiment of the present disclosure, the regenerated silk fibroin may include a color that is within 80% of a measured color of a cocoon-derived silk.

[0127] In an example composition according to an embodiment of the present disclosure, the regenerated silk fibroin may include a transparency that is within 80% of a measured transparency of a cocoon-derived silk.

[0128] In an example composition according to an embodiment of the present disclosure, the regenerated silk fibroin may include a viscosity that is within 80% of a measured viscosity of a cocoon-derived silk.

[0129] In an example composition according to an embodiment of the present disclosure, the regenerated silk fibroin may include a secondary structure tuning (β-sheet induction after methanol treatment/water annealing) within 50% of a cocoon-derived silk.

[0130] An example article according to an embodiment of the present disclosure may be made from regenerated silk fibroin made by a method according to any one or more embodiments of the present disclosure described herein.

[0131] An example article according to an embodiment of the present disclosure may be made from a composition according to any one or more embodiments of the present disclosure described herein.

[0132] An example article according to an embodiment of the present disclosure may be at least one of a silk foam, a film, a silk leather, an adhesive, a building construction material, a thermal insulator, a silk embellishment, a silk sequin, a silk embroidery, a silk thread, a silk mono-material embroidery, or a PDA mechanical sensor.

[0133] The example article may be the adhesive, and the adhesive may exhibit at least 20% of an adhesive strength of a cocoon-derived silk. In some examples, it has been found that the example article with at least 20% of the adhesive strength of the cocoon-derived silk may approach the performance of the cocoon-derived silk.

[0134] The example article may be the film having a film transparency of at least 80% of a comparison transparency of a comparison film made of a cocoon-derived silk.

[0135] An example article according to an embodiment of the present disclosure may exhibit at least one mechanical property that is at least 50% of a same property in an article made from a cocoon-derived silk. In some examples, it has been found that the example article with at least 50% of the same mechanical property as an article made from the cocoon-derived silk may approach the performance of the article made from the cocoon-derived silk.

[0136] An example article according to an embodiment of the present disclosure may include an additional recycled component (e.g., recycled cardboard, wool) therein.

[0137] The disclosure herein also concerns a new procedure for making sustainable embroidery. Embroidery consists of at least a base textile, thread, and embellishments. In some examples, embroidery made from silk may be known as a silk mono-material embroidery. The silk mono-material embroidery's components can be made of silk derived from textile waste or from cocoons. The silk mono-material embroidery can be recycled into a silk fibroin solution. This recycled silk solution can then be used to make films, sponges, pigments, etc. or be processed back into embellishments for use in new embroideries.

[0138] Current embroidery practices in the fashion industry combine base fabric, thread and embellishments made from different types of materials such as plastic, cotton, silk, jute, etc. For producing colored embroideries, mineral and chemical based dyes are used to dye fabrics, threads, and embellishments. Recent shifts in the fashion industry towards sustainability has led to the use of organic fabrics, recycled threads, recycled plastic sequins, and natural dyes. Many of the plastic embellishments commonly used in the fashion industry are categorized as microplastics according to their sizes. Once embroidered onto different types of fabrics with synthetic or organic threads, it makes recycling very complex and nearly impossible. Therefore, this disclosure would be a new and more sustainable approach to embroidery as compared to existing embroidery methods.

[0139] Current embroidery practices result in non-recyclability of textiles and negative environmental impact. The disclosed silk mono-material embroidery is made from a single material, silk, that facilitates streamlined recycling procedures. This disclosure minimizes environmental pollution by reducing the amount of textile waste sent to landfills, conserving valuable resources, and minimizing the environmental burden of waste disposal. This disclosure presents a sustainable alternative process to current embroidery practices of the fashion industry wherein synthetic threads are used to embroider plastic embellishments onto synthetic fabrics. This disclosure involves embellishments made from silk textile waste that would otherwise be discarded into landfills.

[0140] Silk mono-material embroidery offers advantages to current embroidery practices because of the following reasons: sustainability of the source material (i.e., textile waste), single material in different formats assembled to form a mono-material product, simple recycling process without the need for disassembling the embroidery components, non-toxic recycling in a mild chemical (e.g., lithium bromide (LiBr)), and processing silk textile waste into such embroideries and films facilitates a circular economy by encapsulating and circulating existing toxic/synthetic dyes between products instead of being disposed of into the environment after a single use.

[0141] Disclosed herein are silk embellishments, silk sequins, silk threads, and silk mono-material embroidery made from the disclosed compositions and by the disclosed methods.

[0142] Any of the methods disclosed herein may include drying the composition to form a film, and laser cutting the film to form a silk mono-material embroidery.

[0143] Any of the methods disclosed herein may include coating a silk textile with the composition to form a coated silk textile, drying the coated silk textile, and laser cutting the coated silk textile to form a silk mono-material embroidery.

[0144] Disclosed herein is a textile waste stream processing system. The textile waste stream processing system includes a spectroscopic imaging facility (e.g., FTIR or Raman spectroscopy-based) that is operationally coupled to a textile waste storage vessel including at least one waste textile. The waste textile may or may not include silk among other fabric types. The spectroscopic imaging facility is communicatively coupled to a data collector which is structured to collect a plurality of detection values from the spectroscopic imaging facility upon imaging at least one waste textile in the textile waste storage vessel.

[0145] The textile waste stream processing system includes a data storage that is structured to store a plurality of spectra patterns (e.g., FTIR spectra, Raman spectra, etc.) of textiles, the stored plurality of spectra patterns comprising a library of spectra patterns. For example, the textiles may include any of the textiles described herein (e.g., coated textile, silk textile, textiles with embellishments, finished textile, waste textile, dyed/colored textiles, etc.). The stored plurality of spectra patterns in the library of spectra patterns may further include information regarding one or more of fabric types, dye types, dye-binding modes, coating types, age/wear, and successful dissolution parameters. In this way, the spectra patterns are stored along with information associated with the textile that gave rise to that spectra pattern.

[0146] The textile waste stream processing system includes a data acquisition circuit structured to interpret the plurality of detection values from the data collector. The textile waste stream processing system also includes an expert system analysis circuit structured to analyze the plurality of detection values to determine if one has a recognized spectra pattern corresponding to at least one of the stored plurality of spectra patterns from the library of spectra patterns.

[0147] The textile waste stream processing system includes a response circuit structured to provide an instruction in response to the one of the plurality of detection values having a stored spectra pattern. The response circuit may utilize at least one of industry-specific feedback, the library of spectra patterns, or a user input to select an appropriate instruction. The instruction may be an instruction to further process the waste textile or to restrict it from further processing. The instruction may include dissolution parameters for processing the waste textile.

[0148] The library of spectra patterns may be made available to a spectra pattern marketplace for users to access and to update periodically.

[0149] Fig. 5 depicts an example textile waste stream processing system 500. The textile waste stream processing system 500 includes a spectroscopic imaging facility 502 operationally coupled to a textile waste storage vessel 504, the spectroscopic imaging facility communicatively coupled to a

data collector 508, the data collector 508 structured to collect a plurality of detection values 510 from the spectroscopic imaging facility upon imaging at least one waste textile 512 in the textile waste storage vessel 504. A data storage 514 is structured to store a plurality of spectra patterns of textiles, the stored plurality of spectra patterns comprising a library of spectra patterns 518. A data acquisition circuit 520 is structured to interpret the plurality of detection values from the data collector 508. An expert system analysis circuit 522 is structured to analyze the plurality of detection values 510 to determine if one of the plurality of detection values 510 has a recognized spectra pattern corresponding to at least one of the stored plurality of spectra patterns from the library of spectra patterns 518. A response circuit 524 is structured to provide an instruction 528 in response to the one of the plurality of detection values 510 having a stored spectra pattern. The library of spectra patterns 518 is available to a spectra pattern marketplace 530, where users are provided access to the spectra pattern marketplace 530.

[0150] The spectroscopic systems and analyses described herein can be used in a variety of ways throughout the process of recycling silk waste. In some cases, the spectroscopic analysis can be used to identify resistance to dissolution, which can in some cases without wishing to be bound by any particular theory be from the presence of non-silk materials. In particular, FTIR analysis can identify non-silk materials and prompt the exclusion of those materials from the recycling process. The systems and methods described herein provide for the creation and maintenance of a dynamic FTIR (or other spectroscopic modality) spectra library that is updated with new data as it becomes available and is accessible to provide predictive capability regarding dissolution performance. If the spectroscopic analysis identifies the presence of synthetic fibers (e.g., nylon or polyester) in a given piece of waste, then that piece of waste can be discarded or processed in a separate process. Without wishing to be bound by any particular theory, it is believed that a variety of spectroscopic analysis might be useful (e.g., Raman spectroscopy to identify the presence or absence of dyes and/or identify properties of the dyes).

[0151] According to various embodiments, a variety of functionalizing agents may be used with the silk-containing embodiments described herein (e.g., silk membrane, silk composition, silk articles, silk matrix, silk foam, silk microsphere, liquid composition, whipped silk cream, silk meringue, compressed silk meringue, hot-pressed silk meringue, silk leather, silk powder, silk toner, etc.). It should be understood that the examples herein may recite one or a few silk-containing embodiments but are applicable to any silk-containing embodiment, as applicable. In some embodiments, a functionalizing agent may be any compound or molecule that facilitates the attachment to and/or development (e.g., growth) of one or more endothelial cells on a silk membrane. In some embodiments, a functionalizing agent may be any compound or molecule that facilitates the attachment and/or development (e.g., growth) of one or more megakaryocytes and/or

hematopoietic progenitor cells on a silk matrix and/or silk membrane. In some embodiments, a functionalizing agent may be or comprise an agent suitable for facilitating the production of one or more of white blood cells and red blood cells.

[0152] In some embodiments, a functionalizing agent may be or comprise a cell attachment mediator and/or an extracellular matrix protein, for example: collagen (e.g., collagen type I, collagen type III, collagen type IV or collagen type VI), elastin, fibronectin, vitronectin, laminin, fibrinogen, von Willebrand factor, proteoglycans, decorin, perlecan, nidogen, hyaluronan, and/or peptides containing known integrin binding domains e.g. "RGD" integrin binding sequence, or variations thereof, that are known to affect cellular attachment.

[0153] In some embodiments, a functionalizing agent may be any soluble molecule produced by endothelial cells. Non-limiting examples include fibroblast growth factor-1 (FGF1) and vascular endothelial growth factors (VEGF).

[0154] According to some embodiments, a plurality of functionalizing agents may be used. For example, in some embodiments wherein production of platelets is desired, provided compositions may comprise the use of laminin, fibronectin and/or fibrinogen, and type IV collagen in order to facilitate the attachment and growth of endothelial cells on a silk membrane (e.g., a porous silk membrane) and/or attachment of megakaryocytes to a silk matrix.

[0155] In some embodiments, a functionalizing agent may be embedded or otherwise associated with a silk membrane and/or silk matrix such that at least a portion of the functionalizing agent is surrounded by a silk membrane and/or silk matrix as contrasted to a functionalizing agent simply being positioned along the surface of a silk membrane and/or silk matrix. In some embodiments, a functionalizing agent is distributed along and/or incorporated in substantially the entire surface area of a silk membrane/silk wall. In some embodiments, a functionalizing agent is distributed and/or incorporated only at one or more discrete portions of a silk membrane/wall and/or silk matrix. In some embodiments, a functionalizing agent is distributed in and/or along at least one of the lumen-facing side of a silk wall and the matrix-facing side of a silk wall.

[0156] According to various embodiments, any application-appropriate amount of one or more functionalizing agents may be used. In some embodiments, the amount of an individual functionalizing agent may be between about 1 μg/ml and 1,000 μg/ml (e.g., between about 2 and 1,000, 5 and 1,000, 10 and 1,000, 10 and 500, 10 and 100 μg/ml). In some embodiments, the amount of an individual functionalizing agent may be at least 1 μg/ml (e.g., at least 5, 10, 15, 20 25, 50, 100, 200, 300 400, 500, 600, 700, 800, or 900 μg/ml). In some embodiments, the amount of an individual functionalizing agent is at most 1,000 μg/ml (e.g., 900, 800, 700, 600, 500, 400, 300 200, 100, 90, 80, 70, 60, 50, 40, 30, 20, 10, or 5 μg/ml).

[0157] In some aspects, the composition comprises one or more sensing agents, such as a sensing dye. The sensing agents/sensing dyes are environmentally sensitive and produce a measurable response to one or more environmental factors. In some aspects, the environmentally- sensitive agent or dye may be present in the composition in an effective amount to alter the composition from a first chemical -physical state to a second chemical -physical state in response to an environmental parameter (e.g., a change in pH, light intensity or exposure, temperature, pressure or strain, voltage, physiological parameter of a subject, and/or concentration of chemical species in the surrounding environment) or an externally applied stimulus (e.g., optical interrogation, acoustic interrogation, and/or applied heat). In some cases, the sensing dye is present to provide one optical appearance under one given set of environmental conditions and a second, different optical appearance under a different given set of environmental conditions. Suitable concentrations for the sensing agents described herein can be the concentrations for the colorants and additives described elsewhere herein. A person having ordinary skill in the chemical sensing arts can determine a concentration that is appropriate for use in a sensing application of the inks described herein.

[0158] In some aspects, the first and second chemical-physical state may be a physical property of the composition, such as mechanical property, a chemical property, an acoustical property, an electrical property, a magnetic property, an optical property, a thermal property, a radiological property, or an organoleptic property. Exemplary sensing dyes or agents include, but are not limited to, a pH sensitive agent, a thermal sensitive agent, a pressure or strain sensitive agent, a light sensitive agent, or a potentiometric agent.

[0159] Exemplary pH sensitive dyes or agents include, but are not limited to, cresol red, methyl violet, crystal violet, ethyl violet, malachite green, methyl green, 2-(p- dimethylaminophenylazo) pyridine, paramethyl red, metanil yellow, 4-phenylazodiphenylamine, thymol blue, metacresol purple, orange IV, 4-o-Tolylazo-o-toluindine, quinaldine red, 2,4- dinitrophenol, erythrosine disodium salt, benzopurpurine 4B, N,N-dimethyl-p-(m-tolylazo) aniline, p-dimethylaminoazobenene, 4,4'-bis(2-amino-l-naphthylazo)-2,2'-stilbenedisulfonic acid, tetrabromophenolphthalein ethyl ester, bromophenol blue, Congo red, methyl orange, ethyl orange, 4-(4-dimethylamino-l-naphylazo)-3-methoxybenesulfonic acid, bromocresol green, resazurin, 4-phenylazo-l-napthylamine, ethyl red 2-(1-dimethylaminophenyazo) pyridine, 4-(p-ethoxypehnylazo)-m-phenylene-diamine monohydrochloride, resorcin blue, alizarin red S, methyl red, propyl red, bromocresol purple, chlorophenol red, p-nitrophenol, alizarin 2-(2,4-dinitrophenylazo) l-napthol-3,6-disulfonic acid, bromothymol blue, 6,8-dinitro-2,4-(1H) quinazolinedione, brilliant yellow, phenol red, neutral red, m-nitrophenol, cresol red, turmeric, metacresol purple, 4,4'-bis(3-amino-l-naphthylazo)-2,2'-stilbenedisulfonic acid, thymol blue, p-naphtholbenzein, phenolphthalein, o-cresolphthalein, ethyl bis(2,4-dimethylphenyl) ethanoate,

thymolphthalein, nitrazine yellow, alizarin yellow R, alizarin, p-(2,4-dihydroxyphenylazo) benzenesulfonic acid, 5,5'-indigodisulfonic acid, 2,4,6-trinitrotoluene, 1,3,5-trinitrobenezne, and clayton yellow.

[0160] Exemplary light responsive dyes or agents include, but are not limited to, photochromic compounds or agents, such as triarylmethanes, stilbenes, azasilbenes, nitrones, fulgides, spiropyrans, napthopyrans, spiro-oxzines, quinones, derivatives and combinations thereof.

[0161] Exemplary potentiometric dyes include, but are not limited to, substituted amiononaphthylehenylpridinium (ANEP) dyes, such as di-4-ANEPPS, di-8-ANEPPS, and N-(4-Sulfobutyl)-4-(6-(4-(Dibutylamino)phenyl)hexatrienyl)Pyridinium (RH237).

[0162] Exemplary temperature sensitive dyes or agents include, but are not limited to, thermochromic compounds or agents, such as thermochromic liquid crystals, leuco dyes, fluoran dyes, octadecylphosphonic acid.

[0163] Exemplary pressure or strain sensitive dyes or agents include, but are not limited to, spiropyran compounds and agents.

[0164] Exemplary chemi-sensitive dyes or agents include, but are not limited to, antibodies such as immunoglobulin G (IgG) which may change color from blue to red in response to bacterial contamination.

[0165] In some aspects, the compositions comprise one or more additive, dopant, or biologically active agent suitable for a desired intended purpose. In some aspects, the additive or dopant may be present in the composition in an amount effective to impart an optical or organoleptic property to the composition. Exemplary additives or dopants that impart optical or organoleptic properties include, but are not limited to, dyes/pigments, flavorants, aroma compounds, granular or fibrous fillers.

[0166] Additionally or alternatively, the additive, dopant, or biologically active agent may be present in the composition in an amount effective to "functionalize" the composition to impart a desired mechanical property or added functionality to the composition. Exemplary additive, dopants, or biologically active agent that impart the desired mechanical property or added functionality include, but are not limited to: environmentally sensitive/sensing dyes; active biomolecules; conductive or metallic particles; micro and nanofibers (e.g., silk nanofibers for reinforcement, carbon nanofibers); nanotubes; inorganic particles (e.g., hydroxyapatite, tricalcium phosphate, bioglasses); drugs (e.g., antibiotics, small molecules or low molecular weight organic compounds); proteins and fragments or complexes thereof (e.g., enzymes, antigens, antibodies and antigen-binding fragments thereof); DNA/RNA (e.g., siRNA, miRNA, mRNA); cells and fractions thereof (viruses and viral particles; prokaryotic cells such as bacteria; eukaryotic cells such as mammalian cells and plant cells; fungi).

[0167] In some aspects, the additive or dopant comprises a flavoring agent or flavorant.

[0168] Exemplary flavorants include ester flavorants, amino acid flavorants, nucleic acid flavorants, organic acid flavorants, and inorganic acid flavorants, such as, but not limited to, diacetyl, acetylpropionyl, acetoin, isoamyl acetate, benzaldehyde, cinnamaldehyde, ethyl propionate, methyl anthranilate, limonene, ethyl decadienoate, allyl hexanoate, ethyl maltol, ethylvanillin, methyl salicylate, manzanate, glutamic acid salts, glycine salts, guanylic acids salts, inosinic acid salts, acetic acid, ascorbic acid, citric acid, fumaric acid, lactic acid, malic acid, phosphoric acid, tartaric acid, derivatives, and mixtures thereof.

[0169] In some aspects, the additive or dopant comprises an aroma compound. Exemplary aroma compounds include ester aroma compounds, terpene aroma compounds, cyclic terpenes, and aromatic aroma compounds, such as, but not limited to, geranyl acetate, methyl formate, metyl acetate, methyl propionate, methyl butyrate, ethyl acetate, ethyl butyrate, isoamyl acetate, pentyl butrate, pentyl pentanoate, octyl acetate, benzyl acetate, methyl anthranilate, myrecene, geraniol, nerol, citral, cironellal, cironellol, linalool, nerolidol, limonene, camphor, menthol, carone, terpineol, alpha-lonone, thujone, eucalyptol, benzaldehyde, eugenol, cinnamaldehyde, ethyl maltol, vanillin, anisole, anethole, estragole, thymol.

[0170] In some aspects, the additive or dopant comprises a colorant, such as a dye or pigment. In some aspects, the dye or pigment imparts a color or grayscale to the composition. The colorant can be different than the sensing agents and/or sensing dyes below. Any organic and/or inorganic pigments and dyes can be included in the inks. Exemplary pigments suitable for use in the present disclosure include International Color Index or C.I. Pigment Black Numbers 1 , 7, 1 1 and 31 , C.I. Pigment Blue Numbers 15, 15 : 1 , 15 :2, 15 :3, 15 :4, 15 :6, 16, 27, 29, 61 and 62, C.I. Pigment Green Numbers 7, 17, 18 and 36, C.I. Pigment Orange Numbers 5, 13, 16, 34 and 36, C.I. Pigment Violet Numbers 3, 19, 23 and 27, C.I. Pigment Red Numbers 3, 17, 22, 23, 48: 1 , 48:2, 57: 1 , 81 : 1 , 81 :2, 81 :3, 81 :5, 101 , 1 14, 122, 144, 146, 170, 176, 179, 181 , 185, 188, 202, 206, 207, 210 and 249, C.I. Pigment Yellow Numbers 1 , 2, 3, 12, 13, 14, 17, 42, 65, 73, 74, 75, 83, 30, 93, 109, 1 10, 128, 138, 139, 147, 142, 151 , 154 and 180, D&C Red No. 7, D&C Red No. 6 and D&C Red No. 34, carbon black pigment (such as Regal 330, Cabot Corporation), quinacridone pigments (Quinacridone Magenta (228-0122), available from Sun Chemical Corporation, Fort Lee, N.J.), diarylide yellow pigment (such as AAOT Yellow (274- 1788) available from Sun Chemical Corporation); and phthalocyanine blue pigment (such as Blue 15 :3 (294-1298) available from Sun Chemical Corporation). The classes of dyes suitable for use in present invention can be selected from acid dyes, natural dyes, direct dyes (either cationic or anionic), basic dyes, and reactive dyes. The acid dyes, also regarded as anionic dyes, are soluble in water and mainly insoluble in organic solvents and are selected, from yellow acid dyes, orange acid dyes, red acid dyes, violet acid dyes, blue acid dyes, green acid dyes, and black acid dyes. European Patent 0745651, incorporated herein by reference,

describes a number of acid dyes that are suitable for use in the present disclosure. Exemplary yellow acid dyes include Acid Yellow 1 International Color Index or C.I. 10316); Acid Yellow 7 (C.I. 56295); Acid Yellow 17 (C.I. 18965); Acid Yellow 23 (C.I. 19140); Acid Yellow 29 (C.I. 18900); Acid Yellow 36 (C.I. 13065); Acid Yellow 42 (C.I. 22910); Acid Yellow 73 (C.I. 45350); Acid Yellow 99 (C.I. 13908); Acid Yellow 194; and Food Yellow 3 (C.I. 15985). Exemplary orange acid dyes include Acid Orange 1 (C.I. 13090/1); Acid Orange 10 (C.I. 16230); Acid Orange 20 (C.I. 14603); Acid Orange 76 (C.I. 18870); Acid Orange 142; Food Orange 2 (C.I. 15980); and Orange B.

[0171] Exemplary red acid dyes include Acid Red 1. (C.I. 18050); Acid Red 4 (C.I. 14710); Acid Red 18 (C.I. 16255), Acid Red 26 (C.I. 16150); Acid Red 2.7 (C.I. as Acid Red 51 (C.I. 45430, available from BASF Corporation, Mt. Olive, N.J.) Acid Red 52 (C.I. 45100); Acid Red 73 (C.I. 27290); Acid Red 87 (C. I. 45380); Acid Red 94 (C.I. 45440) Acid Red 194; and Food Red 1 (C.I. 14700). Exemplary violet acid dyes include Acid Violet 7 (C.I. 18055); and Acid Violet 49 (C.I. 42640). Exemplary blue acid dyes include Acid Blue 1 (C.I. 42045); Acid Blue 9 (C.I. 42090); Acid Blue 22 (C.I. 42755); Acid Blue 74 (C.I. 73015); Acid Blue 93 (C.I. 42780); and Acid Blue 158A (C.I. 15050). Exemplary green acid dyes include Acid Green 1 (C.I. 10028); Acid Green 3 (C.I. 42085); Acid Green 5 (C.I. 42095); Acid Green 26 (C.I. 44025); and Food Green 3 (C.I. 42053). Exemplary black acid dyes include Acid Black 1 (C.I. 20470); Acid Black 194 (Basantol® X80, available from BASF Corporation, an azo/l :2 CR-complex.

[0172] Exemplary direct dyes for use in the present disclosure include Direct Blue 86 (C.I. 74180); Direct Blue 199; Direct Black 168; Direct Red 253; and Direct Yellow 107/132 (C.I. Not Assigned).

[0173] Exemplary natural dyes for use in the present disclosure include Alkanet (C.I. 75520,75530); Annafto (C.I. 75120); Carotene (C.I. 75130); Chestnut; Cochineal (C.I.75470); Cutch (C.I. 75250, 75260); Divi-Divi; Fustic (C.I. 75240); Hypernic (C.I. 75280); Logwood (C.I. 75200); Osage Orange (C.I. 75660); Paprika; Quercitron (C.I. 75720); Sanrou (C.I. 75100) ; Sandal Wood (C.I. 75510, 75540, 75550, 75560); Sumac; and Tumeric (C.I. 75300). Exemplary reactive dyes for use in the present disclosure include Reactive Yellow 37 (monoazo dye); Reactive Black 31 (disazo dye); Reactive Blue 77 (phthalo cyanine dye) and Reactive Red 180 and Reactive Red 108 dyes. Suitable also are the colorants described in The Printing Ink Manual (5th ed., Leach et al. eds. (2007), pages 289-299. Other organic and inorganic pigments and dyes and combinations thereof can be used to achieve the colors desired.

[0174] In addition to or in place of visible colorants, compositions provided herein can contain ETV fluorophores that are excited in the ETV range and emit light at a higher wavelength (typically 400 nm and above). Examples of ETV fluorophores include but are not limited to materials from the coumarin, benzoxazole, rhodamine, napthalimide, perylene, benzanthrones, benzoxanthones or benzothia- xanthones families. The addition of a UV fluorophore (such as an optical brightener for

instance) can help maintain maximum visible light transmission. The amount of colorant, when present, generally is between 0.05% to 5% or between 0.1% and 1% based on the weight of the composition.

[0175] For non-white compositions, the amount of pigment/dye generally is present in an amount of from at or about 0.1 wt% to at or about 20 wt% based on the weight of the composition. In some applications, a non-white ink can include 15 wt% or less pigment/dye, or 10 wt% or less pigment/dye or 5 wt% pigment/dye, or 1 wt% pigment/dye based on the weight of the composition. In some applications, a non-white ink can include 1 wt% to 10 wt%, or 5 wt% to 15 wt%, or 10 wt% to 20 wt% pigment/dye based on the weight of the composition. In some applications, a non-white ink can contain an amount of dye/pigment that is 1 wt%, 2 wt%, 3 wt%, 4 wt%, 5%, 6 wt%, 7 wt%, 8 wt%, 9 wt%, 10 wt%, 11 wt%, 12 wt%, 13 wt%, 14 wt%, 15%, 16 wt%, 17 wt%, 18 wt%, 19 wt% or 20 wt% based on the weight of the composition.

[0176] For white compositions, the amount of white pigment generally is present in an amount of from at or about 1 wt% to at or about 60 wt% based on the weight of the composition. In some applications, greater than 60 wt% white pigment can be present. Preferred white pigments include titanium dioxide (anatase and rutile), zinc oxide, lithopone (calcined coprecipitate of barium sulfate and zinc sulfide), zinc sulfide, blanc fixe and alumina hydrate and combinations thereof, although any of these can be combined with calcium carbonate. In some applications, a white ink can include 60 wt% or less white pigment, or 55 wt% or less white pigment, or 50 wt% white pigment, or 45 wt% white pigment, or 40 wt% white pigment, or 35 wt% white pigment, or 30 wt% white pigment, or 25 wt% white pigment, or 20 wt% white pigment, or 15 wt% white pigment, or 10 wt% white pigment, based on the weight of the composition. In some applications, a white ink can include 5 wt% to 60 wt%, or 5 wt% to 55 wt%, or 10 wt% to 50 wt%, or 10 wt% to 25 wt%, or 25 wt% to 50 wt%, or 5 wt% to 15 wt%, or 40 wt% to 60 wt% white pigment based on the weight of the composition. In some applications, a non-white ink can an amount of dye/pigment that is 5%, 6 wt%, 7 wt%, 8 wt%, 9 wt%, 10 wt%, 11 wt%, 12 wt%, 13 wt%, 14 wt%, 15%, 16 wt%, 17 wt%, 18 wt%, 19 wt%, 20 wt%, 21 wt%, 22 wt%, 23 wt%, 24 wt%, 25%, 26 wt%, 27 wt%, 28 wt%, 29 wt%, 30 wt%, 31 wt%, 32 wt%, 33 wt%, 34 wt%, 35%, 36 wt%, 37 wt%, 38 wt%, 39 wt%, 40 wt%, 41 wt%, 42 wt%, 43 wt%, 44 wt%, 45%, 46 wt%, 47 wt%, 48 wt%, 49 wt%, 50 wt%, 51 wt%, 52 wt%, 53 wt%, 54 wt%, 55%, 56 wt%, 57 wt%, 58 wt%, 59 wt% or 60 wt% based on the weight of the composition.

[0177] In some aspects, the additive or dopant comprises a conductive additive. Exemplary conductive additives include, but are not limited to graphite, graphite powder, carbon nanotubes, and metallic particles or nanoparticles, such as gold nanoparticles. In some aspects, the conductive additive is biocompatible and non-toxic.

[0178] In some aspects, the additive is a biologically active agent. The term "biologically active agent" as used herein refers to any molecule which exerts at least one biological effect in vivo. For example, the biologically active agent can be a therapeutic agent to treat or prevent a disease state or condition in a subject. Biologically active agents include, without limitation, organic molecules, inorganic materials, proteins, peptides, nucleic acids (e.g., genes, gene fragments, gene regulatory sequences, and antisense molecules), nucleoproteins, polysaccharides, glycoproteins, and lipoproteins. Classes of biologically active compounds that can be incorporated into the composition provided herein include, without limitation, anticancer agents, antibiotics, analgesics, anti-inflammatory agents, immunosuppressants, enzyme inhibitors, antihistamines, anti-convulsants, hormones, muscle relaxants, antispasmodics, ophthalmic agents, prostaglandins, anti-depressants, anti-psychotic substances, trophic factors, osteoinductive proteins, growth factors, and vaccines.

[0179] The term "active agent" may also be used herein to refer to a biological sample (e.g., a sample of tissue or fluid, such as for instance blood) or a component thereof, and/or to a biologically active entity or compound, and/or to a structurally or functionally labile entity.

[0180] Exemplary active agents include, but are not limited to, therapeutic agents, diagnostic agents (e.g., contrast agents), and any combinations thereof. In some embodiments, the active agent present in a silk matrix (e.g., a silk microsphere), composition, or the like can include a labile active agent, e.g., an agent that can undergo chemical, physical, or biological change, degradation and/or deactivation after exposure to a specified condition, e.g., high temperatures, high humidity, light exposure, and any combinations thereof. In some embodiments, the active agent present in the silk matrix (e.g., a silk microsphere), composition, or the like can include a temperature-sensitive active agent, e.g., an active agent that will lose at least about 30% or more, of its original activity or bioactivity, upon exposure to a temperature of at least about 10° C. or above, including at least about 15° C. or above, at least about room temperature or above, or at least about body temperature (e.g., about 37° C.) or above.

[0181] The active agent can be generally present in the silk matrix (e.g., a silk microsphere), composition, or the like in an amount of about 0.01% (w/w) to about 70% (w/w), or about 0.1% (w/w) to about 50% (w/w), or about 1% (w/w) to about 30% (w/w). The active agent can be present on a surface of the silk matrix (e.g., a silk microsphere), composition, or the like and/or encapsulated and dispersed in the silk matrix (e.g., a silk microsphere), composition, or the like homogeneously or heterogeneously or in a gradient. In some embodiments, the active agent can be added into the silk solution, which is then subjected to the methods described herein for preparing a silk matrix (e.g., a silk microsphere), composition, or the like. In some embodiments, the active agent can be coated on a surface of the silk matrix (e.g., a silk microsphere), composition, or the like. In some embodiments, the active agent can be loaded in a silk matrix (e.g., a silk microsphere), composition, or the like by

incubating the silk microsphere in a solution of the active agent for a period of time, during which an amount of the active agent can diffuse into the silk matrix (e.g., a silk microsphere), composition, or the like, and thus distribute within the silk matrix (e.g., a silk microsphere), composition, or the like.

[0182] In some aspects, the additive is a therapeutic agent. As used herein, the term "therapeutic agent" means a molecule, group of molecules, complex or substance administered to an organism for diagnostic, therapeutic, preventative medical, or veterinary purposes. As used herein, the term "therapeutic agent" includes a "drug" or a "vaccine." This term include externally and internally administered topical, localized and systemic human and animal pharmaceuticals, treatments, remedies, nutraceuticals, cosmeceuticals, biologicals, devices, diagnostics and contraceptives, including preparations useful in clinical and veterinary screening, prevention, prophylaxis, healing, wellness, detection, imaging, diagnosis, therapy, surgery, monitoring, cosmetics, prosthetics, forensics and the like. This term can also be used in reference to agriceutical, workplace, military, industrial and environmental therapeutics or remedies comprising selected molecules or selected nucleic acid sequences capable of recognizing cellular receptors, membrane receptors, hormone receptors, therapeutic receptors, microbes, viruses or selected targets comprising or capable of contacting plants, animals and/or humans. This term can also specifically include nucleic acids and compounds comprising nucleic acids that produce a therapeutic effect, for example deoxyribonucleic acid (DNA), ribonucleic acid (RNA), nucleic acid analogues (e.g., locked nucleic acid (LNA), peptide nucleic acid (PNA), xeno nucleic acid (XNA)), or mixtures or combinations thereof, including, for example, DNA nanoplexes, siRNA, microRNA, shRNA, aptamers, ribozymes, decoy nucleic acids, antisense nucleic acids, RNA activators, and the like. Generally, any therapeutic agent can be included in the composition provided herein.

[0183] The term "therapeutic agent" also includes an agent that is capable of providing a local or systemic biological, physiological, or therapeutic effect in the biological system to which it is applied. For example, the therapeutic agent can act to control infection or inflammation, enhance cell growth and tissue regeneration, control tumor growth, act as an analgesic, promote anti-cell attachment, and enhance bone growth, among other functions. Other suitable therapeutic agents can include anti-viral agents, hormones, antibodies, or therapeutic proteins. Other therapeutic agents include prodrugs, which are agents that are not biologically active when administered but, upon administration to a subject are converted to biologically active agents through metabolism or some other mechanism. Additionally, a silk-based drug delivery composition can contain one therapeutic agent or combinations of two or more therapeutic agents.

[0184] A therapeutic agent can include a wide variety of different compounds, including chemical compounds and mixtures of chemical compounds, e.g., small organic or inorganic molecules; saccharines; oligosaccharides; polysaccharides; biological macromolecules, e.g., peptides, proteins,

and peptide analogs and derivatives; peptidomimetics; antibodies and antigen binding fragments thereof; nucleic acids; nucleic acid analogs and derivatives; an extract made from biological materials such as bacteria, plants, fungi, or animal cells; animal tissues; naturally occurring or synthetic compositions; and any combinations thereof. In some aspects, the therapeutic agent is a small molecule.

[0185] The term "bioactivity," as used herein in reference to an active agent, generally refers to the ability of an active agent to interact with a biological target and/or to produce an effect on a biological target. For example, bioactivity can include, without limitation, elicitation of a stimulatory, inhibitory, regulatory, toxic or lethal response in a biological target. The biological target can be a molecule or a cell. For example, a bioactivity can refer to the ability of an active agent to modulate the effect/activity of an enzyme, block a receptor, stimulate a receptor, modulate the expression level of one or more genes, modulate cell proliferation, modulate cell division, modulate cell morphology, or any combination thereof. In some instances, a bioactivity can refer to the ability of a compound to produce a toxic effect in a cell. Exemplary cellular responses include, but are not limited to, lysis, apoptosis, growth inhibition, and growth promotion; production, secretion, and surface expression of a protein or other molecule of interest by the cell; membrane surface molecule activation including receptor activation; transmembrane ion transports; transcriptional regulations; changes in viability of the cell; changes in cell morphology; changes in presence or expression of an intracellular component of the cell; changes in gene expression or transcripts; changes in the activity of an enzyme produced within the cell; and changes in the presence or expression of a ligand and/or receptor (e.g., protein expression and/or binding activity). Methods for assaying different cellular responses are well known to one of skill in the art, e.g., western blot for determining changes in presence or expression of an endogenous protein of the cell, or microscopy for monitoring the cell morphology in response to the active agent, or FISH and/or qPCR for the detection and quantification of changes in nucleic acids. Bioactivity can be determined in some embodiments, for example, by assaying a cellular response.

[0186] In reference to an antibody, the term "bioactivity" includes, but is not limited to, epitope or antigen binding affinity, the in vivo and/or in vitro stability of the antibody, the immunogenic properties of the antibody, e.g., when administered to a human subject, and/or the ability to neutralize or antagonize the bioactivity of a target molecule in vivo or in vitro. The aforementioned properties or characteristics can be observed or measured using art-recognized techniques including, but not limited to, scintillation proximity assays, ELISA, ORIGEN immunoassay (IGEN), fluorescence quenching, fluorescence ELISA, competitive ELISA, SPR analysis including, but not limited to, SPR analysis using a BIAcore biosensor, *in vitro* and *in vivo* neutralization assays (see, for example, International Publication No. WO 2006/062685), receptor binding, and

immunohistochemistry with tissue sections from different sources including human, primate, or any other source as needed. In reference to an immunogen, the "bioactivity" includes immunogenicity, the definition of which is discussed in detail later. In reference to a virus, the "bioactivity" includes infectivity, the definition of which is discussed in detail later. In reference to a contrast agent, e.g., a dye, the "bioactivity" refers to the ability of a contrast agent when administered to a subject to enhance the contrast of structures or fluids within the subject's body. The bioactivity of a contrast agent also includes, but is not limited to, its ability to interact with a biological environment and/or influence the response of another molecule under certain conditions.

[0187] As used herein, the term "small molecule" can refer to compounds that are "natural product-like," however, the term "small molecule" is not limited to "natural product-like" compounds. Rather, a small molecule is typically characterized in that it contains several carbon—carbon bonds, and has a molecular weight of less than 5000 Daltons (5 kDa), preferably less than 3 kDa, still more preferably less than 2 kDa, and most preferably less than 1 kDa. In some cases it is preferred that a small molecule have a molecular weight equal to or less than 700 Daltons.

[0188] Exemplary therapeutic agents include, but are not limited to, those found in Harrison's Principles of Internal Medicine, 13th Edition, Eds. T.R. Harrison et al. McGraw-Hill N.Y., NY; Physicians' Desk Reference, 50th Edition, 1997, Oradell New Jersey, Medical Economics Co.; Pharmacological Basis of Therapeutics, 8th Edition, Goodman and Gilman, 1990; United States Pharmacopeia, The National Formulary, ETSP XII NF XVII, 1990, the complete contents of all of which are incorporated herein by reference.

[0189] Therapeutic agents include the herein disclosed categories and specific examples. It is not intended that the category be limited by the specific examples. Those of ordinary skill in the art will recognize also numerous other compounds that fall within the categories and that are useful according to the present disclosure. Examples include a radiosensitizer, a steroid, a xanthine, a beta-2-agonist bronchodilator, an anti-inflammatory agent, an analgesic agent, a calcium antagonist, an angiotensin-converting enzyme inhibitors, a beta-blocker, a centrally active alpha- agonist, an alpha-1 -antagonist, an anticholinergic/antispasmodic agent, a vasopressin analogue, an anti arrhythmic agent, an antiparkinsonian agent, an antiangina/antihypertensive agent, an anticoagulant agent, an antiplatelet agent, a sedative, an ansiolytic agent, a peptidic agent, a biopolymeric agent, an antineoplastic agent, a laxative, an antidiarrheal agent, an antimicrobial agent, an antifungal agent, a vaccine, a protein, or a nucleic acid. In a further aspect, the pharmaceutically active agent can be coumarin, albumin, steroids such as betamethasone, dexamethasone, methylprednisolone, prednisolone, prednisone, triamcinolone, budesonide, hydrocortisone, and pharmaceutically acceptable hydrocortisone derivatives; xanthines such as theophylline and doxophylline; beta-2-agonist bronchodilators such as salbutamol, fenterol, clenbuterol, bambuterol, salmeterol, fenoterol;

antiinflammatory agents, including antiasthmatic anti-inflammatory agents, antiarthritis antiinflammatory agents, and non-steroidal antiinflammatory agents, examples of which include but are not limited to sulfides, mesalamine, budesonide, salazopyrin, diclofenac, pharmaceutically acceptable diclofenac salts, nimesulide, naproxene, acetaminophen, ibuprofen, ketoprofen and piroxicam; analgesic agents such as salicylates; calcium channel blockers such as nifedipine, amlodipine, and nicardipine; angiotensin converting enzyme inhibitors such as captopril, benazepril hydrochloride, fosinopril sodium, trandolapril, ramipril, lisinopril, enalapril, quinapril hydrochloride, and moexipril hydrochloride; beta-blockers (i.e., beta adrenergic blocking agents) such as sotalol hydrochloride, timolol maleate, esmolol hydrochloride, carteolol, propanolol hydrochloride, betaxolol hydrochloride, penbutolol sulfate, metoprolol tartrate, metoprolol succinate, acebutolol hydrochloride, atenolol, pindolol, and bisoprolol fumarate; centrally active alpha-2-agonists such as clonidine; alpha- 1 -antagonists such as doxazosin and prazosin; anticholinergic/antispasmodic agents such as dicyclomine hydrochloride, scopolamine hydrobromide, glycopyrrolate, clidinium bromide, flavoxate, and oxybutynin; vasopressin analogues such as vasopressin and desmopressin; antiarrhythmic agents such as quinidine, lidocaine, tocainide hydrochloride, mexiletine hydrochloride, digoxin, verapamil hydrochloride, propafenone hydrochloride, flecainide acetate, procainamide hydrochloride, moricizine hydrochloride, and disopyramide phosphate; antiparkinsonian agents, such as dopamine, L-Dopa/Carbidopa, selegiline, dihydroergocryptine, pergolide, lisuride, apomorphine, and bromocryptine; antiangina agents and antihypertensive agents such as isosorbide mononitrate, isosorbide dinitrate, propranolol, atenolol and verapamil; anticoagulant and antiplatelet agents such as Coumadin, warfarin, acetylsalicylic acid, and ticlopidine; sedatives such as benzodiazapines and barbiturates; ansiolytic agents such as lorazepam, bromazepam, and diazepam; peptidic and biopolymeric agents such as calcitonin, leuprolide and other LHRH agonists, hirudin, cyclosporin, insulin, somatostatin, protirelin, interferon, desmopressin, somatotropin, thymopentin, pidotimod, erythropoietin, interleukins, melatonin, granulocyte/macrophage-CSF, and heparin; antineoplastic agents such as etoposide, etoposide phosphate, cyclophosphamide, methotrexate, 5-fluorouracil, vincristine, doxorubicin, cisplatin, hydroxyurea, leucovorin calcium, tamoxifen, flutamide, asparaginase, altretamine, mitotane, and procarbazine hydrochloride; laxatives such as senna concentrate, casanthranol, bisacodyl, and sodium picosulphate; antidiarrheal agents such as difenoxine hydrochloride, loperamide hydrochloride, furazolidone, diphenoxylate hdyrochloride, and microorganisms; vaccines such as bacterial and viral vaccines; antimicrobial agents such as penicillins, cephalosporins, and macrolides, antifungal agents such as imidazolic and triazolic derivatives; and nucleic acids such as DNA sequences encoding for biological proteins, and antisense oligonucleotides.

[0190] Anti-cancer agents include alkylating agents, platinum agents, antimetabolites, topoisomerase inhibitors, antitumor antibiotics, antimitotic agents, aromatase inhibitors, thymidylate synthase inhibitors, DNA antagonists, farnesyltransferase inhibitors, pump inhibitors, histone acetyltransferase inhibitors, metalloproteinase inhibitors, ribonucleoside reductase inhibitors, TNF alpha agonists/antagonists, endothelinA receptor antagonists, retinoic acid receptor agonists, immuno-modulators, hormonal and antihormonal agents, photodynamic agents, and tyrosine kinase inhibitors.

[0191] Antibiotics include aminoglycosides (e.g., gentamicin, tobramycin, netilmicin, streptomycin, amikacin, neomycin), bacitracin, corbapenems (e.g., imipenem/cislastatin), cephalosporins, colistin, methenamine, monobactams (e.g., aztreonam), penicillins (e.g., penicillin G, penicillinV, methicillin, natcillin, oxacillin, cloxacillin, dicloxacillin, ampicillin, amoxicillin, carbenicillin, ticarcillin, piperacillin, mezlocillin, azlocillin), polymyxin B, quinolones, and vancomycin; and bacteriostatic agents such as chloramphenicol, clindanyan, macrolides (e.g., erythromycin, azithromycin, clarithromycin), lincomyan, nitrofurantoin, sulfonamides, tetracyclines (e.g., tetracycline, doxycycline, minocycline, demeclocyline), and trimethoprim. Also included are metronidazole, fluoroquinolones, and ritampin.

[0192] Enzyme inhibitors are substances which inhibit an enzymatic reaction. Examples of enzyme inhibitors include edrophonium chloride, N-methylphysostigmine, neostigmine bromide, physostigmine sulfate, tacrine, tacrine, 1 -hydroxy maleate, iodotubercidin, p- bromotetramiisole, 10-(alpha-diethylaminopropionyl)-phenothiazine hydrochloride, calmidazolium chloride, hemicholinium-3,3,5-dinitrocatechol, diacylglycerol kinase inhibitor I, diacylglycerol kinase inhibitor II, 3-phenylpropargylamine, N°-monomethyl-Larginine acetate, carbidopa, 3-hydroxybenzylhydrazine, hydralazine, clorgyline, deprenyl, hydroxylamine, iproniazid phosphate, 6-MeO-tetrahydro-9H-pyrido-indole, nialamide, pargyline, quinacrine, semi carb azide, tranylcypromine, N,N-diethylaminoethyl-2,2-diphenylvalerate hydrochloride, 3 - isobutyl- l-methylxanthne, papaverine, indomethacind, 2-cyclooctyl-2 -hydroxy ethylamine hydrochloride, 2,3-dichloro-a-methylbenzylamine (DCMB), 8,9-dichloro-2,3,4, 5 -tetrahydro- lH-2-benzazepine hydrochloride, p-amino glutethimide, p-aminoglutethimide tartrate, 3- iodotyrosine, alpha-methyltyrosine, acetazolamide, dichlorphenamide, 6-hydroxy-2- benzothiazolesulfonamide, and allopurinol.

[0193] Antihistamines include pyrilamine, chlorpheniramine, and tetrahydrazoline, among others.

[0194] Anti-inflammatory agents include corticosteroids, nonsteroidal anti-inflammatory drugs (e.g., aspirin, phenylbutazone, indomethacin, sulindac, tolmetin, ibuprofen, piroxicam, and fenamates), acetaminophen, phenacetin, gold salts, chloroquine, D-Penicillamine, methotrexate colchicine, allopurinol, probenecid, and sulfinpyrazone.

[0195] Muscle relaxants include mephenesin, methocarbomal, cyclobenzaprine hydrochloride, trihexylphenidyl hydrochloride, levodopa/carbidopa, and biperiden.

[0196] Anti-spasmodics include atropine, scopolamine, oxyphenonium, and papaverine.

[0197] Analgesics include aspirin, phenybutazone, idomethacin, sulindac, tolmetic, ibuprofen, piroxicam, fenamates, acetaminophen, phenacetin, morphine sulfate, codeine sulfate, meperidine, nalorphine, opioids (e.g., codeine sulfate, fentanyl citrate, hydrocodone bitartrate, loperamide, morphine sulfate, noscapine, norcodeine, normorphine, thebaine, nor- binaltorphimine, buprenorphine, chlomaltrexamine, funaltrexamione, nalbuphine, nalorphine, naloxone, naloxonazine, naltrexone, and naltrindole), procaine, lidocain, tetracaine and dibucaine. Ophthalmic agents include sodium fluorescein, rose bengal, methacholine, adrenaline, cocaine, atropine, alpha-chymotrypsin, hyaluronidase, betaxalol, pilocarpine, timolol, timolol salts, and combinations thereof.

[0198] Prostaglandins are art recognized and are a class of naturally occurring chemically related long-chain hydroxy fatty acids that have a variety of biological effects.

[0199] Anti-depressants are substances capable of preventing or relieving depression.

[0200] Examples of anti-depressants include imipramine, amitriptyline, nortriptyline, protriptyline, desipramine, amoxapine, doxepin, maprotiline, tranylcypromine, phenelzine, and isocarboxazide.

[0201] Trophic factors are factors whose continued presence improves the viability or longevity of a cell trophic factors include, without limitation, platelet-derived growth factor (PDGP), neutrophil-activating protein, monocyte chemoattractant protein, macrophage- inflammatory protein, platelet factor, platelet basic protein, and melanoma growth stimulating activity; epidermal growth factor, transforming growth factor (alpha), fibroblast growth factor, platelet- derived endothelial cell growth factor, insulin-like growth factor, glial derived growth neurotrophic factor, ciliary neurotrophic factor, nerve growth factor, bone growth/cartilage- inducing factor (alpha and beta), bone morphogenetic proteins, interleukins (e.g., interleukin inhibitors or interleukin receptors, including interleukin 1 through interleukin 10), interferons (e.g., interferon alpha, beta and gamma), hematopoietic factors, including erythropoietin, granulocyte colony stimulating factor, macrophage colony stimulating factor and granulocyte- macrophage colony stimulating factor; tumor necrosis factors, and transforming growth factors (beta), including beta-l, beta-2, beta-3, inhibin, and activin.

[0202] Hormones include estrogens (e.g., estradiol, estrone, estriol, diethylstibestrol, quinestrol, chlorotrianisene, ethinyl estradiol, mestranol), anti-estrogens (e.g., clomiphene, tamoxifen), progestins (e.g., medroxyprogesterone, norethindrone, hydroxyprogesterone, norgestrel), antiprogestin (mifepristone), androgens (e.g, testosterone cypionate, fluoxymesterone, danazol, testolactone), anti-androgens (e.g., cyproterone acetate, flutamide), thyroid hormones (e.g., triiodothyronne, thyroxine, propylthiouracil, methimazole, and iodixode), and pituitary hormones (e.g., corticotropin, sumutotropin, oxytocin, and vasopressin). Hormones are commonly employed in

hormone replacement therapy and / or for purposes of birth control. Steroid hormones, such as prednisone, are also used as immunosuppressants and anti-inflammatories. In some aspects, the additive is an agent that stimulates tissue formation, and/or healing and regrowth of natural tissues, and any combinations thereof. Agents that increase formation of new tissues and/or stimulates healing or regrowth of native tissue at the site of injection can include, but are not limited to, fibroblast growth factor (FGF), transforming growth factor-beta (TGF-beta, platelet-derived growth factor (PDGF), epidermal growth factors (EGFs), connective tissue activated peptides (CTAPs), osteogenic factors including bone morphogenic proteins, heparin, angiotensin II (A-II) and fragments thereof, insulin-like growth factors, tumor necrosis factors, interleukins, colony stimulating factors, erythropoietin, nerve growth factors, interferons, biologically active analogs, fragments, and derivatives of such growth factors, and any combinations thereof.

[0203] In some aspects, the silk composition can further comprise at least one additional material for soft tissue augmentation, e.g., dermal filler materials, including, but not limited to, poly(methyl methacrylate) microspheres, hydroxylapatite, poly(L-lactic acid), collagen, elastin, and glycosaminoglycans, hyaluronic acid, commercial dermal filler products such as BOTOX® (from Allergan), DYSPORT®, COSMODERM®, EVOLENCE®, RADIESSE®,RESTYLANE®, JUVEDERM® (from Allergan), SCULPTRA®, PERLANE®, and CAPTIQEIE®, and any combinations thereof.

[0204] In some aspects, the additive is a wound healing agent. As used herein, a “wound healing agent" is a compound or composition that actively promotes wound healing process.

[0205] Exemplary wound healing agents include, but are not limited to dexpanthenol; growth factors; enzymes, hormones; povidon-iodide; fatty acids; anti-inflammatory agents; antibiotics; antimicrobials; antiseptics; cytokines; thrombin; angalgesics; opioids; aminoxyls; furoxans; nitrosothiols; nitrates and anthocyanins; nucleosides, such as adenosine; and nucleotides, such as adenosine diphosphate (ADP) and adenosine triphosphate (ATP); neutotransmitter/neuromodulators, such as acetylcholine and 5-hydroxytryptamine (serotonin/5- HT); histamine and catecholamines, such as adrenalin and noradrenalin; lipid molecules, such as 5 sphingosine-1-phosphate and lysophosphatidic acid; amino acids, such as arginine and lysine; peptides such as the bradykinins, substance P and calcium gene-related peptide (CGRP); nitric oxide; and any combinations thereof.

[0206] In certain aspects, the active agents provided herein are immunogens. In one aspect, the immunogen is a vaccine. Most vaccines are sensitive to environmental conditions under which they are stored and/or transported. For example, freezing may increase reactogenicity (e.g., capability of causing an immunological reaction) and/or loss of potency for some vaccines (e.g., HepB, and DTaP/IPV/FQB), or cause hairline cracks in the container, leading to contamination. Further, some vaccines (e.g., BCG, Varicella, and MMR) are sensitive to heat. Many vaccines (e.g., BCG, MMR,

Varicella, Meningococcal C Conjugate, and most DTaP-containing vaccines) are light sensitive. See, e.g., Galazka et al., Thermostability of vaccines, in Global Programme for Vaccines & Immunization (World Health Organization, Geneva, 1998); Peetermans et al., Stability of freeze-dried rubella virus vaccine (Cendehill strain) at various temperatures, 1 J. Biological Standardization 179 (1973). Thus, the compositions and methods provided herein also provide for stabilization of vaccines regardless of the cold chain and/or other environmental conditions.

[0207] In some aspects, the additive is a cell, e.g., a biological cell. Cells useful for incorporation into the composition can come from any source, e.g., mammalian, insect, plant, etc. In some aspects, the cell can be a human, rat or mouse cell. In general, cells to be used with the compositions provided herein can be any types of cells. In general, the cells should be viable when encapsulated within compositions. In some aspects, cells that can be used with the composition include, but are not limited to, mammalian cells (e.g. human cells, primate cells, mammalian cells, rodent cells, etc.), avian cells, fish cells, insect cells, plant cells, fungal cells, spore cells, bacterial cells, and hybrid cells. In some aspects, exemplary cells that can be can be used with the compositions include platelets, activated platelets, stem cells, totipotent cells, pluripotent cells, and/or embryonic stem cells. In some aspects, exemplary cells that can be encapsulated within compositions include, but are not limited to, primary cells and/or cell lines from any tissue. For example, cardiomyocytes, myocytes, hepatocytes, keratinocytes, melanocytes, neurons, astrocytes, embryonic stem cells, adult stem cells, hematopoietic stem cells, hematopoietic cells (e.g. monocytes, neutrophils, macrophages, etc.), ameloblasts, fibroblasts, chondrocytes, osteoblasts, osteoclasts, neurons, sperm cells, egg cells, liver cells, epithelial cells from lung, epithelial cells from gut, epithelial cells from intestine, liver, epithelial cells from skin, etc., and/or hybrids thereof, can be included in the silk/platelet compositions disclosed herein. Those skilled in the art will recognize that the cells listed herein represent an exemplary, not comprehensive, list of cells. Cells can be obtained from donors (allogenic) or from recipients (autologous). Cells can be obtained, as a non-limiting example, by biopsy or other surgical means known to those skilled in the art.

[0208] In some aspects, the cell can be a genetically modified cell. A cell can be genetically modified to express and secrete a desired compound, e.g. a bioactive agent, a growth factor, differentiation factor, cytokines, and the like. Methods of genetically modifying cells for expressing and secreting compounds of interest are known in the art and easily adaptable by one of skill in the art.

[0209] Differentiated cells that have been reprogrammed into stem cells can also be used.

[0210] For example, human skin cells reprogrammed into embryonic stem cells by the transduction of Oct3/4, Sox2, c-Myc and Klf4 (Junying Yu, et. ah, Science, 2007, 318 , 1917-1920 and Takahashi K. et. ah, Cell, 2007, 131 , 1-12).

[0211] Unless otherwise specified or indicated by context, the terms "a", "an", and "the" mean "one or more." For example, "a molecule" should be interpreted to mean "one or more molecules."

[0212] As used herein, "about", "approximately," "substantially," and "significantly" will be understood by persons of ordinary skill in the art and will vary to some extent on the context in which they are used. If there are uses of the term which are not clear to persons of ordinary skill in the art given the context in which it is used, "about" and "approximately" will mean plus or minus ≤10% of the particular term and "substantially" and "significantly" will mean plus or minus >10% of the particular term.

[0213] As used herein, the terms "include" and "including" have the same meaning as the terms "comprise" and "comprising." The terms "comprise" and "comprising" should be interpreted as being "open" transitional terms that permit the inclusion of additional components further to those components recited in the claims. The terms "consist" and "consisting of" should be interpreted as being "closed" transitional terms that do not permit the inclusion of additional components other than the components recited in the claims. The term "consisting essentially of" should be interpreted to be partially closed and allowing the inclusion only of additional components that do not fundamentally alter the nature of the claimed subject matter.

[0214] All methods described herein can be performed in any suitable order unless otherwise indicated herein or otherwise clearly contradicted by context. The use of any and all examples, or exemplary language (e.g., "such as") provided herein, is intended merely to better illuminate the invention and does not pose a limitation on the scope of the invention unless otherwise claimed. No language in the specification should be construed as indicating any non-claimed element as essential to the practice of the invention.

[0215] All references, including publications, patent applications, and patents, cited herein are hereby incorporated by reference to the same extent as if each reference were individually and specifically indicated to be incorporated by reference and were set forth in its entirety herein.

[0216] Preferred aspects of this invention are described herein, including the best mode known to the inventors for carrying out the invention. Variations of those preferred aspects may become apparent to those of ordinary skill in the art upon reading the foregoing description. The inventors expect a person having ordinary skill in the art to employ such variations as appropriate, and the inventors intend for the invention to be practiced otherwise than as specifically described herein. Accordingly, this invention includes all modifications and equivalents of the subject matter recited in the claims appended hereto as permitted by applicable law. Moreover, any combination of the above-described elements in all possible variations thereof is encompassed by the invention unless otherwise indicated herein or otherwise clearly contradicted by context.

[0217] The methods and systems described herein may be deployed in part or in whole through a machine having a computer, computing device, processor, circuit, and/or server that executes computer readable instructions, program codes, instructions, and/or includes hardware configured to functionally execute one or more operations of the methods and systems disclosed herein. The terms computer, computing device, processor, circuit, and/or server, as utilized herein, should be understood broadly.

[0218] Any one or more of the terms computer, computing device, processor, circuit, and/or server include a computer of any type, capable to access instructions stored in communication thereto such as upon a non-transient computer readable medium, whereupon the computer performs operations of systems or methods described herein upon executing the instructions. In certain embodiments, such instructions themselves comprise a computer, computing device, processor, circuit, and/or server. Additionally or alternatively, a computer, computing device, processor, circuit, and/or server may be a separate hardware device, one or more computing resources distributed across hardware devices, and/or may include such aspects as logical circuits, embedded circuits, sensors, actuators, input and/or output devices, network and/or communication resources, memory resources of any type, processing resources of any type, and/or hardware devices configured to be responsive to determined conditions to functionally execute one or more operations of systems and methods herein.

[0219] Network and/or communication resources include, without limitation, local area network, wide area network, wireless, internet, or any other known communication resources and protocols. Example and non-limiting hardware, computers, computing devices, processors, circuits, and/or servers include, without limitation, a general purpose computer, a server, an embedded computer, a mobile device, a virtual machine, and/or an emulated version of one or more of these. Example and non-limiting hardware, computers, computing devices, processors, circuits, and/or servers may be physical, logical, or virtual. A computer, computing device, processor, circuit, and/or server may be: a distributed resource included as an aspect of several devices; and/or included as an interoperable set of resources to perform described functions of the computer, computing device, processor, circuit, and/or server, such that the distributed resources function together to perform the operations of the computer, computing device, processor, circuit, and/or server. In certain embodiments, each computer, computing device, processor, circuit, and/or server may be on separate hardware, and/or one or more hardware devices may include aspects of more than one computer, computing device, processor, circuit, and/or server, for example as separately executable instructions stored on the hardware device, and/or as logically partitioned aspects of a set of executable instructions, with some aspects of the hardware device comprising a part of a first computer, computing device, processor, circuit, and/or server, and some aspects of the hardware device comprising a part of a second computer, computing device, processor, circuit, and/or server.

[0220] A computer, computing device, processor, circuit, and/or server may be part of a server, client, network infrastructure, mobile computing platform, stationary computing platform, or other computing platform. A processor may be any kind of computational or processing device capable of executing program instructions, codes, binary instructions and the like. The processor may be or include a signal processor, digital processor, embedded processor, microprocessor or any variant such as a co-processor (math co-processor, graphic co-processor, communication co-processor and the like) and the like that may directly or indirectly facilitate execution of program code or program instructions stored thereon. In addition, the processor may enable execution of multiple programs, threads, and codes. The threads may be executed simultaneously to enhance the performance of the processor and to facilitate simultaneous operations of the application. By way of implementation, methods, program codes, program instructions and the like described herein may be implemented in one or more threads. The thread may spawn other threads that may have assigned priorities associated with them; the processor may execute these threads based on priority or any other order based on instructions provided in the program code. The processor may include memory that stores methods, codes, instructions and programs as described herein and elsewhere. The processor may access a storage medium through an interface that may store methods, codes, and instructions as described herein and elsewhere. The storage medium associated with the processor for storing methods, programs, codes, program instructions or other type of instructions capable of being executed by the computing or processing device may include but may not be limited to one or more of a CD-ROM, DVD, memory, hard disk, flash drive, RAM, ROM, cache and the like.

[0221] A processor may include one or more cores that may enhance speed and performance of a multiprocessor. In embodiments, the process may be a dual core processor, quad core processors, other chip-level multiprocessor and the like that combine two or more independent cores (called a die).

[0222] The methods and systems described herein may be deployed in part or in whole through a machine that executes computer readable instructions on a server, client, firewall, gateway, hub, router, or other such computer and/or networking hardware. The computer readable instructions may be associated with a server that may include a file server, print server, domain server, internet server, intranet server and other variants such as secondary server, host server, distributed server and the like. The server may include one or more of memories, processors, computer readable transitory and/or non-transitory media, storage media, ports (physical and virtual), communication devices, and interfaces capable of accessing other servers, clients, machines, and devices through a wired or a wireless medium, and the like. The methods, programs, or codes as described herein and elsewhere may be executed by the server. In addition, other devices required for execution of methods as

described in this application may be considered as a part of the infrastructure associated with the server.

[0223] The server may provide an interface to other devices including, without limitation, clients, other servers, printers, database servers, print servers, file servers, communication servers, distributed servers, and the like. Additionally, this coupling and/or connection may facilitate remote execution of instructions across the network. The networking of some or all of these devices may facilitate parallel processing of program code, instructions, and/or programs at one or more locations without deviating from the scope of the disclosure. In addition, all the devices attached to the server through an interface may include at least one storage medium capable of storing methods, program code, instructions, and/or programs. A central repository may provide program instructions to be executed on different devices. In this implementation, the remote repository may act as a storage medium for methods, program code, instructions, and/or programs.

[0224] The methods, program code, instructions, and/or programs may be associated with a client that may include a file client, print client, domain client, internet client, intranet client and other variants such as secondary client, host client, distributed client and the like. The client may include one or more of memories, processors, computer readable transitory and/or non-transitory media, storage media, ports (physical and virtual), communication devices, and interfaces capable of accessing other clients, servers, machines, and devices through a wired or a wireless medium, and the like. The methods, program code, instructions, and/or programs as described herein and elsewhere may be executed by the client. In addition, other devices utilized for execution of methods as described in this application may be considered as a part of the infrastructure associated with the client.

[0225] The client may provide an interface to other devices including, without limitation, servers, other clients, printers, database servers, print servers, file servers, communication servers, distributed servers, and the like. Additionally, this coupling and/or connection may facilitate remote execution of methods, program code, instructions, and/or programs across the network. The networking of some or all of these devices may facilitate parallel processing of methods, program code, instructions, and/or programs at one or more locations without deviating from the scope of the disclosure. In addition, all the devices attached to the client through an interface may include at least one storage medium capable of storing methods, program code, instructions, and/or programs. A central repository may provide program instructions to be executed on different devices. In this implementation, the remote repository may act as a storage medium for methods, program code, instructions, and/or programs.

[0226] The methods and systems described herein may be deployed in part or in whole through network infrastructures. The network infrastructure may include elements such as computing

devices, servers, routers, hubs, firewalls, clients, personal computers, communication devices, routing devices and other active and passive devices, modules, and/or components as known in the art. The computing and/or non-computing device(s) associated with the network infrastructure may include, apart from other components, a storage medium such as flash memory, buffer, stack, RAM, ROM and the like. The methods, program code, instructions, and/or programs described herein and elsewhere may be executed by one or more of the network infrastructural elements.

[0227] The methods, program code, instructions, and/or programs described herein and elsewhere may be implemented on a cellular network having multiple cells. The cellular network may either be frequency division multiple access (FDMA) network or code division multiple access (CDMA) network. The cellular network may include mobile devices, cell sites, base stations, repeaters, antennas, towers, and the like.

[0228] The methods, program code, instructions, and/or programs described herein and elsewhere may be implemented on or through mobile devices. The mobile devices may include navigation devices, cell phones, mobile phones, mobile personal digital assistants, laptops, palmtops, netbooks, pagers, electronic books readers, music players, and the like. These mobile devices may include, apart from other components, a storage medium such as a flash memory, buffer, RAM, ROM and one or more computing devices. The computing devices associated with mobile devices may be enabled to execute methods, program code, instructions, and/or programs stored thereon. Alternatively, the mobile devices may be configured to execute instructions in collaboration with other devices. The mobile devices may communicate with base stations interfaced with servers and configured to execute methods, program code, instructions, and/or programs. The mobile devices may communicate on a peer to peer network, mesh network, or other communications network. The methods, program code, instructions, and/or programs may be stored on the storage medium associated with the server and executed by a computing device embedded within the server. The base station may include a computing device and a storage medium. The storage device may store methods, program code, instructions, and/or programs executed by the computing devices associated with the base station.

[0229] The methods, program code, instructions, and/or programs may be stored and/or accessed on machine readable transitory and/or non-transitory media that may include: computer components, devices, and recording media that retain digital data used for computing for some interval of time; semiconductor storage known as random access memory (RAM); mass storage typically for more permanent storage, such as optical discs, forms of magnetic storage like hard disks, tapes, drums, cards and other types; processor registers, cache memory, volatile memory, non-volatile memory; optical storage such as CD, DVD; removable media such as flash memory (e.g., USB sticks or keys), floppy disks, magnetic tape, paper tape, punch cards, standalone RAM disks, Zip drives, removable

mass storage, off-line, and the like; other computer memory such as dynamic memory, static memory, read/write storage, mutable storage, read only, random access, sequential access, location addressable, file addressable, content addressable, network attached storage, storage area network, bar codes, magnetic ink, and the like.

[0230] Certain operations described herein include interpreting, receiving, and/or determining one or more values, parameters, inputs, data, or other information. Operations including interpreting, receiving, and/or determining any value parameter, input, data, and/or other information include, without limitation: receiving data via a user input; receiving data over a network of any type; reading a data value from a memory location in communication with the receiving device; utilizing a default value as a received data value; estimating, calculating, or deriving a data value based on other information available to the receiving device; and/or updating any of these in response to a later received data value. In certain embodiments, a data value may be received by a first operation, and later updated by a second operation, as part of the receiving a data value. For example, when communications are down, intermittent, or interrupted, a first operation to interpret, receive, and/or determine a data value may be performed, and when communications are restored an updated operation to interpret, receive, and/or determine the data value may be performed.

[0231] Certain logical groupings of operations herein, for example methods or procedures of the current disclosure, are provided to illustrate aspects of the present disclosure. Operations described herein are schematically described and/or depicted, and operations may be combined, divided, re-ordered, added, or removed in a manner consistent with the disclosure herein. It is understood that the context of an operational description may require an ordering for one or more operations, and/or an order for one or more operations may be explicitly disclosed, but the order of operations should be understood broadly, where any equivalent grouping of operations to provide an equivalent outcome of operations is specifically contemplated herein. For example, if a value is used in one operational step, the determining of the value may be required before that operational step in certain contexts (e.g. where the time delay of data for an operation to achieve a certain effect is important), but may not be required before that operation step in other contexts (e.g. where usage of the value from a previous execution cycle of the operations would be sufficient for those purposes). Accordingly, in certain embodiments an order of operations and grouping of operations as described is explicitly contemplated herein, and in certain embodiments re-ordering, subdivision, and/or different grouping of operations is explicitly contemplated herein.

[0232] The methods and systems described herein may transform physical and/or or intangible items from one state to another. The methods and systems described herein may also transform data representing physical and/or intangible items from one state to another.

[0233] The elements described and depicted herein, including in flow charts, block diagrams, and/or operational descriptions, depict and/or describe specific example arrangements of elements for purposes of illustration. However, the depicted and/or described elements, the functions thereof, and/or arrangements of these, may be implemented on machines, such as through computer executable transitory and/or non-transitory media having a processor capable of executing program instructions stored thereon, and/or as logical circuits or hardware arrangements. Example arrangements of programming instructions include at least: monolithic structure of instructions; standalone modules of instructions for elements or portions thereof; and/or as modules of instructions that employ external routines, code, services, and so forth; and/or any combination of these, and all such implementations are contemplated to be within the scope of embodiments of the present disclosure Examples of such machines include, without limitation, personal digital assistants, laptops, personal computers, mobile phones, other handheld computing devices, medical equipment, wired or wireless communication devices, transducers, chips, calculators, satellites, tablet PCs, electronic books, gadgets, electronic devices, devices having artificial intelligence, computing devices, networking equipment, servers, routers and the like. Furthermore, the elements described and/or depicted herein, and/or any other logical components, may be implemented on a machine capable of executing program instructions. Thus, while the foregoing flow charts, block diagrams, and/or operational descriptions set forth functional aspects of the disclosed systems, any arrangement of program instructions implementing these functional aspects are contemplated herein. Similarly, it will be appreciated that the various steps identified and described above may be varied, and that the order of steps may be adapted to particular applications of the techniques disclosed herein. Additionally, any steps or operations may be divided and/or combined in any manner providing similar functionality to the described operations. All such variations and modifications are contemplated in the present disclosure. The methods and/or processes described above, and steps thereof, may be implemented in hardware, program code, instructions, and/or programs or any combination of hardware and methods, program code, instructions, and/or programs suitable for a particular application. Example hardware includes a dedicated computing device or specific computing device, a particular aspect or component of a specific computing device, and/or an arrangement of hardware components and/or logical circuits to perform one or more of the operations of a method and/or system. The processes may be implemented in one or more microprocessors, microcontrollers, embedded microcontrollers, programmable digital signal processors or other programmable device, along with internal and/or external memory. The processes may also, or instead, be embodied in an application specific integrated circuit, a programmable gate array, programmable array logic, or any other device or combination of devices that may be configured to process electronic signals. It will further be appreciated that one or more of the

processes may be realized as a computer executable code capable of being executed on a machine readable medium.

[0234] The computer executable code may be created using a structured programming language such as C, an object oriented programming language such as C++, or any other high-level or low-level programming language (including assembly languages, hardware description languages, and database programming languages and technologies) that may be stored, compiled or interpreted to run on one of the above devices, as well as heterogeneous combinations of processors, processor architectures, or combinations of different hardware and computer readable instructions, or any other machine capable of executing program instructions.

[0235] Thus, in one aspect, each method described above and combinations thereof may be embodied in computer executable code that, when executing on one or more computing devices, performs the steps thereof. In another aspect, the methods may be embodied in systems that perform the steps thereof, and may be distributed across devices in a number of ways, or all of the functionality may be integrated into a dedicated, standalone device or other hardware. In another aspect, the means for performing the steps associated with the processes described above may include any of the hardware and/or computer-readable instructions described above. All such permutations and combinations are contemplated in embodiments of the present disclosure.

[0236] While the invention has been illustrated and described in detail in the foregoing drawings and description, the same is to be considered as illustrative and not restrictive in character, it being understood that only illustrative embodiments thereof have been shown and described and that all changes and modifications that come within the spirit of the invention are desired to be protected. For example, any of the features or functions of any of the embodiments disclosed herein may be incorporated into any of the other embodiments disclosed herein.

[0237] The following examples illustrate some embodiments and aspects of the invention. It will be apparent to those skilled in the relevant art that various modifications, additions, substitutions, and the like can be performed without altering the spirit or scope of the invention, and such modifications and variations are encompassed within the scope of the invention as defined in the claims which follow. The following examples do not in any way limit the invention.

[0238] EXAMPLES

[0239] Example 1

[0240] In one example, textiles/textile waste are segregated into different color categories before conducting the extraction process. In some examples, silk fibroin extracted from a combination of white fabrics results in a light-yellow aqueous solution comparable to silk fibroin solution extracted from Bombyx Mori cocoons. Colored textiles/textile waste originate from various sources making tracking or identifying and separating the chemical molecules used to dye each fabric from the silk

fibers an arduous task. Hence, this Example focuses on recycling the chemical dye bonded to the silk fibers into a colored silk fibroin solution. This silk fibroin solution offers versatility in terms of fabricating films, solids, pigment, or the like. The following steps are to be carried out with fabric groups of the same color (See D. N. Rockwood, R. C. Preda, T. Yücel, X. Wang, M. L. Lovett and D. L. Kaplan, "Materials fabrication from Bombyx mori silk fibroin," Nature Protocols, vol. 6, no. 10, pp. 1612-1631, 2011, which is incorporated herein in its entirety by reference for all purposes):

[0241] 1. Pack silk-based fabric/fabric scraps into a glass beaker with 5 times (by weight) the amount of 9.3M solution of lithium bromide in water (LiBr) to dissolve the silk-based textiles.

[0242] 2. Over the course of a few days, 100% colored liquid solution may be found in the container if fabrics were 100% silk. If the fabrics were silk blend, suspension of foreign fibers such as cotton and polyester may be found.

[0243] 3. Fill a large plastic beaker with deionized water of at least 40 times the weight of the colored liquid solution obtained in the previous step.

[0244] 4. Hydrate the dialysis tubing for at least two minutes.

[0245] 5. Seal the bottom of the tubing and fill it with the colored liquid solution.

[0246] 6. Remove any air bubbles from the tubing and seal the top with an added float at the top.

[0247] 7. Add a magnetic stir bar to the water bath and place it on a magnetic stir plate (125rpm max).

[0248] 8. Change the water 8 times within a period of 40 hours (4 times a day, with a gap of 3 hours between each change).

[0249] 9. Place the acquired silk solution in conical tubes for centrifugation.

[0250] 10. Centrifuge at 10,000 RPM for 20 minutes at 4°C to segregate any impurities present in the solution to the side.

[0251] 11. Filter the acquired silk solution through a Grade 4 Qualitative Filter Paper with particle retention of sizes 20-25 μm.

[0252] 12. Silk solution with or without dye is ready.

[0253] **Example 2**

[0254] Silk mono-material embroidery (Fig. 2) is made by fabricating and assembling the following components made of silk: 1) Silk fabric for the base of the embroidery from silk fibers derived from cocoons or woven into textiles, or silk fibers derived from silk textile waste through a mechanical or chemical process or woven into textiles.; 2) Silk threads for attaching the embellishments onto the fabric from silk fibers derived from cocoons, or silk fibers derived from waste silk textile mechanically or chemically; 3) Silk sequins (or other embellishment) from silk films derived from cocoons (including B. mori), or silk films derived from silk textile waste (Fig. 3), pressed silk fibers/composite silk materials, or silk solids.

[0255] This silk mono-material embroidery can be entirely recycled by dissolving it in Lithium Bromide (LiBr) and extracting silk fibroin solution from it. The silk fibroin solution can be used to make recycled silk films that can be laser cut into sequins and used for embroidery again. (Fig. 4) The silk fibroin solution (if recycled from colored fabrics) can also be processed into a powder that can be used as pigment for silk-based products.

[0256] In addition to the features described above and elsewhere herein, the present disclosure also includes the following clauses:

1. A method, comprising:
analyzing a finished textile to identify a dye-binding mode of a dye, the finished textile comprising silk and the dye;
selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the identified dye-binding mode; and
regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

2. The method of clause 1, the method further comprising: identifying the dye-binding mode as reactive; and selecting the one or more regeneration parameters in response to the reactive dye-binding mode.

3. The method of clause 1, the method further comprising: identifying the dye-binding mode as direct; and selecting one or more regeneration parameters in response to the direct dye-binding mode.

4. The method of clause 1, the method further comprising: identifying the dye-binding mode as electrostatic; and selecting one or more regeneration parameters in response to the electrostatic dye-binding mode.

5. The method of clause 1, wherein the dye is present at least one of throughout the finished textile or in a pattern on the finished textile.

6. The method of clause 1, wherein analyzing the finished textile includes analyzing by at least one of FTIR spectroscopy or Raman spectroscopy.

7. The method of clause 1, wherein analyzing the finished textile includes analyzing by at least one of a chemical assay or an enzymatic assay.

8. The method of clause 1, the method further comprising: sorting the finished textile in response to the identified dye-binding mode.

9. The method of clause 8, wherein sorting includes excluding undesirable finished textile from silk fibroin regeneration.

10. The method of clause 1, further comprising:

analyzing the finished textile spectroscopically to determine a proportion of silk in the finished textile;

sorting the finished textile in response to the determined proportion; and

selecting one or more regeneration parameters of the silk fibroin regeneration further in response to the determined proportion.

11. The method of clause 1, further comprising:

determining or estimating a molecular weight of a silk fibroin of the silk in the finished textile; and

selecting one or more regeneration parameters of the silk fibroin regeneration of the finished textile further in response to the determined or estimated molecular weight.

12. The method of clause 1, further comprising:

determining or estimating a molecular weight of a silk fibroin of the silk in the finished textile; and

further sorting the finished textile in response to the determined or estimated molecular weight.

13. A method, comprising:

analyzing a finished textile spectroscopically to identify an aspect of a dye, the finished textile comprising silk and the dye;

selecting one or more regeneration parameters of a silk fibroin regeneration process to be applied to the finished textile in response to the identified aspect of the dye; and

regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

14. The method of clause 13, the method further comprising: sorting the finished textile in response to the identified aspect of the dye.

15. The method of clause 14, wherein sorting includes excluding undesirable finished textile from silk fibroin regeneration.

16. The method of clause 13, wherein the aspect of the dye is at least one of a color, an intensity, a substantivity, a chemical formula, a fluorescence, or a dye-binding type.

17. The method of clause 13, wherein the dye is present at least one of throughout the finished textile or in a pattern on or of the finished textile.

18. The method of clause 13, the method further comprising: determining or estimating a molecular weight of a silk fibroin of the silk; and selecting one or more regeneration parameters of the silk fibroin regeneration of the finished textile further in response to the determined or estimated molecular weight.

19. The method of clause 13, the method further comprising: determining or estimating a molecular weight of a silk fibroin of the silk; and further sorting the finished textile in response to the determined or estimated molecular weight.

20. The method of clause 16, the method further comprising: identifying the dye is a blue dye; and selecting one or more regeneration parameters of the silk fibroin regeneration in response to the blue dye.

21. The method of clause 16, the method further comprising: identifying the dye is a red dye; and selecting one or more regeneration parameters of the silk fibroin regeneration in response to the red dye.

22. The method of clause 16, the method further comprising: identifying the dye is a black dye; and selecting one or more regeneration parameters of the silk fibroin regeneration in response to the black dye.

23. The method of clause 16, the method further comprising: identifying the dye is a green dye; and selecting one or more regeneration parameters of the silk fibroin regeneration in response to the green dye.

24. The method of clause 16, the method further comprising: identifying the dye is a yellow dye; and selecting one or more regeneration parameters of the silk fibroin regeneration in response to the yellow dye.

25. The method of clause 16, the method further comprising: identifying the dye is an orange dye; and selecting one or more regeneration parameters of the silk fibroin regeneration in response to the orange dye.

26. The method of clause 16, the method further comprising: identifying the dye is a violet dye; and selecting one or more regeneration parameters of the silk fibroin regeneration response to the violet dye.

27. The method of clause 16, the method further comprising: identifying the dye-binding type is reactive; and selecting one or more regeneration parameters of the silk fibroin regeneration in response to the reactive dye-binding type.

28. The method of clause 16, the method further comprising: identifying the dye-binding type is direct; and selecting one or more regeneration parameters of the silk fibroin regeneration in response to the direct dye-binding type.

29. The method of clause 16, the method further comprising: identifying the dye-binding mode as electrostatic; and selecting one or more regeneration parameters in response to the electrostatic dye-binding mode.

30. A method, comprising:

analyzing a finished textile to determine a proportion of silk in the finished textile;

selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the determined proportion; and

regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

31. The method of clause 30, the method further comprising: sorting the finished textile in response to the determined proportion.

32. The method of clause 31, wherein sorting excludes undesirable finished textile from the silk fibroin regeneration in response to the determined proportion not meeting a threshold.

33. The method of clause 30, the method further comprising: restricting undesirable finished textile from the silk fibroin regeneration if the determined proportion does not meet a defined minimum proportion.

34. The method of clause 30, wherein analyzing the finished textile includes analyzing by FTIR spectroscopy.

35. The method of clause 30, the method further comprising: further analyzing the finished textile spectroscopically to identify a dye-binding mode;

further sorting the finished textile in response to the identified dye-binding mode; and

selecting one or more regeneration parameters of the silk fibroin regeneration further in response to the identified dye-binding mode.

36. The method of clause 30, the method further comprising: determining or estimating a molecular weight of a silk fibroin of the silk; and selecting one or more regeneration parameters of the silk fibroin regeneration of the finished textile further in response to the determined or estimated molecular weight.

37. The method of clause 30, the method further comprising: determining or estimating a molecular weight of a silk fibroin of the silk; and further sorting the finished textile in response to the determined or estimated molecular weight.

38. The method of clause 30, wherein the proportion is between 5% and 95% or between 10% and 80%, including but not limited to, at least 5%, at least 15%, at least 20%, at least 25%, at least 50%, at least 60%, at least 80%, or at least 90%, and at most 95%, at most 75%, at most 50%, at most 25%, at most 10%, or at most 5%.

39. A method, comprising:

analyzing a finished textile to determine a composition of the finished textile, the finished textile comprising silk;

selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the determined composition; and

regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

40. The method of clause 39, the method further comprising: sorting the finished textile in response to the determined composition.

41. The method of clause 40, wherein sorting excludes undesirable finished textile from the silk fibroin regeneration in response to the determined composition not meeting a threshold.

42. The method of clause 39, the method further comprising: restricting undesirable finished textile from the silk fibroin regeneration if the determined proportion does not meet a defined minimum composition.

43. The method of clause 39, wherein analyzing the finished textile includes analyzing by at least one of FTIR spectroscopy or Raman spectroscopy.

44. The method of clause 39, the method further comprising: further analyzing the finished textile spectroscopically to identify a dye-binding mode;

further sorting the finished textile in response to the identified dye-binding mode; and

selecting one or more regeneration parameters of the silk fibroin regeneration further in response to the identified dye-binding mode.

45. The method of clause 39, the method further comprising: determining or estimating a molecular weight of a silk fibroin of the silk; and selecting one or more regeneration parameters of the silk fibroin regeneration of the finished textile further in response to the determined or estimated molecular weight.

46. The method of clause 39, the method further comprising: determining or estimating a molecular weight of a silk fibroin of the silk; and further sorting the finished textile in response to the determined or estimated molecular weight.

47. A method, comprising:

determining or estimating a molecular weight of a silk fibroin of silk in a finished textile;

selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile further in response to the determined or estimated molecular weight; and

regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

48. The method of clause 47, the method further comprising: excluding undesirable finished textile from silk fibroin regeneration in response to determining or estimating the molecular weight is below a threshold.

49. The method of clause 47, the method further comprising: excluding undesirable finished textile from silk fibroin regeneration in response to determining or estimating the molecular weight is above a threshold.

50. A method, comprising:

imaging a finished textile to determine at least one of an age or a wear value of the finished textile, the finished textile comprising silk;

selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the at least one determined age or determined wear value; and

regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

51. The method of clause 50, the method further comprising: sorting the finished textile in response to the at least one determined age or determined wear value.

52. The method of clause 51, wherein sorting excludes undesirable finished textile from silk fibroin regeneration.

53. The method of clause 50, the method further comprising: determining or estimating a molecular weight of a silk fibroin of the silk; and selecting one or more regeneration parameters of the silk fibroin regeneration of the finished textile further in response to the determined or estimated molecular weight.

54. The method of clause 50, the method further comprising: determining or estimating a molecular weight of a silk fibroin of the silk; and sorting the finished textile in response to the determined or estimated molecular weight.

55. A method, comprising:

determining at least one of an age or a wear value of a finished textile, the finished textile comprising silk, wherein determining comprises at least one of imaging the finished textile, performing a microscopic analysis of the finished textile, or performing a mechanical analysis of the finished textile;

selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the at least one determined age or determined wear value; and

regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

56. The method of clause 55, sorting the finished textile in response to the at least one determined age or determined wear value.

57. The method of clause 56, wherein sorting excludes undesirable finished textile from silk fibroin regeneration.

58. The method of clause 55, the method further comprising: determining or estimating a molecular weight of a silk fibroin of the silk; and selecting one or more regeneration parameters of the silk fibroin regeneration of the finished textile further in response to the determined or estimated molecular weight.

59. The method of clause 55, the method further comprising: determining or estimating a molecular weight of a silk fibroin of the silk; and sorting the finished textile in response to the determined or estimated molecular weight.

60. A method, comprising:

identifying a presence of a coating of a finished textile, the finished textile comprising silk;

selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the presence of the coating; and

regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

61. The method of clause 60, wherein the coating is at least one of a waterproofing material, a synthetic plastic, an antibacterial material, a conductive material, or a flame-retardant material.

62. The method of clause 60, sorting the finished textile in response to at least one of the presence or an absence of the coating.

63. The method of clause 62, wherein sorting excludes undesirable finished textile from silk fibroin regeneration.

64. The method of clause 60, wherein identifying comprises at least one of spectroscopically analyzing the finished textile, imaging the finished textile, performing a chemical assay of the finished textile, or performing an enzymatic assay of the finished textile.

65. The method of clause 60, the method further comprising: restricting undesirable finished textile from the silk fibroin regeneration in response to identifying the presence.

66. The method of clause 60, removing, in response to identifying the presence, the coating from the finished textile prior to silk fibroin regeneration.

67. A method, comprising:

identifying a presence or an absence of a coating of a finished textile, the finished textile comprising silk;

sorting the finished textile in response to at least one of the presence or the absence of the coating;

removing, in response to the presence, the coating from the finished textile;

analyzing the finished textile to determine a proportion of silk in the finished textile;

sorting the finished textile in response to the determined proportion;

analyzing the finished textile to identify an aspect of a dye;

sorting the finished textile in response to the identified aspect of the dye;

selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to at least one of the determined proportion, the presence or the absence of the coating, or the aspect of the dye; and

regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

68. The method of clause 67, the method further comprising: restricting undesirable finished textile from the silk fibroin regeneration in response to the determined proportion not meeting a threshold.

69. The method of clause 67, wherein analyzing comprises at least one of spectroscopically analyzing the finished textile, imaging the finished textile, performing a chemical assay of the finished textile, or performing an enzymatic assay of the finished textile.

70. The method of clause 67, wherein the aspect of the dye is at least one of a presence, a color, an intensity, a substantivity, a chemical formula, a fluorescence, or a binding type (e.g., direct, reactive, electrostatic).

71. The method of clause 67, the method further comprising: bleaching the finished textile in response to the aspect of the dye.

72. A method, comprising:

identifying a presence or an absence of a coating of a finished textile, the finished textile comprising silk;

removing, in response to the presence, the coating from the finished textile;

analyzing the finished textile to determine a proportion of silk in the finished textile;

analyzing the finished textile to identify an aspect of a dye;

selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to at least one of the determined proportion, the presence or the absence of the coating, or the aspect of the dye; and

regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

73. The method of any one of the preceding clauses, wherein the finished textile is a post-consumer waste product.

74. The method of any one of the preceding clauses, wherein the finished textile is at least one of a dyed textile, a coated textile, a waterproofed textile, a silkscreened textile, or a heterogenous product.

75. The method of any one of the preceding clauses, wherein the regenerated silk fibroin solution comprises a color that is within 80% of a measured color of a cocoon-derived silk.

76. The method of any one of the preceding clauses, wherein the regenerated silk fibroin solution comprises a transparency that is within 80% of a measured transparency of a cocoon-derived silk.

77. The method of any one of the preceding clauses, wherein the regenerated silk fibroin solution comprises a viscosity that is within 80% of a measured viscosity of a cocoon-derived silk.

78. The method of any one of the preceding clauses, wherein the regenerated silk fibroin solution comprises a secondary structure tuning (β-sheet induction after methanol treatment/water annealing) within 50% of a cocoon-derived silk.

79. The method of any one of the preceding clauses, the method further comprising: identifying at least one pattern of the finished textile; and selecting one or more regeneration parameters of the silk fibroin regeneration further in response to the at least one pattern.

80. The method of the immediately preceding clause, wherein the at least one pattern is a woven pattern, a printed pattern, or a mixed media pattern.

81. The method of any one of the preceding clauses, the method further comprising: mechanically processing the finished textile.

82. The method of the immediately preceding clause, wherein mechanically processing is at least one of shredding, flaking, cutting, milling, or grinding.

83. The method of any one of the preceding clauses, wherein silk fibroin regeneration comprises the steps of:

dissolving a degummed, rinsed, and dried finished textile in a salt solution to form a silk fibroin solution;

dialyzing the silk fibroin solution against distilled water to obtain a dialyzed silk fibroin solution in water;

centrifuging the dialyzed silk fibroin solution twice to obtain a supernatant; and

diluting the supernatant in deionized water to obtain a regenerated silk fibroin solution.

84. The method of clause 83, wherein the dialyzed silk fibroin solution is a 5–8 % wt silk fibroin solution.

85. The method of clause 83, wherein dissolving is in an oven at 60 °C for 4 hours.

86. The method of clause 83, wherein the silk fibroin solution is stirred after an hour of elapsed dissolving time.

87. The method of clause 83, wherein the silk fibroin solution is stirred after two hours of elapsed dissolving time.

88. The method of clause 83, wherein the salt solution is a LiBr solution.

89. The method of clause 83, wherein the salt solution is a 20 w/v% LiBr solution

90. The method of clause 89, wherein a concentration of LiBr is 9.3M.

91. The method of clause 83, wherein dialyzing is in dialysis cassettes.

92. The method of clause 83, wherein dialyzing is for 36 hours.

93. The method of clause 83, wherein centrifuging is at 8000 rpm.

94. The method of clause 83, the method further comprising: storing the regenerated silk fibroin solution at 4 °C

95. The method of any one of the preceding clauses, wherein the silk fibroin regeneration includes at least one organic solvent.

96. The method of any one of the preceding clauses, the method further comprising: collecting a precipitated material; and drying the precipitated material.

97. The method of any one of the preceding clauses, the method further comprising: collecting a precipitated material; and repeating the silk fibroin regeneration.

98. The method of any one of the preceding clauses, the method further comprising: collecting a precipitated material to be used as a moldable material.

99. The method of any one of the preceding clauses, the method further comprising: determining a presence of at least one of a non-silk fiber/component or a contaminant of the finished textile; and at least one of:

sorting the finished textile in response to the presence; or

selecting one or more regeneration parameters of the silk fibroin regeneration further in response to the presence.

100. The method of any one of the preceding clauses, wherein selecting one or more regeneration parameters includes selecting one or more dissolution operational parameters.

101. The method of the immediately preceding clause, wherein the one or more dissolution operational parameters include a dissolution temperature, a dissolution length of time, or a combination thereof.

102. The method of any one of the preceding clauses, wherein selecting one or more regeneration parameters of a silk fibroin regeneration process includes selecting one or more chemical composition parameters.

103. The method of the immediately preceding clause, wherein the one or more chemical composition parameters includes a dissolution chemical composition, a desalinated chemical composition, a concentrated chemical composition, a dilute chemical composition, or a combination thereof.

104. The method of any one of the preceding clauses, wherein the molecular weight is between 5 kDa to 400 kDa, between 3.5 kDa and 120 kDa, or between 20 kDa and 125 kDa, including but not limited to, at least 5 kDa, at least 20 kDa, at least 30 kDa, at least 40 kDa, at least 50 kDa, at least 60 kDa, at least 100 kDa, at least 200 kDa, at least 300 kDa, at least 400 kDa, at most 400 kDa, at most 375 kDa, at most 350 kDa, at most 325 kDa, at most 300 kDa, at most 200 kDa, at most 100 kDa, at most 50 kDa, at most 20 kDa, or at most 5 kDa.

105. A composition comprising a regenerated silk fibroin made by the method of any one of the preceding clauses.

106. A composition comprising:

a silk fibroin; and

a regenerated silk fibroin made by a method of any one of clauses 1 to 104.

107. A composition comprising a regenerated silk fibroin made by the method of any one of clauses 1 to 104 and a functionalizing agent.

108. A composition comprising:

a silk fibroin;

a regenerated silk fibroin made by a method of any one of clauses 1 to 104; and

a functionalizing agent.

109. The composition of any one of clauses 105 to the immediately preceding clause, wherein the regenerated silk fibroin comprises a color that is within 80% of a measured color of a cocoon-derived silk.

110. The composition of any one of clauses 105 to the immediately preceding clause, wherein the regenerated silk fibroin comprises a transparency that is within 80% of a measured transparency of a cocoon-derived silk.

111. The composition of any one of clauses 105 to the immediately preceding clause, wherein the regenerated silk fibroin comprises a viscosity that is within 80% of a measured viscosity of a cocoon-derived silk.

112. The composition of any one of clauses 105 to the immediately preceding clause, wherein the regenerated silk fibroin comprises a secondary structure tuning (β-sheet induction after methanol treatment/water annealing) within 50% of a cocoon-derived silk.

113. An article made from regenerated silk fibroin made by the method of clauses 1 to 104.

114. An article made from the composition of any one of clauses 105 to 112.

115. The article of clause 113 or 114, wherein the article is at least one of a silk foam, a film, a silk leather, an adhesive, a building construction material, a thermal insulator, a silk embellishment, a silk sequin, a silk embroidery, a silk thread, a silk mono-material embroidery, or a PDA mechanical sensor.

116. The article of clause 115, wherein the article is the adhesive and the adhesive exhibits at least 20% of an adhesive strength of a cocoon-derived silk.

117. The article of any one clauses 114 to 116, wherein the article exhibits at least one mechanical property that is at least 50% of a same property in an article made from a cocoon-derived silk.

118. The article of clause 115, wherein the article is the film having a film transparency of at least 80% of a comparison transparency of a comparison film made of a cocoon-derived silk.

119. The article of any one clauses 114 to 118 wherein the article further comprises an additional recycled component (e.g., recycled cardboard, wool) therein.

120. A silk embellishment made from the composition of any one of clauses 105 to 112.
121. A silk sequin made from the composition of any one of clauses 105 to 112.
122. A silk thread made from the composition of any one of clauses 105 to 112.
123. A silk mono-material embroidery made from the composition of any one of clauses 105 to 112.
124. A method of any one of clauses 1 through 104, the method further comprising: drying the composition to form a film; and laser cutting the film to form a silk mono-material embroidery.
125. A method of any one of clauses 1 through 104, the method further comprising: coating a silk textile with the composition to form a coated silk textile, drying the coated silk textile; and laser cutting the coated silk textile to form a silk mono-material embroidery.
126. A textile waste stream processing system, comprising:
a spectroscopic imaging facility operationally coupled to a textile waste storage vessel, the spectroscopic imaging facility communicatively coupled to a data collector, the data collector structured to collect a plurality of detection values from the spectroscopic imaging facility upon imaging at least one waste textile in the textile waste storage vessel;
a data storage structured to store a plurality of spectra patterns of textiles, the stored plurality of spectra patterns comprising a library of spectra patterns;
a data acquisition circuit structured to interpret the plurality of detection values from the data collector;
an expert system analysis circuit structured to analyze the plurality of detection values to determine if one of the plurality of detection values has a recognized spectra pattern corresponding to at least one of the stored plurality of spectra patterns from the library of spectra patterns; and
a response circuit structured to provide an instruction in response to the one of the plurality of detection values having a stored spectra pattern.
127. The textile waste stream processing system of clause 126, wherein the instruction is to further process the waste textile.
128. The textile waste stream processing system of clause 126, wherein the instruction comprises one or more dissolution parameters for processing the waste textile.
129. The textile waste stream processing system of clause 126, wherein the instruction is restricting the waste textile from further processing.
130. The textile waste stream processing system of clause 126, wherein the spectroscopic imaging facility is at least one of an FTIR or a Raman spectroscopic imaging facility.
131. The textile waste stream processing system of clause 126, wherein the library of spectra patterns is available to a spectra pattern marketplace, where users are provided access to the spectra pattern marketplace.

132. The textile waste stream processing system of clause 131, wherein the library of spectra pattern is updated periodically by one or more users.

133. The textile waste stream processing system of clause 126, wherein the response circuit utilizes at least one of industry-specific feedback, the library of spectra patterns, or a user input to select an appropriate instruction in response to the one of the plurality of detection values having a stored spectra pattern.

134. The textile waste stream processing system of clause 126, wherein the stored plurality of spectra patterns in the library of spectra patterns further includes information regarding one or more of fabric types, dye types, dye-binding modes, coating types, age/wear, and successful dissolution parameters.

CLAIMS

What is claimed is:

1. A method, comprising:

analyzing a finished textile to identify a dye-binding mode of a dye, the finished textile comprising silk and the dye;

selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the identified dye-binding mode; and

regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

2. The method of claim 1, the method further comprising: sorting the finished textile in response to the identified dye-binding mode.

3. The method of claim 1, further comprising:

analyzing the finished textile spectroscopically to determine a proportion of silk in the finished textile;

sorting the finished textile in response to the determined proportion; and

selecting one or more regeneration parameters of the silk fibroin regeneration further in response to the determined proportion.

4. The method of claim 1, further comprising:

determining or estimating a molecular weight of a silk fibroin of the silk in the finished textile; and

selecting one or more regeneration parameters of the silk fibroin regeneration of the finished textile further in response to the determined or estimated molecular weight.

5. The method of claim 1, further comprising:

determining or estimating a molecular weight of a silk fibroin of the silk in the finished textile; and

further sorting the finished textile in response to the determined or estimated molecular weight.

6. A method, comprising:

analyzing a finished textile spectroscopically to identify an aspect of a dye, the finished textile comprising silk and the dye;

selecting one or more regeneration parameters of a silk fibroin regeneration process to be applied to the finished textile in response to the identified aspect of the dye; and

regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

7. A method, comprising:

analyzing a finished textile to determine a proportion of silk in the finished textile;

selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the determined proportion; and

regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

8. The method of claim 7, the method further comprising: restricting undesirable finished textile from the silk fibroin regeneration if the determined proportion does not meet a defined minimum proportion.

9. A method, comprising:

determining at least one of an age or a wear value of a finished textile, the finished textile comprising silk, wherein determining comprises at least one of imaging the finished textile, performing a microscopic analysis of the finished textile, or performing a mechanical analysis of the finished textile;

selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the at least one determined age or determined wear value; and

regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

10. A method, comprising:

identifying a presence of a coating of a finished textile, the finished textile comprising silk;

selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile in response to the presence of the coating; and

regenerating a silk fibroin solution from the finished textile using the one or more regeneration parameters.

11. The method of any one of the preceding claims, wherein silk fibroin regeneration comprises the steps of:

dissolving a degummed, rinsed, and dried finished textile in a salt solution to form a silk fibroin solution;

dialyzing the silk fibroin solution against distilled water to obtain a dialyzed silk fibroin solution in water;

centrifuging the dialyzed silk fibroin solution twice to obtain a supernatant; and

diluting the supernatant in deionized water to obtain a regenerated silk fibroin solution.

12. The method of any one of the preceding claims, the method further comprising:

determining a presence of at least one of a non-silk fiber/component or a contaminant of the finished textile; and at least one of:

sorting the finished textile in response to the presence; or

selecting one or more regeneration parameters of the silk fibroin regeneration further in response to the presence.

13. The method of any one of the preceding claims, wherein selecting one or more regeneration parameters includes selecting one or more dissolution operational parameters.

14. The method of any one of the preceding claims, wherein selecting one or more regeneration parameters of a silk fibroin regeneration process includes selecting one or more chemical composition parameters.

15. A composition comprising a regenerated silk fibroin made by the method of any one of the preceding claims.

16. The composition of any one of the preceding claims, wherein the regenerated silk fibroin comprises a color that is within 80% of a measured color of a cocoon-derived silk.

17. The composition of any one of the preceding claims, wherein the regenerated silk fibroin comprises a transparency that is within 80% of a measured transparency of a cocoon-derived silk.

18. The composition of any one of the preceding claims, wherein the regenerated silk fibroin comprises a viscosity that is within 80% of a measured viscosity of a cocoon-derived silk.

19. The composition of any one of the preceding claims, wherein the regenerated silk fibroin comprises a secondary structure tuning (β-sheet induction after methanol treatment/water annealing) within 50% of a cocoon-derived silk.

20. An article made from regenerated silk fibroin made by the method of any one of the preceding claims.

21. A silk mono-material embroidery made from the composition of any one of the preceding claims.

22. A textile waste stream processing system, comprising:

a spectroscopic imaging facility operationally coupled to a textile waste storage vessel, the spectroscopic imaging facility communicatively coupled to a data collector, the data collector structured to collect a plurality of detection values from the spectroscopic imaging facility upon imaging at least one waste textile in the textile waste storage vessel;

a data storage structured to store a plurality of spectra patterns of textiles, the stored plurality of spectra patterns comprising a library of spectra patterns;

a data acquisition circuit structured to interpret the plurality of detection values from the data collector;

an expert system analysis circuit structured to analyze the plurality of detection values to determine if one of the plurality of detection values has a recognized spectra pattern corresponding to at least one of the stored plurality of spectra patterns from the library of spectra patterns; and

a response circuit structured to provide an instruction in response to the one of the plurality of detection values having a stored spectra pattern.

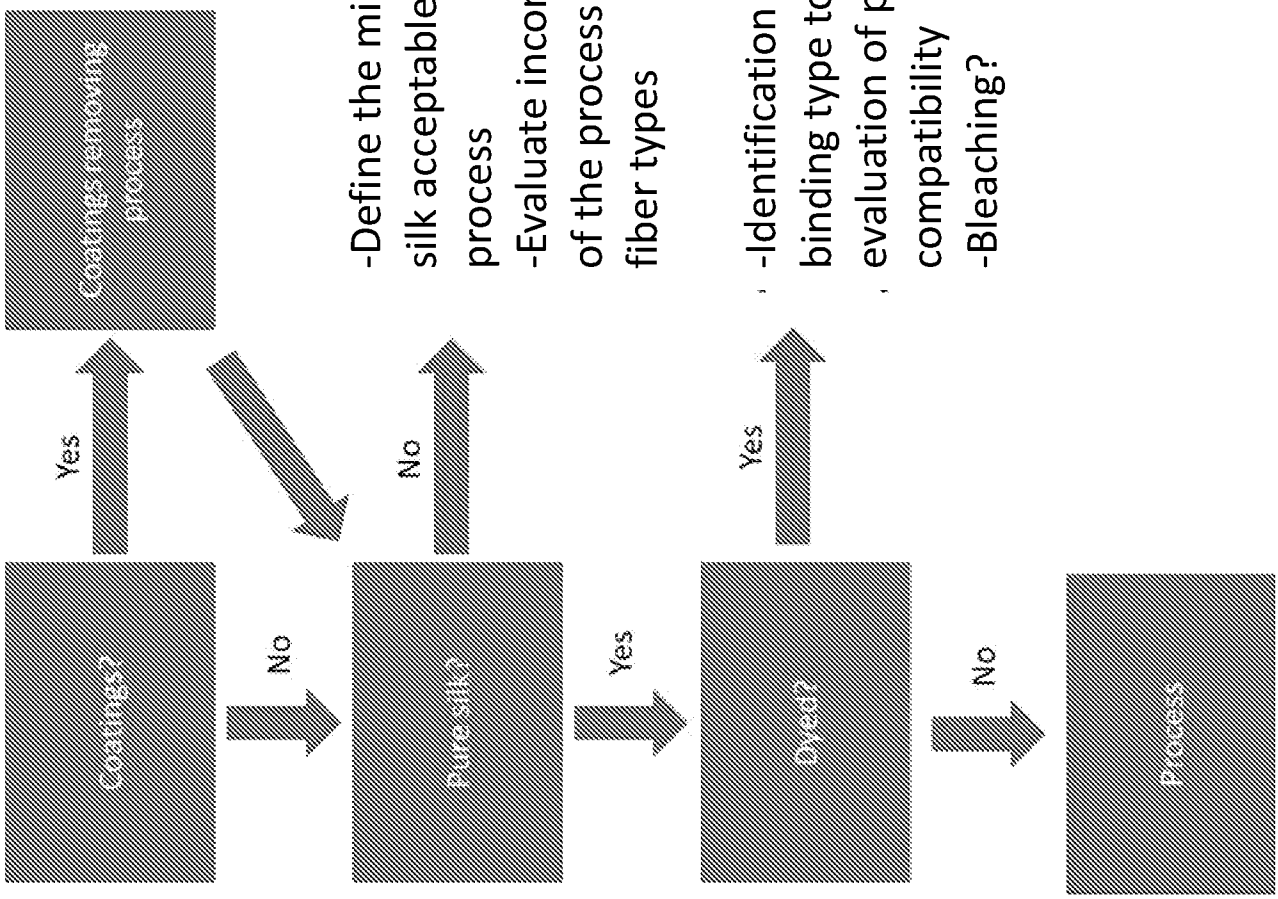

Coating?
Yes
Coating/removing process
No
-Define the minimum % of silk acceptable for the process
-Evaluate incompatibility of the process for specific fiber types
Pure silk?
No
Yes
-Identification of the dye binding type to silk and evaluation of process compatibility
-Bleaching?
Dyed?
Yes
No
Process

FIG. 1

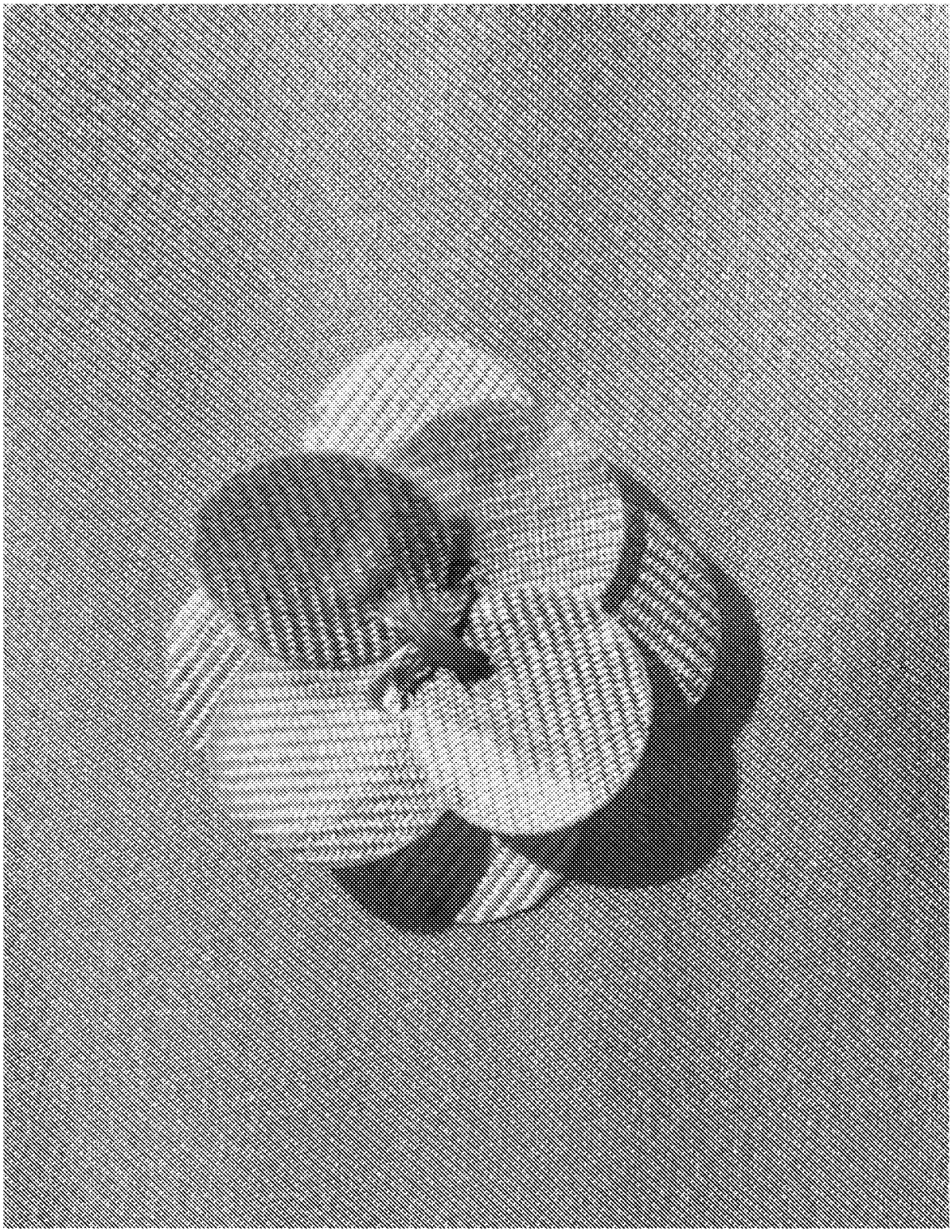


FIG. 2

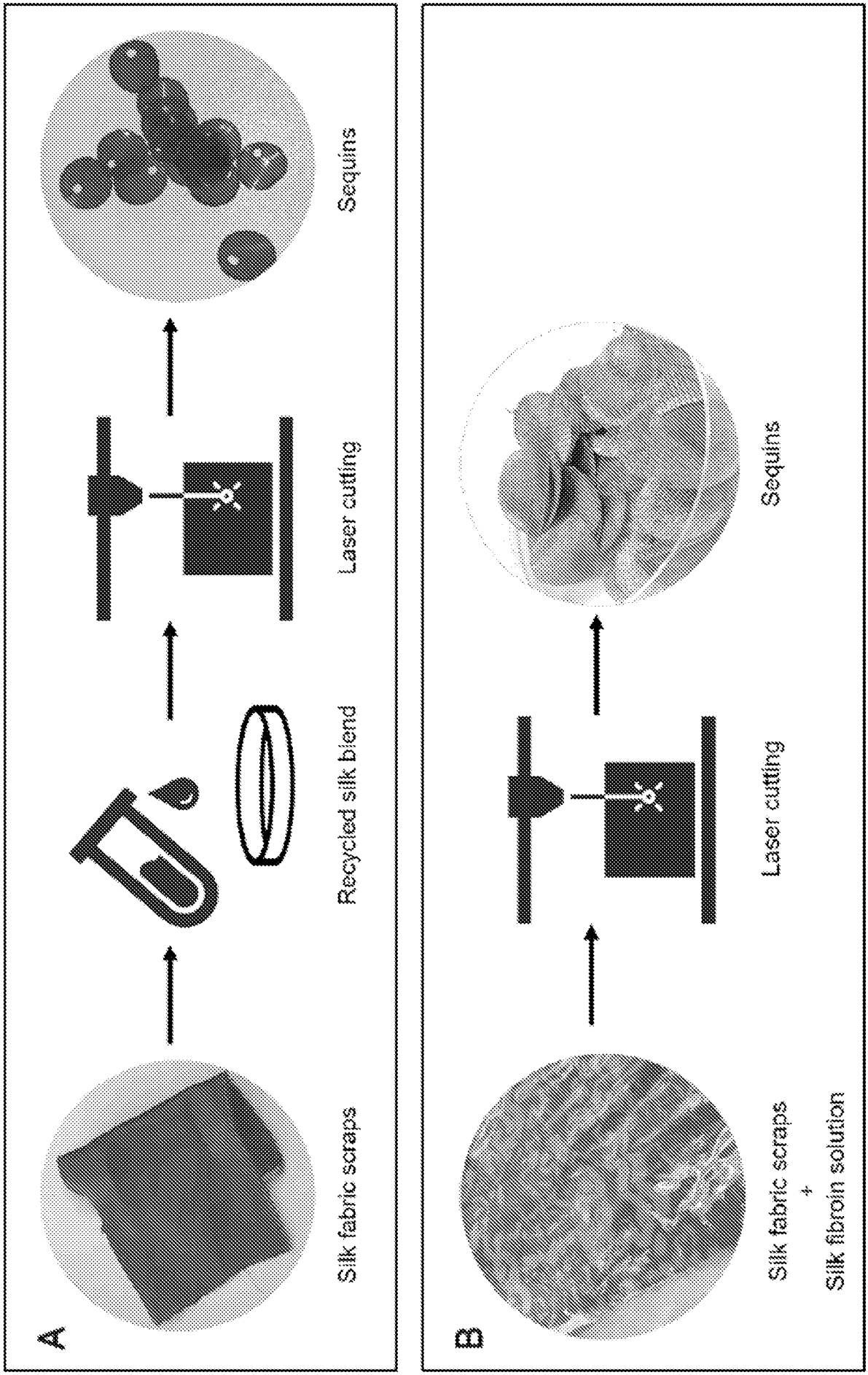

A
Silk fabric scraps
Recycled silk blend
Laser cutting
Sequins
B
Silk fabric scraps
+
Silk fibroin solution
Laser cutting
Sequins

FIG. 3

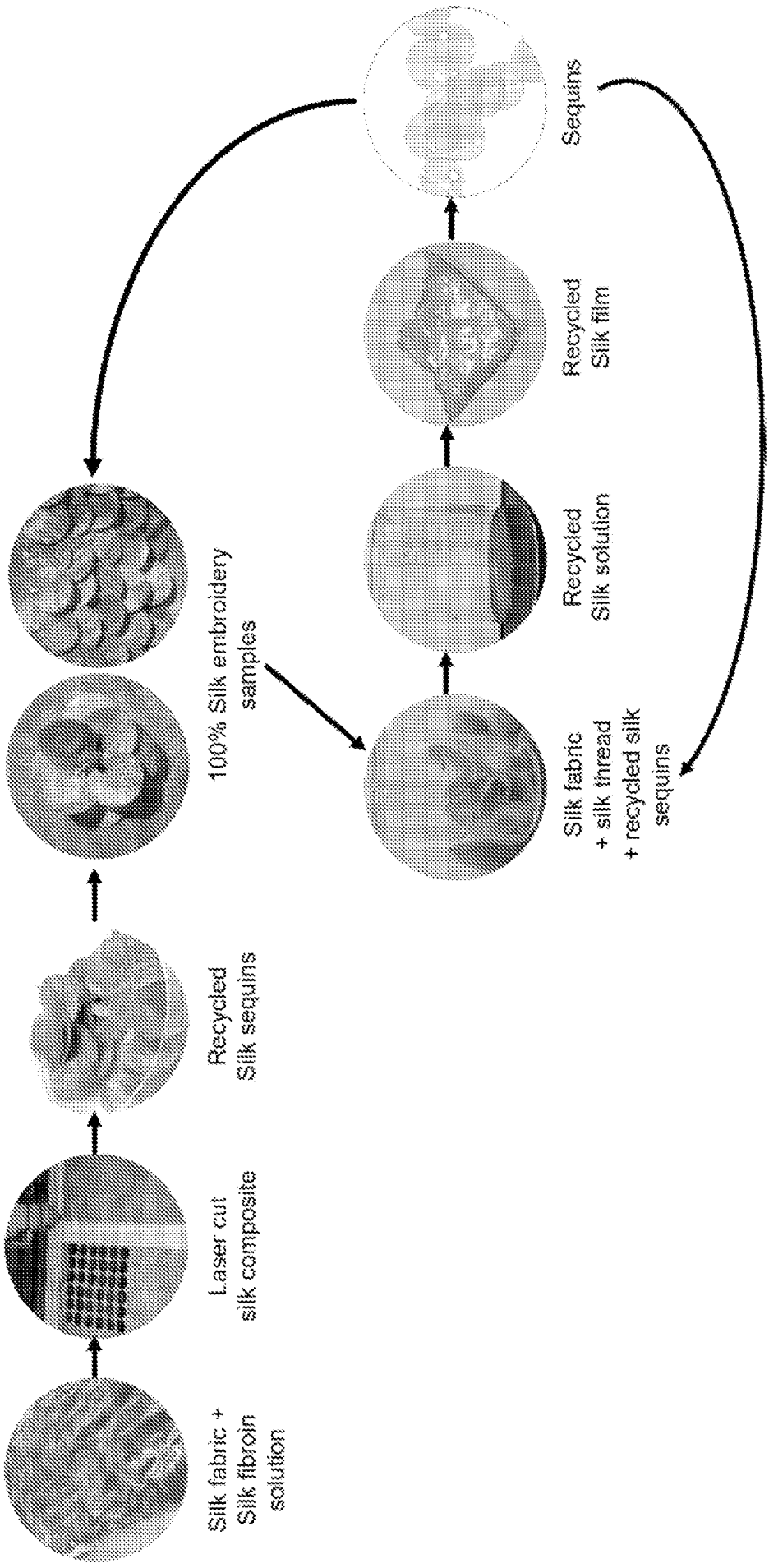

Silk fabric + Silk fibroin solution
Laser cut silk composite
Recycled Silk sequins
100% Silk embroidery samples
Silk fabric + silk thread + recycled silk sequins
Recycled Silk solution
Recycled Silk film
Sequins

FIG. 4

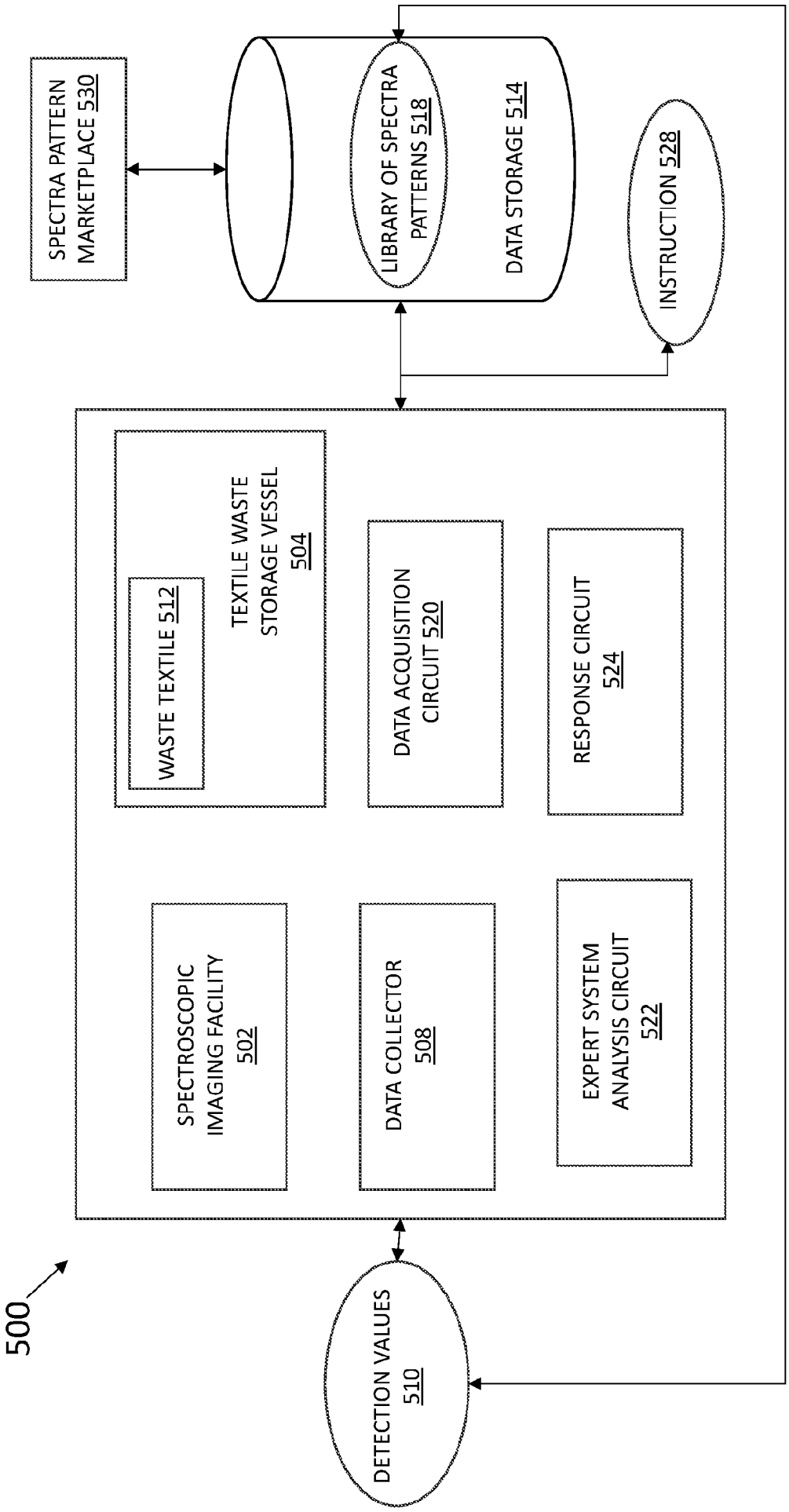

500
SPECTRA PATTERN MARKETPLACE 530
LIBRARY OF SPECTRA PATTERNS 518
DATA STORAGE 514
INSTRUCTION 528
WASTE TEXTILE 512
TEXTILE WASTE STORAGE VESSEL 504
DATA ACQUISITION CIRCUIT 520
RESPONSE CIRCUIT 524
SPECTROSCOPIC IMAGING FACILITY 502
DATA COLLECTOR 508
EXPERT SYSTEM ANALYSIS CIRCUIT 522
DETECTION VALUES 510

FIG. 5

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2024/039880

A. CLASSIFICATION OF SUBJECT MATTER

IPC: ***C08L 89/00*** (2023.01); ***D06M 11/00*** (2023.01); ***D06M 23/14*** (2023.01); ***D06P 5/02*** (2023.01); *D06M 13/52* (2023.01); *D06M 10/04* (2023.01)

CPC: ***C08L 89/00; D06M 11/00; D06M 7/00; D06M 23/14; D06P 5/02;*** *D06M 10/04; Y02W 30/50; Y02P 20/145; D06M 2101/12*

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

See Search History Document

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

See Search History Document

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

See Search History Document

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 2005/010493 A2 (FQUBED, INC.) 03 February 2005 (03.02.2005) pg 26, ln 15-17; pg 10, ln 34-35; pg 25, ln 33-34	1-11
A	US 2006/0273279 A1 (KAPLAN et al.) 07 December 2006 (07.12.2006) para [0025], [0043]-[0047], [0419]	1-11
A	Lu, Linlin, et al. "Progress in recycling and valorization of waste silk." Science of the Total Environment 830 (2022): 154812. [online URL= https://www.sciencedirect.com/science/article/pii/S0048969722019052 pg 1, col 1-pg 5 col 1	1-11

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"D" document cited by the applicant in the international application
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
18 November 2024 (18.11.2024)	**27 November 2024 (27.11.2024)**

Name and mailing address of the ISA/US	Authorized officer
COMMISSIONER FOR PATENTS **MAIL STOP PCT, ATTN: ISA/US** **P.O. Box 1450** **Alexandria, VA 22313-1450** **UNITED STATES OF AMERICA**	**KARI RODRIQUEZ**
Facsimile No. **571-273-8300**	Telephone No. **PCT Help Desk: 571-272-4300**

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2024/039880

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☑ Claims Nos.: **12-21**
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

This application contains the following inventions or groups of inventions which are not so linked as to form a single general inventive concept under PCT Rule 13.1.

Group I: Claims 1-11 drawn to a method including analyzing a finished textile and selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile.

Group II: Claim 22, drawn to a textile waste stream processing system.

The inventions listed in the above-mentioned groups do not relate to a single general inventive concept under PCT Rule 13.1 because, under PCT Rule 13.2, they lack the same or corresponding special technical features for the following reasons:

Special Technical Features

Group I includes the special technical feature of analyzing a finished textile and selecting one or more regeneration parameters of a silk fibroin regeneration to be applied to the finished textile, not included in the other groups.

Group II includes the special technical feature of an expert system analysis circuit structured to analyze a plurality of detection values to determine if one of the plurality of detection values has a recognized spectra pattern and a response circuit structured to provide an instruction in response to the one of the plurality of detection values having a stored spectra pattern, not included in the other groups.

Common Technical Features:

The only technical feature shared by Groups I and II that would otherwise unify the groups is a textile. However, this shared technical feature does not represent a contribution over prior art, because the shared technical feature is disclosed by WO 2005/010493 A2 to FQUBED, INC. (hereinafter FQUBED).

FQUBED discloses a textile (pg 10, ln 34-35: Preferred fabrics include those comprised of natural materials such as cotton, wool, leather, silk).

Accordingly, the inventions listed as the above Groups lack unity of invention under PCT Rule 13 because the do not share a same or corresponding special technical feature providing a contribution over prior art.

item 4. (contd) Claims 12-21 are determined to be unsearchable because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a) and are, therefore, not included in any claim group.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2024/039880

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying additional fees, this Authority did not invite payment of additional fees.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☑ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.: **1-11**

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.

☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.

☐ No protest accompanied the payment of additional search fees.