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(54) **RECOVERING POLY(VINYL BUTYRAL)**  
**POLYMER**

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(57) **ABSTRACT**

A method of recovering poly(vinyl butyral) (PVB). The process comprises mixing a PVB feed composition with a solvent, a catalyst, and, optionally, butyraldehyde to produce an equilibrated PVB-containing composition. The PVB feed composition comprises at least two different PVB-containing compositions. An additional step includes determining a residual polyvinyl alcohol (PVOH) content of the equilibrated PVB-containing composition. A further step includes adjusting the amount of butyraldehyde used in the mixing step based on the determined residual PVOH content.

**Related U.S. Application Data**

(60) Provisional application No. 63/263,162, filed on Oct. 28, 2021, provisional application No. 63/368,383, filed on Jul. 14, 2022, provisional application No. 63/377,410, filed on Sep. 28, 2022, provisional ap-

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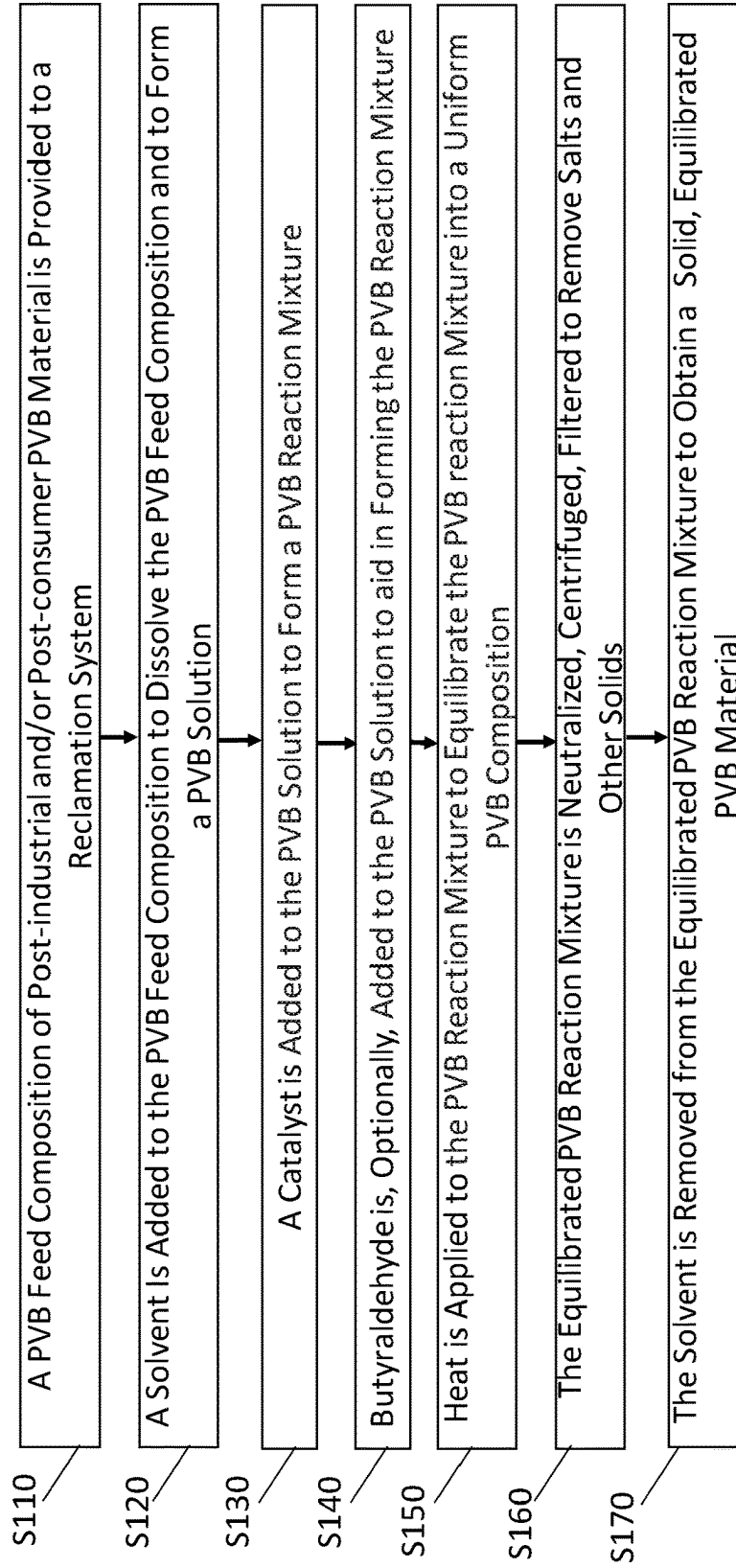


FIG. 1A

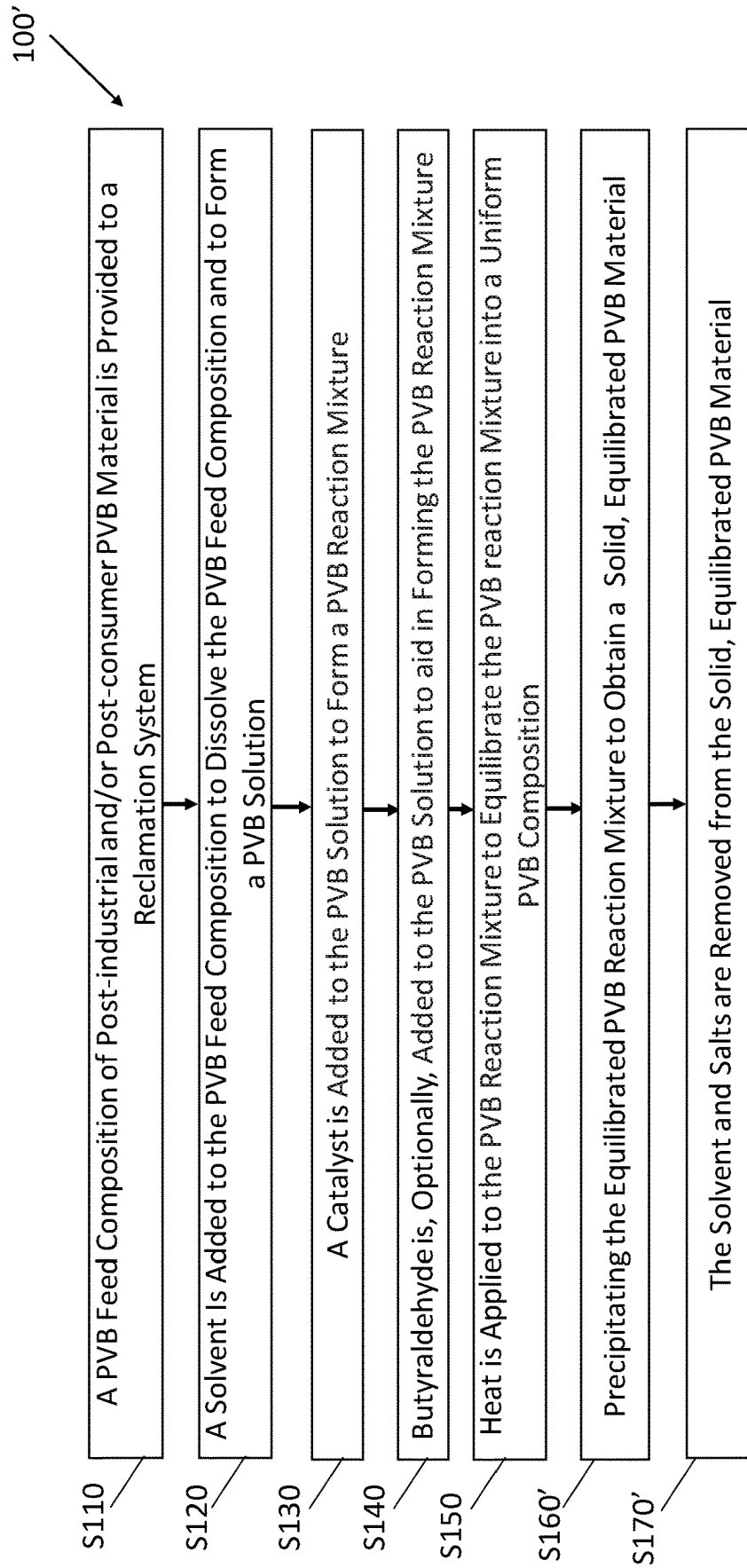


FIG. 1B

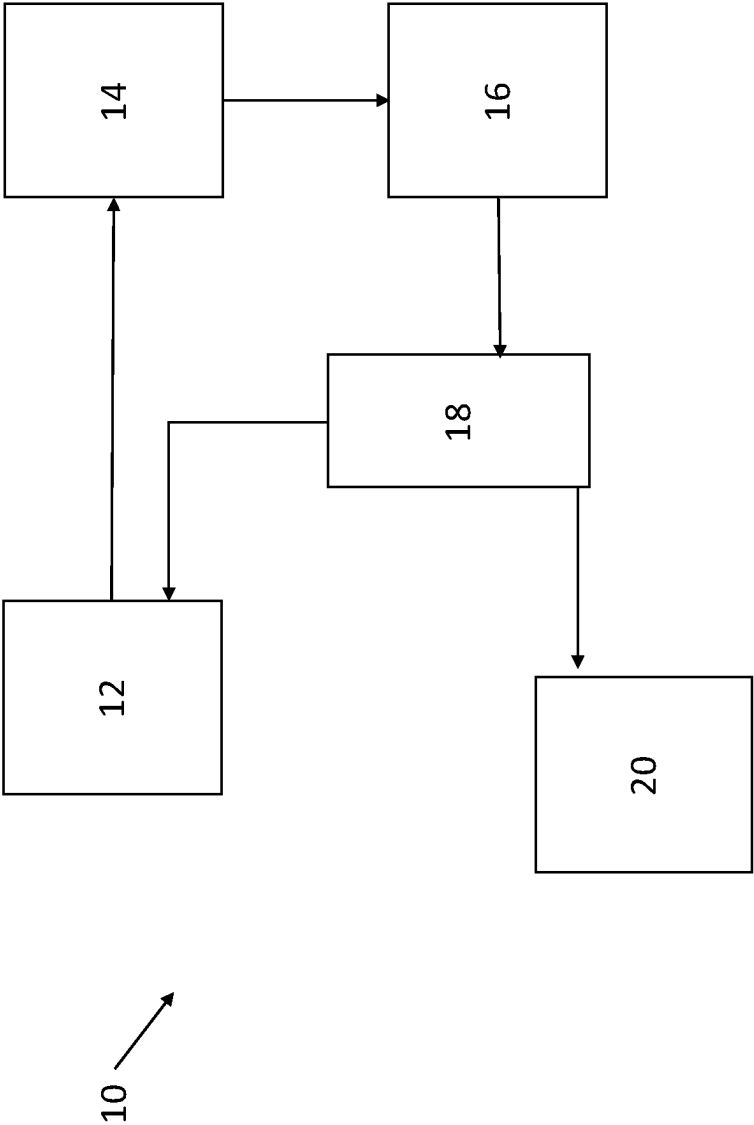


FIG. 2A

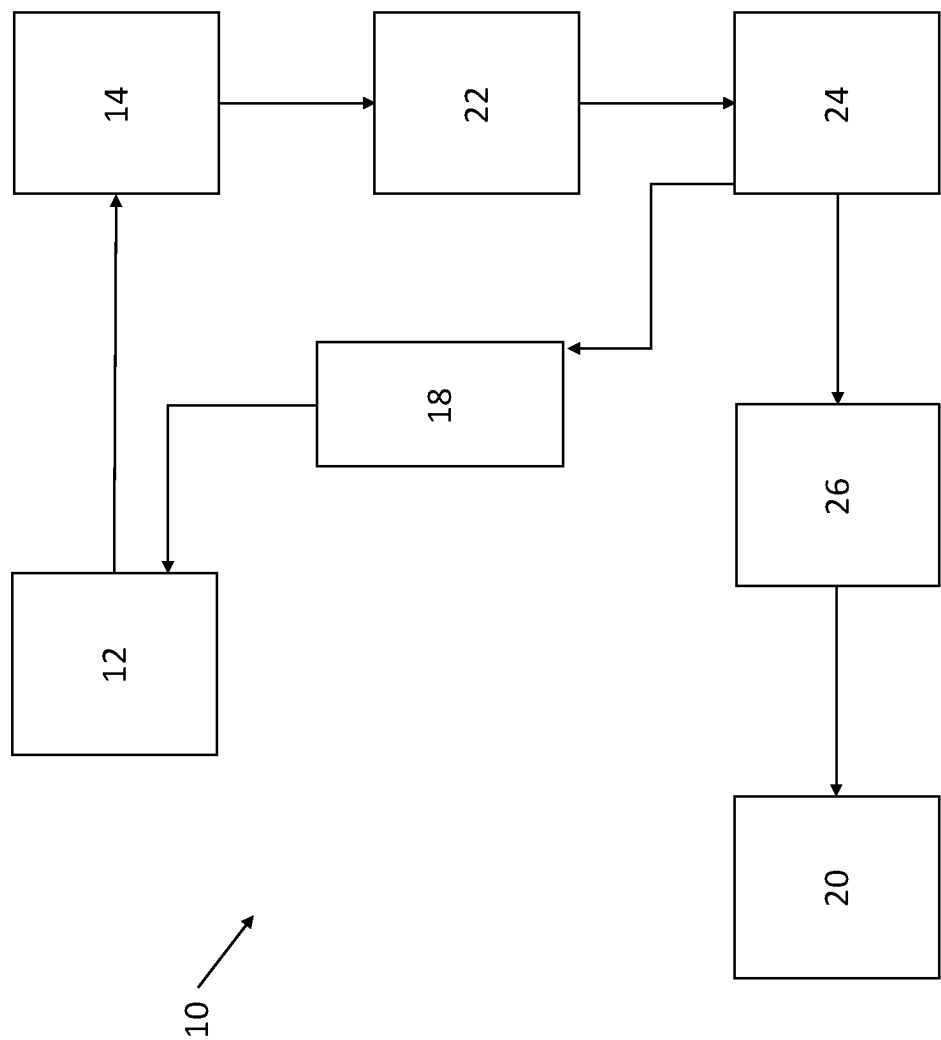


FIG. 2B

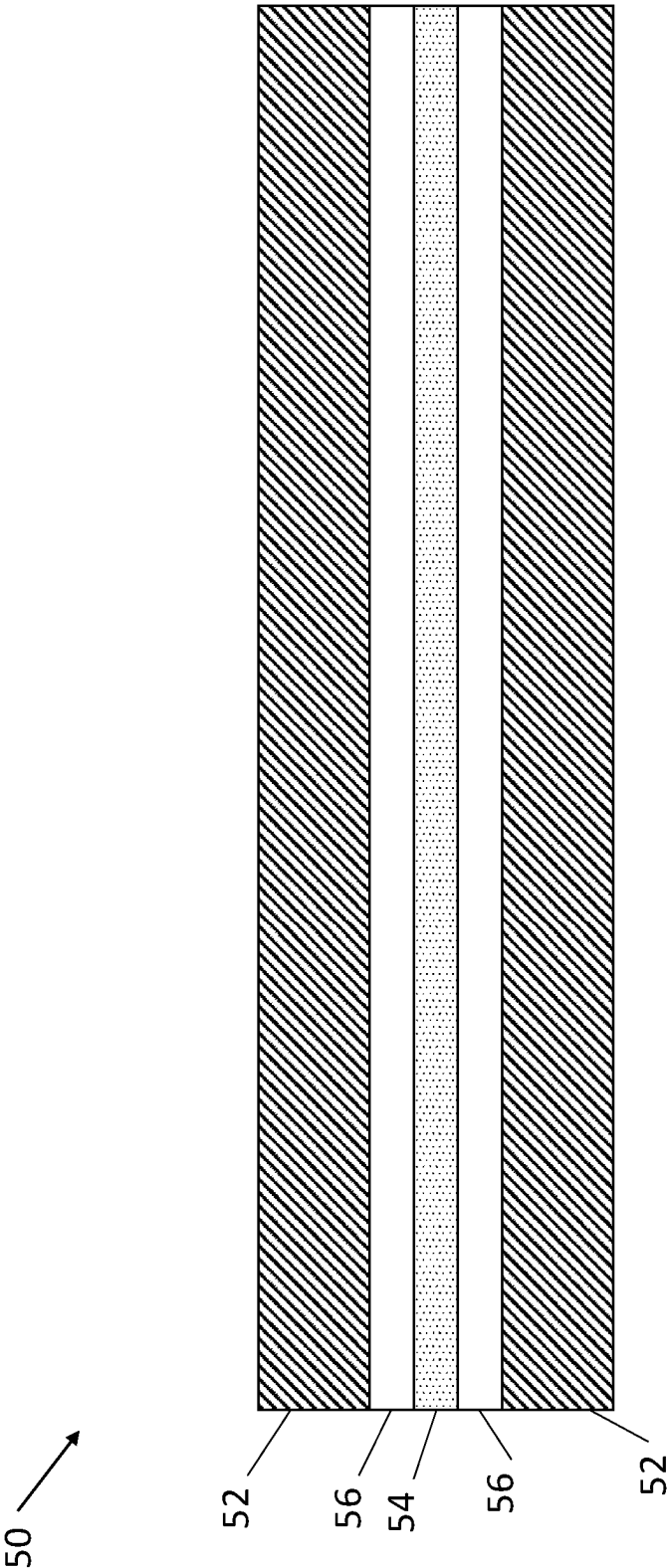


FIG. 3

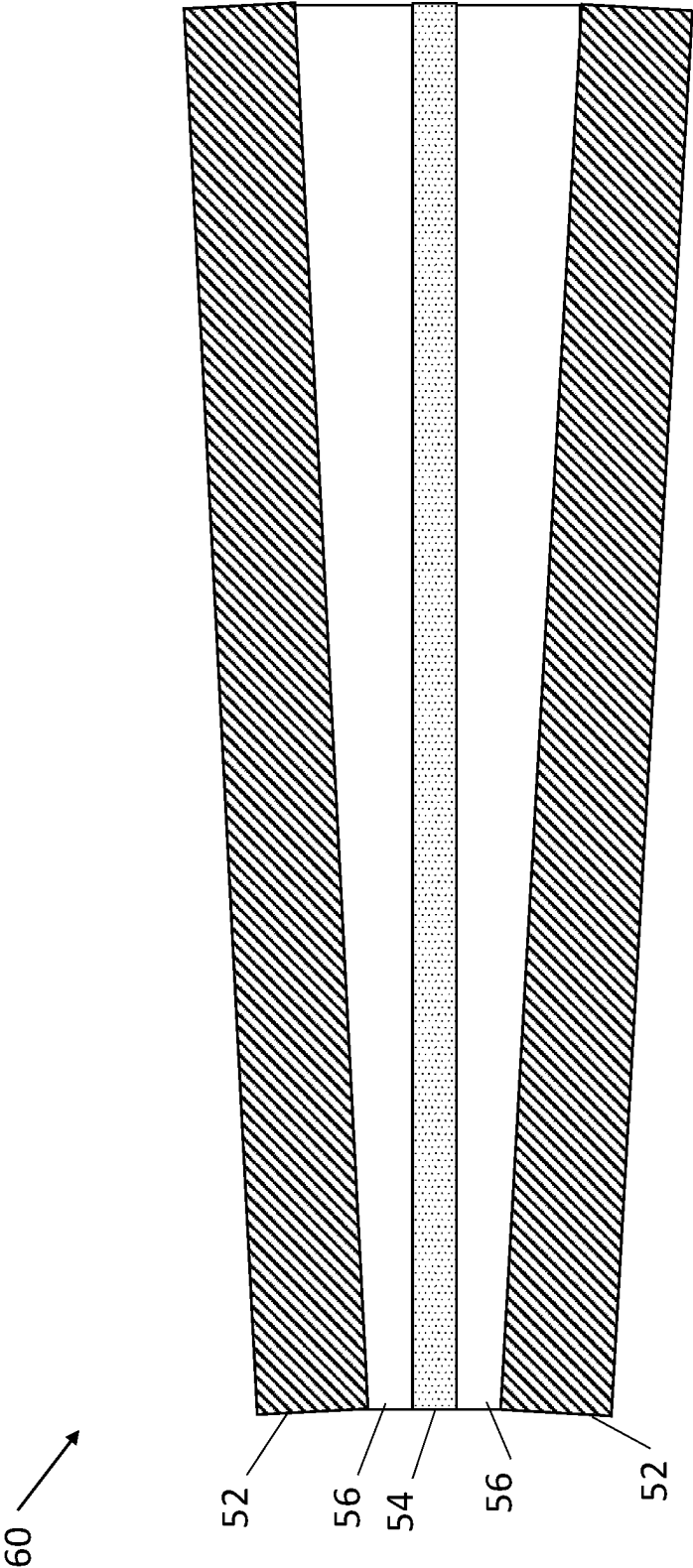


FIG. 4

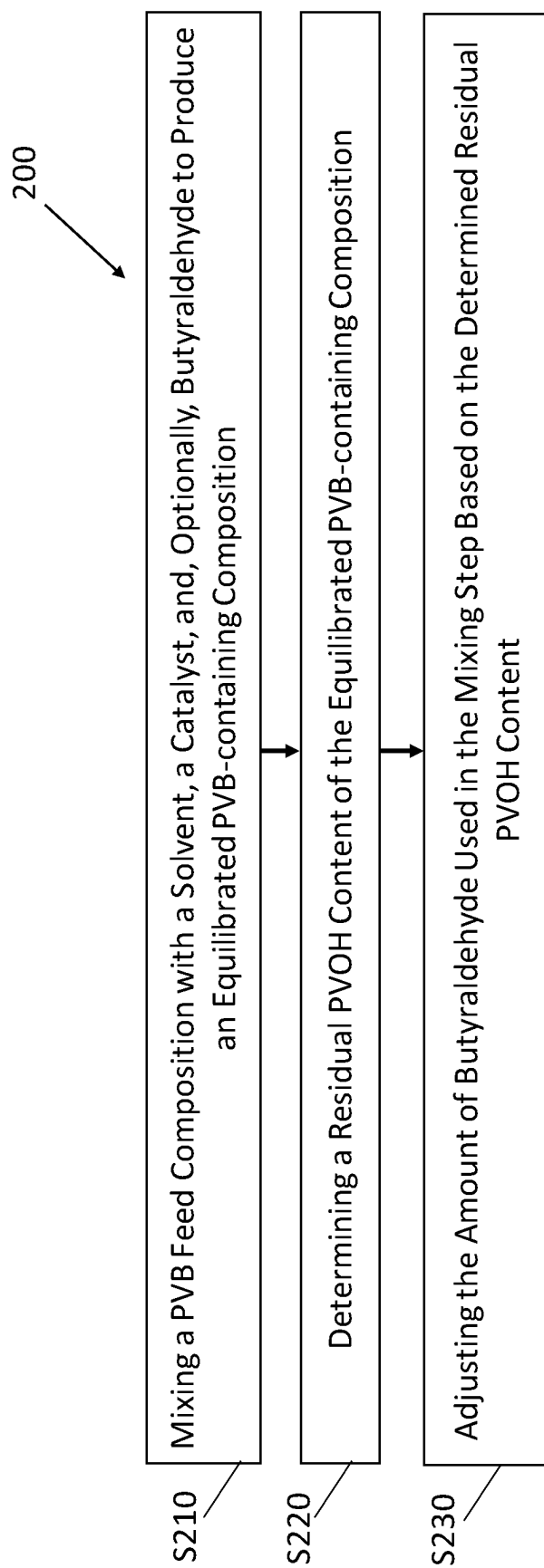


FIG. 5



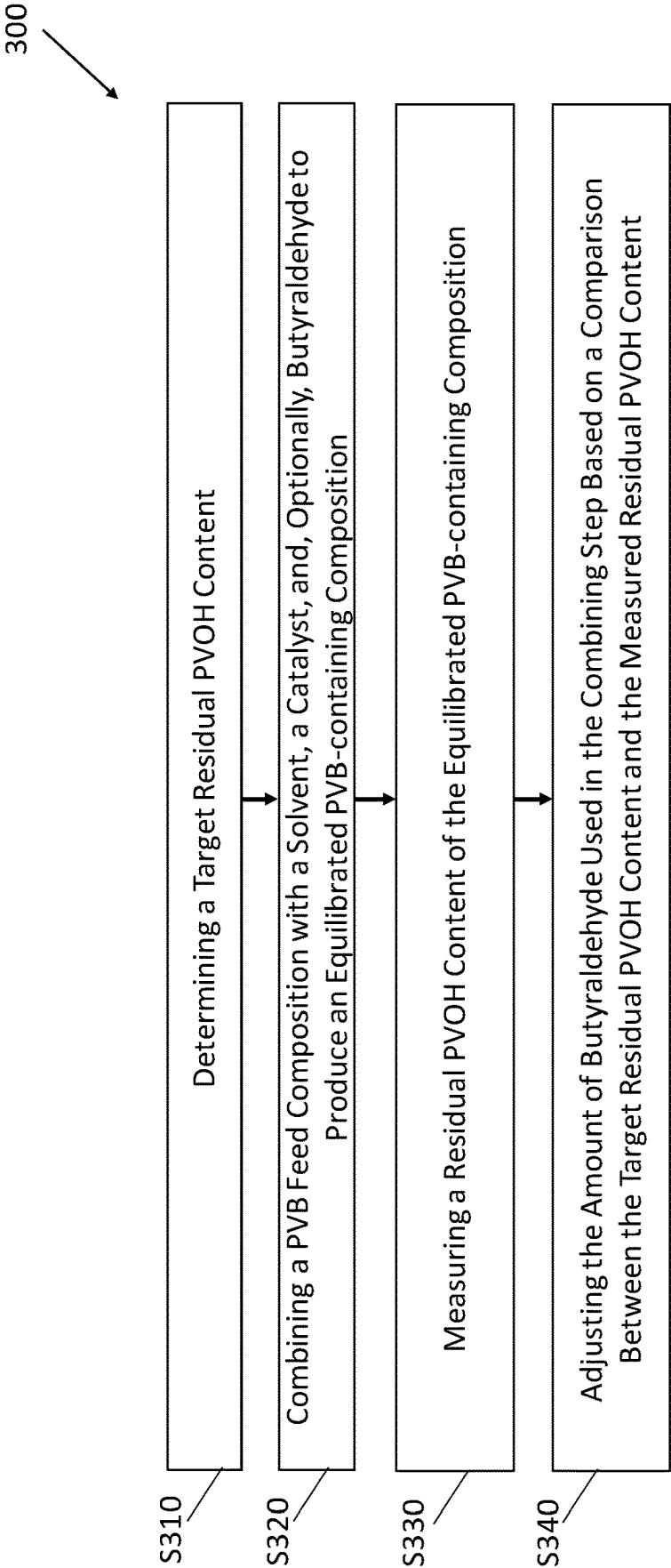


FIG. 6

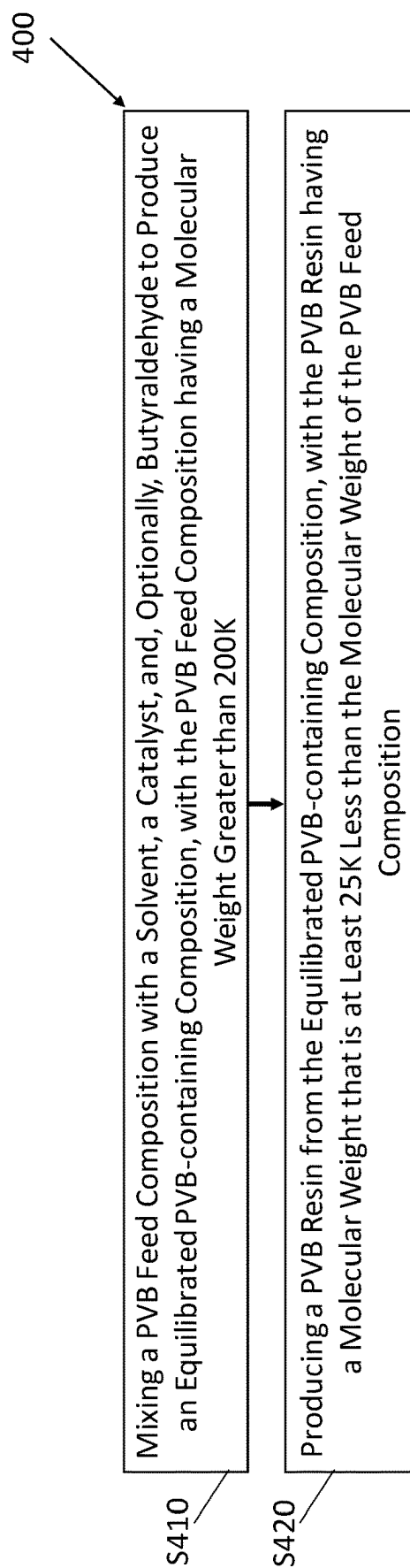


FIG. 7

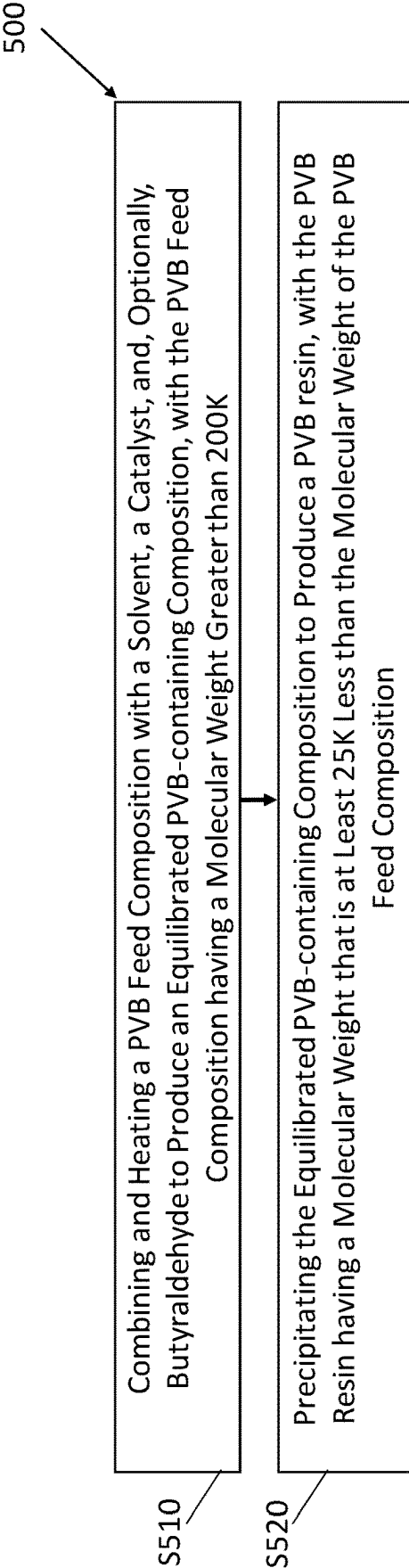


FIG. 8

## RECOVERING POLY(VINYL BUTYRAL) POLYMER

### FIELD OF THE INVENTION

**[0001]** The present invention is related to the field of poly(vinyl butyral) resin manufacture, and, specifically, the present invention is in the field of the recovery, recycling, and reuse of post-industrial and post-consumer poly(vinyl butyral).

### DESCRIPTION OF RELATED ART

**[0002]** Laminated glass panels, such as automobile windshields and architectural safety glass, are typically composed of two sheets of glass laminated together with an interposed, plasticized polymer layer. Poly(vinyl butyral) ("PVB") is a common polymer that typically forms the main component in the polymeric interlayer of the vast majority of automotive windshields and architectural safety glass. Commonly, PVB resin is manufactured through a synthesis process that begins with the separation of ethane directly from natural gas or from the petroleum refining process. Ethane is then steam cracked to produce ethene (ethylene), which, along with acetic acid feedstock, is used to produce vinyl acetate monomers. Vinyl acetate monomers, through free-radical polymerization, are polymerized to poly(vinyl acetate). Poly(vinyl acetate) is hydrolyzed to poly(vinyl alcohol), which is then reacted with butyraldehyde to form poly(vinyl butyral).

**[0003]** The above-described synthesis process is energy intensive and dependent upon the use of non-renewable feedstocks. Consequently, the prospect of recycling PVB resin from post-industrial PVB material and post-consumer PVB material has been long considered in the art as a potentially valuable source of PVB that would be less costly to produce than virgin PVB resin and that could significantly reduce the environmental footprint of PVB production. Exemplary sources of post-industrial PVB material include PVB trim that is recycled and PVB rolls that are out of specification, damaged, or otherwise unusable. Exemplary sources of post-consumer PVB material include previously-used automotive windshields and architectural safety glass, as well as other previously-used consumer products such as electric power devices (e.g., solar photovoltaic devices) and electronic display devices.

**[0004]** Despite the long felt need in the art, there are several problems with recycling PVB. For example, both post-industrial and post-consumer PVB materials generally comprise mixtures of distinct PVB compositions, as obtained from various products and/or from differing manufacturers, as well as different additives such as plasticizer, UV absorbers, solar absorbers and the like. As a result, post-industrial and/or post-consumer PVB mixtures may comprise PVB of different poly(vinyl butyral) compositions, including different polyvinyl alcohol (PVOH) contents. Such compositional differences within the mixtures of recycled PVB invariably lead to unacceptable high haze and/or discoloration of the recovered PVB despite the PVB being removed of other contaminants. Specifically, when PVB materials of significant compositional difference are mixed together, the chemical incompatibility results in a hazy or cloudy material due to immiscible microdomains with different refractive indices, which greatly limits its utility in recycling.

**[0005]** Additionally, when recycling PVB materials having different compositions, it can be difficult to produce a recovered PVB material with a specific composition necessary for particular end-uses. For instance, it may be necessary to recycle a PVB feed composition comprised of two different PVB-containing compositions and to produce a recovered PVB material that can be used as skin layers of an acoustic trilayer interlayer. Alternatively, it may be necessary to use the recovered PVB material as a core layer of an acoustic trilayer interlayer. However, in some interlayers, the residual PVOH content of PVB material used in skin layers is significantly different from the residual PVOH content of PVB material used in core layers.

**[0006]** As such, there exists a need to process post-industrial and/or post-consumer PVB material in a manner that can accurately control the composition (e.g., the residual PVOH content) of the recovered PVB, such that the recovered PVB is produced with a specific composition (e.g., having uniform residual PVOH content) necessary to be used within interlayers of new laminated glass panels.

### SUMMARY

**[0007]** One aspect of the present invention concerns a process of recovering poly(vinyl butyral) (PVB). The process comprises mixing a PVB feed composition with a solvent, a catalyst, and, optionally, butyraldehyde to produce an equilibrated PVB-containing composition. The PVB feed composition comprises at least two different PVB-containing compositions. An additional step includes determining a residual polyvinyl alcohol (PVOH) content of the equilibrated PVB-containing composition. A further step includes adjusting the amount of butyraldehyde used in the mixing step based on the determined residual PVOH content.

**[0008]** Another aspect of the present invention concerns a process of recovering poly(vinyl butyral) (PVB). The process comprises establishing a target residual polyvinyl alcohol (PVOH) content. An additional step includes combining a PVB feed composition with a solvent, a catalyst, and, optionally, butyraldehyde to produce an equilibrated PVB-containing composition. The PVB feed composition comprises at least two different PVB-containing compositions. An additional step includes measuring a residual PVOH content of the equilibrated PVB-containing composition. A further step includes adjusting the amount of butyraldehyde used in the combining step based on a comparison between the target residual PVOH content and the measured residual PVOH content.

**[0009]** Another aspect of the present invention concerns a process of recovering poly(vinyl butyral) (PVB). The process comprises a step of mixing a PVB feed composition with a solvent, a catalyst, and, optionally, butyraldehyde to produce an equilibrated PVB-containing composition. The PVB feed composition has a molecular weight greater than 200K Daltons. An additional step includes producing a PVB resin from the equilibrated PVB-containing composition, with the PVB resin having a molecular weight that is at least 25K less than the molecular weight of the PVB feed composition.

**[0010]** Another aspect of the present invention concerns a process of recovering poly(vinyl butyral) (PVB). The process comprises a step of combining and heating a PVB feed composition with a solvent, a catalyst, and, optionally, butyraldehyde to produce an equilibrated PVB-containing composition. The PVB feed composition has a molecular

weight greater than 200K Daltons. An additional step includes precipitating the PVB-containing composition to produce a PVB resin, with the PVB resin having a molecular weight that is at least 25K less than the molecular weight of the PVB feed composition.

#### BRIEF DESCRIPTION OF THE DRAWINGS

[0011] FIG. 1A is a flowchart of a process for recovering PVB according to embodiments of the present invention;

[0012] FIG. 1B is a flowchart of another process for recovering PVB according to embodiments of the present invention;

[0013] FIG. 2A is a schematic illustration of a PVB reclamation system according to embodiments of the present invention.

[0014] FIG. 2B is a schematic illustration of another PVB reclamation system according to embodiments of the present invention;

[0015] FIG. 3 is a schematic illustration of a laminated glass panel comprising a pair of glass plates opposing a polymer interlayer, with the polymer interlayer comprising a trilayer with a pair of skin layers opposing a core layer;

[0016] FIG. 4 is another schematic illustration of a laminated glass panel comprising a pair of glass plates opposing a polymer interlayer, with the polymer interlayer having a wedge shape;

[0017] FIG. 5 is a flowchart of another process for recovering PVB according to embodiments of the present invention;

[0018] FIG. 6 is a flowchart of yet another process for recovering PVB according to embodiments of the present invention;

[0019] FIG. 7 is a flowchart of yet another process for recovering PVB according to embodiments of the present invention; and

[0020] FIG. 8 is a flowchart of yet another process for recovering PVB according to embodiments of the present invention.

#### DETAILED DESCRIPTION

[0021] Embodiments of the present invention are directed to methods of recovering, recycling, and/or re-using poly (vinyl butyral) ("PVB"). More particularly, embodiments of the present invention are directed to methods of recycling post-industrial and/or post-consumer PVB material to obtain PVB polymer of sufficient quality that can be used to form polymer interlayers and/or laminated glass panels comprising polymer interlayers. In some embodiments, the PVB polymer recycled from post-industrial and/or post-consumer PVB material may be used other applications.

[0022] In more detail, FIG. 1A illustrates an exemplary PVB recovery process 100 for recycling PVB according to embodiments of the present invention. The method includes a step S110 of providing post-industrial and/or post-consumer PVB material to a reclamation system. An exemplary embodiment of a reclamation system 10 is illustrated schematically in FIG. 2A, which may be in the form of a continuous reclamation system of interconnected tanks, in which the various steps and processes discussed herein may take place. It should be understood, however, that embodiments contemplate the use of various reclamation systems, such as a single, batch reactor reclamation system. For instance, a single, batch reactor may comprise a tank in the

form of a continuous stirred-tank reactor (CSTR) or another similar reactor, such as a counter-current screw press extractor or other equipment known to one of skill in the art. One or more (or all) of the steps of the process 100 described herein may be carried out within the single, batch reactor. Alternatively, a continuous reclamation system, as illustrated by the reclamation system 10, may be used, with the continuous reclamation system comprising a plurality of interconnected tanks (e.g., CSTRs) or compartments in a tubular arrangement. In the continuous reclamation system, each of the steps may be independently carried out within one or more of the plurality of tanks or compartments. Benefits of the continuous reclamation system may include higher throughput and higher efficiencies compared to the single, batch reactor.

[0023] The PVB material added to the reclamation system 10 may be referred to herein as a "PVB feed composition," and as noted above may comprise post-industrial and/or post-consumer PVB material. In some embodiments, however, new or virgin PVB material may be used. Nevertheless, the PVB feed composition may be added to a mixing tank 12 of the reclamation system 10. The mixing tank 12 may be configured with various mechanisms necessary to combine, mix, and/or agitate the materials added into the tank 12. In some embodiments, the mixing tank 12 may also be configured to apply heat to the materials added into the mixing tank 12. In some embodiments, a solvent may be added, as a step S120, into the mixing tank 12 of the reclamation system 10 to dissolve the PVB feed composition and to form a PVB solution. In some embodiments, the solvent may be added to the mixing tank 12 prior to the PVB feed composition being added to the mixing tank 12. In other embodiments, the solvent may be added to the mixing tank 12 after the PVB feed composition has been added to the mixing tank 12.

[0024] An additional step S130 may comprise adding a catalyst into the mixing tank 12 to form a PVB reaction mixture from the PVB solution (i.e., the PVB feed composition and solvent). In various embodiments, the catalyst may be added to the mixing tank 12 prior to or after the addition of the PVB feed composition. An additional step S140 may comprise, optionally, adding butyraldehyde, into the mixing tank 12 to aid in forming the PVB reaction mixture from the PVB solution. In various embodiments, the butyraldehyde may be added to the mixing tank 12 prior to or after the addition of PVB feed composition. During and/or prior to step S140, the PVB reaction mixture may be heated within the mixing tank 12 to facilitate mixing and/or to allow some initial equilibration of different PVB-containing compositions within the PVB feed composition. From the mixing tank 12, the PVB reaction mixture may be transferred to a holding tank 14 where the PVB reaction mixture, in step S150, is heated for an amount of time to equilibrate the PVB reaction mixture into a uniform PVB composition. As used herein, the term "equilibrated" is used to mean that the composition that is equilibrated has been processed to at least include a generally uniform residual polyvinyl alcohol (PVOH) content.

[0025] Once the PVB reaction mixture has been equilibrated (so as to form a uniform PVB composition), the equilibrated PVB reaction mixture may be transferred to a filtering tank 16 where the equilibrated PVB reaction mixture may be filtered, in step S160, to remove salts and other remaining solids. Such filtering may be performed by mesh

screens, centrifugal filtration systems, or other suitable methods. In some embodiments, the PVB reaction mixture may undergo centrifugation prior to filtering. In some embodiments, the step S160 may further include the addition of a base to the equilibrated PVB reaction mixture. In such embodiments, the base may be added prior to or after the filtering of the equilibrated PVB reaction mixture. For example, the base may be added prior to filtration and/or centrifugation so as to neutralize the catalyst, such that the catalyst may then be extracted via the filtration and/or centrifugation.

**[0026]** The filtered, equilibrated PVB reaction mixture may be transferred to a solvent column 18, where in step S170, remaining solvent may be extracted, such as through evaporation. In certain embodiments, the solvent may be reused in the PVB recovery process 100 by being added back into the mixing tank 12 from the solvent column 18. With the solvent removed from the equilibrated PVB reaction mixture, the remaining solids comprise solid, equilibrated PVB material, which may be provided to an extruder 20, so as to be used in an extrusion process to form various PVB products, such as in the manufacture of polymer interlayers and/or laminated glass panels comprising polymer interlayers, or to be used in a resin manufacturing process and re-acetalized to form PVB resin. The recovered PVB may also be used in other applications, such as adhesives that can be formed with the recovered PVB, as well as flooring, ceramic compositions, binders, coatings, inks, dispersions, and other applications.

**[0027]** An alternative PVB recovery process 100' for recycling PVB according to embodiments of the present invention is shown in FIG. 1B. The process 100' generally includes the same steps S110-S150 as discussed above with respect to the process 100. The process 100' may be implemented using a reclamation system 10' illustrated schematically in FIG. 2B. In contrast to process 100, however, after the PVB reaction mixture has been equilibrated in process 100' (so as to form a uniform PVB composition) within the holding tank 14, the equilibrated PVB reaction mixture may be transferred to a precipitation tank 22, as illustrated in FIG. 2B, instead of the filtering tank 16. In the precipitation tank 22, solid, equilibrated PVB material may be precipitated out of the equilibrated PVB reaction mixture. In some embodiments, the PVB reaction mixture may be neutralized, via a base, prior to, during, or after precipitation. Regardless, the resulting solid, equilibrated PVB material may be transferred from the precipitation tank 22 to a wash tank 24 for washing to remove remaining salts and other impurities, as provided in step S170' of FIG. 1B. In addition, solvent may be removed and recovered from the wash tank 24, so as to be re-introduced to the mixing tank 12 (e.g., via the solvent column 18) as was previously described.

**[0028]** Remaining with FIG. 2B, from the wash tank 24, the solid, equilibrated PVB material may be transferred to a drying tank 26 where any remaining water and other fluids can be removed. The resulting product is a recovered PVB polymer, which can be provided to the extruder 20, so as to be used in an extrusion process to form various PVB products, such as in the manufacture of polymer interlayers and/or laminated glass panels comprising polymer interlayers, or to be used in a resin manufacturing process and re-acetalized to form PVB resin. The recovered PVB may also be used in other applications, such as adhesives that can

be formed with the recovered PVB, as well as flooring, ceramic compositions, binders, coatings, inks, dispersions, and other applications.

**[0029]** The following description provides further specificity to the PVB recovery processes 100 and/or 100' described above. In some embodiments, the PVB feed composition may comprise at least two different PVB-containing compositions. For example, the PVB feed composition may include a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different PVB content (e.g., based on weight percent (wt. %)) than the second PVB-containing composition. Alternatively, or in addition, the first PVB-containing composition may have a different PVOH content (e.g., based on weight percent (wt. %)) than the second PVB-containing composition. Alternatively, or in addition, the first PVB-containing composition may have a different vinyl acetate content (e.g., based on weight percent (wt. %)) than the second PVB-containing composition. Alternatively, or in addition, the first PVB-containing composition may have a different plasticizer content (e.g., based on parts per hundred resin parts ("phr")) than the second PVB-containing composition. Alternatively, or in addition, the first PVB-containing composition may have a different plasticizer type than the second PVB-containing composition. Alternatively, or in addition, the first PVB-containing composition may have a different molecular weight than the second PVB-containing composition. Alternatively, or in addition, the first PVB-containing composition may have various additional (or fewer) additives than the second PVB-containing composition. Such additives may include, for example, adhesion control agents, UV stabilizers, antioxidants, IR absorbers, and colorants.

**[0030]** Turning first to the PVOH content of the PVB feed composition, the PVB feed composition will commonly have a residual PVOH content from about 9 to about 25 wt. % or more, although other amounts are possible, depending on the specific starting materials. For example, the various PVB-containing compositions of the PVB feed composition may each include a residual PVOH content from about 0 to 8 wt. %, from about 8 to 12 wt. %, from about 12 to 16 wt. %, from about 16 to 20 wt. %, from about 20 to 26 wt. %, from about 26 to 32 wt. %, from about 32 to 40 wt. %, from about 40 to 90 wt. %, or from about 90 to 100 wt. %. Alternatively, or in addition, the various PVB-containing compositions of the PVB feed composition may each include a residual PVOH content of at least 8, at least 9, at least 9.5, at least 10, at least 10.5, at least 11, at least 11.5, at least 12, at least 12.5, at least 13, at least 13.5, at least 14, at least 14.5, at least 15, at least 15.5, at least 16, at least 16.5, at least 17, at least 17.5, at least 18, at least 18.5, at least 19, at least 19.5, at least 20, at least 25, at least 26, at least 30, at least 32, at least 35, at least 40, at least 50, at least 60, at least 70, at least 80, or at least 90 wt. % and/or not more than 100, not more than 90, not more than 80, not more than 70, not more than 60, not more than 50, not more than 40, not more than 35, not more than 32, not more than 30, not more than 25, not more than 22, not more than 20, not more than 19, not more than 18, not more than 17, not more than 16, not more than 15, not more than 14, not more than 13, not more than 12, not more than 11, not more than 10, not more than 9, or not more than 8 wt. %.

**[0031]** As discussed above, the PVB feed composition may comprise a plurality of different PVB-containing com-

positions, with each having different PVOH contents. For instance, a first PVB-containing composition of the PVB feed composition may have a residual PVOH content from about 9 to 15 wt. %, while a second PVB-containing composition of the PVB feed composition may have a residual PVOH content from about 15 to 25 wt. %. Nevertheless, such exemplary PVOH amounts are only provided for illustrative purposes, and other ranges of residual PVOH contents (or different PVOH ranges) may also be possible, depending on the materials used and the sources of the materials. For instance, the plurality of different PVB-containing compositions of a given PVB feed composition may each have any of the residual PVOH contents (or PVOH ranges) listed above in the previous paragraph. Furthermore, in some specific embodiments, the first and second PVB-containing compositions from the PVB feed composition may have a relative difference in residual PVOH content of: at least at least 0.5 wt. %, at least 1 wt. %, at least 2 wt. %, at least 4 wt. %, at least 6 wt. %, at least 8 wt. %, at least 10 wt. %, at least 12 wt. %, at least 14 wt. %, or at least 16 wt. % and/or from 0.5 and 16 wt. %, from 0.5 and 14 wt. %, from 1 and 12 wt. %, from 2 and 10 wt. %, or from 4 and 8 wt. %.

**[0032]** The PVB feed composition may also include varying amounts and types of plasticizer, which is generally used to soften the PVB and/or to lower the glass transition temperature ( $T_g$ ) of the PVB. Contemplated types of plasticizers include, but are not limited to, esters of a polybasic acid, a polyhydric alcohol, triethylene glycol di-(2-ethylbutyrate), triethylene glycol di-(2-ethylhexanoate) (known as “3-GEH”), triethylene glycol diheptanoate, tetraethylene glycol diheptanoate, dihexyl adipate, dioctyl adipate, hexyl cyclohexyladipate, mixtures of heptyl and nonyl adipates, diisononyl adipate, heptylnonyl adipate, dibutyl sebacate, and polymeric plasticizers such as oil-modified sebacic alkyds and mixtures of phosphates and adipates, and mixtures and combinations thereof. In some embodiments, 3-GEH is particularly preferred. Other examples of suitable plasticizers can include, but are not limited to, tetraethylene glycol di-(2-ethylhexanoate) (“4-GEH”), di(butoxyethyl) adipate, and bis(2-(2-butoxyethoxy)ethyl) adipate, dioctyl sebacate, nonylphenyl tetraethylene glycol, and mixtures thereof.

**[0033]** Other suitable plasticizers may include blends of two or more distinct plasticizers, including but not limited to those plasticizers described above. Still other suitable plasticizers, or blends of plasticizers, may be formed from aromatic groups, such polyadipates, epoxides, phthalates, terephthalates, benzoates, toluates, mellitates and other specialty plasticizers. Further examples include, but are not limited to, dipropylene glycol dibenzoate, tripropylene glycol dibenzoate, polypropylene glycol dibenzoate, isodecyl benzoate, 2-ethylhexyl benzoate, diethylene glycol benzoate, propylene glycol dibenzoate, 2,2,4-trimethyl-1,3-pentanediol dibenzoate, 2,2,4-trimethyl-1,3-pentanediol isobutyrate, 1,3-butanediol dibenzoate, diethylene glycol di-o-toluate, triethylene glycol di-o-toluate, dipropylene glycol di-o-toluate, 1,2-octyl dibenzoate, tri-2-ethylhexyl trimellitate, di-2-ethylhexyl terephthalate, bis-phenol A bis(2-ethylhexanoate), ethoxylated nonylphenol, and mixtures thereof. In some embodiments, the plasticizer can be selected from the group consisting of dipropylene glycol dibenzoates, tripropylene glycol dibenzoates, and combinations thereof.

**[0034]** As noted above, the plasticizer content of PVB feed composition (e.g., resin or recycled PVB) will be measured in phr, on a weight per weight basis. For example, if 30 grams of plasticizer is added to 100 grams of polymer resin, the plasticizer content of the resulting plasticized polymer would be 30 phr. The PVB feed composition may have various amounts and/or types of plasticizer. For example, the various PVB-containing compositions of the PVB feed composition may each include a plasticizer content of at least about 0 to 10 phr, from about 10 to 20 phr, from about 20 to 30 phr, from about 30 to 40 phr, from about 40 to 50 phr, from about 50 to 60 phr, from about 60 to 70 phr, from about 70 to 80 phr, from about 80 to 90 phr, or from about 90 to 100 phr. Alternatively, or in addition, the various PVB-containing compositions of the PVB feed composition may each include a plasticizer content of at least about 20 phr, at least about 25 phr, at least about 30 phr, at least about 35 phr, at least about 38 phr, at least about 40 phr, at least about 45 phr, at least about 50 phr, at least about 55 phr, at least about 60 phr, at least about 65 phr, at least about 67 phr, at least about 70 phr, at least about 75 phr of one or more plasticizers.

**[0035]** In some embodiments, the various PVB-containing compositions of the PVB feed composition may each also include not more than about 100 phr, not more than about 85 phr, not more than about 80 phr, not more than about 75 phr, not more than about 70 phr, not more than about 65 phr, not more than about 60 phr, not more than about 55 phr, not more than about 50 phr, not more than about 45 phr, not more than about 40 phr, not more than about 38 phr, not more than about 35 phr, or not more than about 30 phr of one or more plasticizers.

**[0036]** In addition, the PVB feed composition may have various PVB contents. Such PVB contents may be defined as butyral or acetal contents. The various PVB-containing compositions of the PVB feed composition may each include PVB contents and/or butyral/acetal contents of at least about 50 wt. %, at least about 55 wt. %, at least about 60 wt. %, at least about 65 wt. %, at least about 70 wt. %, at least about 75 wt. %, at least 80 wt. %, at least 85 wt. %, at least 90 wt. %, at least 95 wt. %, or up to 100 wt. %, and/or from 50 to 100 wt. %, from 50 to 90 wt. %, from 50 to 80 wt. %, from 50 to 70 wt. %, from 50 to 60 wt. %, from 60 to 100 wt. %, from 60 to 90 wt. %, from 60 to 80 wt. %, from 60 to 70 wt. %, from 70 to 100 wt. %, from 70 to 90 wt. %, from 70 to 80 wt. %, from 80 to 100 wt. %, from 80 to 90 wt. %, or from 90 to 100 wt. %.

**[0037]** The PVB feed composition may have various vinyl acetate contents. The various PVB-containing compositions of the PVB feed composition may each include vinyl acetate contents of up to about 15 wt. %, up to about 10 wt. %, or up to about 5 wt. %, and/or from 5 to 15 wt. %, from 5 to 10 wt. %, or from 10 to 15 wt. %.

**[0038]** In addition, the PVB feed composition may have various molecular weights. The various PVB-containing compositions of the PVB feed composition may each include molecular weights from about 50,000 to about 600,000, about 70,000 to about 450,000, about 100,000 to about 425,000, about 150,000 to 350,000, and/or 200,000 to 300,000 Daltons, as measured by size exclusion chromatography using low angle laser light scattering. As used herein, the term “molecular weight” means the weight average molecular weight. Alternatively, or in addition, certain specific embodiments, the various PVB-containing composi-

tions of the PVB feed composition may each include molecular weights from about 100,000 to about 175,000 Daltons, from about 120,000 to about 150,000 Daltons, from about 200,000 to about 250,000, from about 250,000 to about 350,000 Daltons, and/or from about 275,000 to about 325,000 Daltons.

**[0039]** Furthermore, the PVB feed composition may have various other additives or impurities. The various PVB-containing compositions of the PVB feed composition may each include differing amounts of adhesion control agents (ACAs), UV stabilizers, anti-oxidants, IR absorbers or blockers (e.g., indium tin oxide, antimony tin oxide, lanthanum hexaboride ( $\text{LaB}_6$ ) and cesium tungsten oxide), colorants (e.g., dyes and pigments), anti-blocking agents, flame retardants, processing aides, flow enhancing additives, lubricants, impact modifiers, nucleating agents, thermal stabilizers, UV absorbers, dispersants, surfactants, chelating agents, coupling agents, adhesives, primers, reinforcement additives, and fillers, among other additives known to those of ordinary skill in the art

**[0040]** Before adding the PVB feed composition to the reclamation systems **10** and/or **10'**, as provided in step **S110** in the PVB recovery processes **100** and/or **100'**, the material of the PVB feed composition may be cut, chopped, and/or shredded to form small (such as 2 to 20 mm, although other sizes, including up to 1 cm, can be used depending on the equipment and other factors) diameter chips, granules, pellets or flakes of PVB material. As described above, the PVB feed composition may comprise a plurality of different PVB-containing compositions. As such, the flakes, chips and/or pellets of PVB material may be combined together to form a mixed composition (e.g., having a mixed amount of PVOH or other elements). Such a PVB feed composition may then be provided to the reclamation systems **10** and/or **10'**, which as noted previously, may be in the form of a single, batch reactor recycling system or a continuous reclamation recycling system.

**[0041]** Turning again to step **S120** of the PVB recovery processes **100** and/or **100'**, the solvent that is added to the reclamation systems **10** and/or **10'** may comprise various solvents sufficient to selectively dissolve the components of the PVB feed composition and to form a solution or mixture. Examples of suitable solvents may include a mixture of one or more alcohols, e.g., ethanol, methanol or isopropanol. The solvent is configured to dissolve the PVB feed composition to form a PVB solution that has the form of a hazy, viscous solution. In some embodiments, some amount of water may also be added to the solvent and/or to the PVB feed composition to assist with dissolving of the PVB feed composition. Other alcohols, such as alcohols having more than three (3) carbon atoms, such as n-butanol, or alcohols having more than one hydroxyl group (i.e., diols and triols) can also be used.

**[0042]** Turning to step **S130**, the catalyst may comprise various catalysts, such as sulfuric acid, sulfonic acid, sulfonated acid, methane sulfonic acid, or p-tolylsulfonic acid (p-toluenesulfonic acid). Turning to step **S140**, the butyraldehyde may also be optionally used to assist in forming the PVB reaction mixture and to create the equilibrated PVB. As will be described in more detail below, the inventors have found that the amount of butyraldehyde used in the PVB recovery processes **100** and/or **100'** may affect the residual PVOH content of the resulting, equilibrated PVB. As such, embodiments of the present invention include

the feature of adjusting the amount of butyraldehyde used in the PVB recovery processes **100** and/or **100'** so as to precisely control the amount of residual PVOH of the resulting, equilibrated PVB.

**[0043]** When the above-described materials are added to the mixing tank **12** (i.e., the PVB feed composition, the solvent, the catalyst, and, optionally, butyraldehyde), the mixing tank **12** may combine, mix, and/or agitate the materials so as to form the PVB reaction mixture. In some embodiments, the mixing tank **12** may also be configured to apply heat to the materials added into the mixing tank **12**. Upon adding the catalyst and butyraldehyde, the PVB reaction mixture may comprise about 15 to 25 wt. % PVB material (or from 17 wt. % to 23 wt. %, or from 18 wt. % to 22 wt. %, or about 20 wt. % PVB material), about 75 to 85 wt. % solvent (or from 77 wt. % to 83 wt. %, or from 78 wt. % to 82 wt. %, or about 80 wt. % solvent), about 1 wt. % butyraldehyde (or from 0.25 wt. % to 2.0 wt. %, or from 0.5 wt. % to 1.5 wt. %, or from 0.75 wt. % to 1.25 wt. % butyraldehyde), about 0.3 wt. % water (or from 0.1 wt. % to 0.5 wt. % or from 0.2 wt. % to 0.4 wt. % water), and/or about 0.05 wt. % catalyst. However, as noted above and as discussed in more detail below, the amount of butyraldehyde used in the PVB recovery processes **100** and/or **100'** may be adjusted as required to achieve a target residual PVOH content of the resulting, equilibrated PVB.

**[0044]** In step **S150**, the PVB reaction mixture may be transferred to the holding tank **14** to facilitate equilibration of individual PVB-containing compositions within the PVB feed composition (now in the form of the PVB reaction mixture). In more detail, the PVB reaction mixture may be heated for a period of time. The mixture may be heated to a temperature of between 65° C. and 85° C., between 70° C. and 80° C., between 70° C. and 78° C., or about 78° C. The period of time of heating may be at least 1 hour, at least 2 hours, at least 3 hours, and/or between 1 and 5 hours, between 2 and 4 hours, or about 3 hours. The heating, catalyst, and butyraldehyde permit the PVB reaction mixture to equilibrate the various PVB-containing compositions of the PVB feed composition into a uniform composition. For instance, the PVB reaction mixture will equilibrate such that the PVB portion of the mixture will reach a uniform residual PVOH content.

**[0045]** Turning to step **S160** of the PVB recovery process **100**, the equilibrated PVB reaction mixture is transferred to the filtering tank **16**, where the mixture can be cooled and filtered to remove salt and other impurities. In some embodiments, the PVB reaction mixture may be cooled to a temperature below the temperature to which the mixture was heated in step **S150**. In some embodiments, a base will be added to the equilibrated PVB reaction mixture to neutralize the mixture before filtering. In some embodiments, the base may comprise potassium hydroxide ("KOH"), sodium hydroxide, or the like. In some embodiments, the amount of base added to the mixture may comprise about 0.05 wt. % of the mixture. In some specific embodiments, the amount of base may be at least 0.02 wt. %, at least 0.03 wt. %, at least 0.04 wt. %, at least 0.05 wt. %, at least 0.06 wt. %, at least 0.07 wt. %, at least 0.08 wt. % of the equilibrated PVB reaction mixture. In other embodiments, the base may form from 0.02 wt. % to 0.08 wt. %, from 0.03 wt. % to 0.07 wt. %, from 0.04 wt. % to 0.06 wt. %, or about 0.05 wt. % of the equilibrated PVB reaction mixture. Regardless, embodiments may provide for a sufficient amount of base to be



added such that the pH of the equilibrated PVB reaction mixture (which is about 2.0 to 2.5 before neutralization) reaches from about 5.0 to about 7.0, or at least about 5, 5.1, 5.2, 5.3, 5.4, 5.5, 5.6, 5.7, 5.8, 5.9, 6.0, 6.1, 6.2, 6.3, 6.4, 6.5, 6.6, 6.7, 6.8, 6.9 or 7.0, or about 5.1 to about 6.9, or about 5.2 to about 6.8, or about 5.3 to about 6.7, or about 5.4 to about 6.7, or about 5.5 to about 6.7, or about 5.6 to about 6.7, or about 5.7 to about 6.7, or about 5.8 to about 6.7, or about 5.9 to about 6.7.

**[0046]** The step S160 may further comprise centrifuging and filtering the equilibrated PVB reaction mixture (e.g., neutralized as described above) to remove salts and other solids from the mixture. In some embodiments, the filter may comprise a screen, mesh, cloth, or other similar filtering element. The filtered, equilibrated PVB reaction mixture may, in step S170, be provided to the solvent column 18, where the solvent may be extracted (e.g., via evaporative distillation, water-vapor stripping, extrusion evaporation, or the like). In certain embodiments, the solvent may be reused in the PVB recovery process 100 by being added back into the mixing tank 12 from the solvent column 18. With the solvent and catalyst removed, the remaining solids comprise solid, equilibrated PVB, which may be provided to the extruder 20 (e.g., for use in the formation of polymer interlayers).

**[0047]** Alternatively, as provided in the PVB recovery process 100', after the PVB reaction mixture has been equilibrated (so as to form a uniform PVB composition) within the holding tank 14, the equilibrated PVB reaction mixture may be transferred to the precipitation tank 22, where solid PVB may be precipitated out of the equilibrated PVB reaction mixture, as provided in step S160'. In some embodiments, the equilibrated PVB reaction mixture may be neutralized with the base prior to, during, or after precipitation. The resulting solid, equilibrated PVB may be transferred from the precipitation tank 22 to the wash tank 24 for removing salts and other impurities, as provided in step S170'. In addition, the solvent may be recovered from the wash tank 24 and re-introduced to the mixing tank 12 (e.g., via the solvent column 18) as was previously described. Specifically, the solvent may be removed from the wash tank 24 to the solvent column 18 (e.g., via evaporative distillation, water-vapor stripping, extrusion evaporation, or the like).

**[0048]** From the wash tank 24, the solid, equilibrated PVB may be transferred to a drying tank 26 where any remaining water and other fluids can be removed. The resulting product is a recovered PVB polymer (which has been equilibrated so as to have a uniform residual PVOH content), which can be provided to the extruder 20, so as to be used in an extrusion process to form various PVB products, such as in the manufacture of polymer interlayers and/or laminated glass panels comprising polymer interlayers, or to be used in a resin manufacturing process and re-acetalized to form PVB resin. The recovered PVB may also be used in other applications, such as adhesives that can be formed with the recovered PVB, as well as flooring, ceramic compositions, binders, coatings, inks, dispersions, and other applications.

**[0049]** In certain embodiments, variations, additions, and/or subtractions may be made to the steps S110 to S170 of process 100 (or steps S110 to S170' of process 100') described above. Additionally, the recovered PVB polymer may be subjected to further treatment, as required for particular end uses.

**[0050]** The PVB that is recovered using the PVB recovery processes 100 and/or 100' described above may be used to form a resin, resin layer, polymer interlayer and/or a laminated glass panel comprising a polymer interlayer. The terms "polymer interlayer sheet," "interlayer," "polymer layer", and "polymer melt sheet" as used herein, may designate a single-layer sheet or a multilayered interlayer. A "single-layer sheet," as the name implies, is a single polymer layer extruded as one layer. A multilayered interlayer, on the other hand, may comprise multiple layers, including separately extruded layers, co-extruded layers, or any combination of separately and co-extruded layers. Thus, the multilayered interlayer could comprise, for example: two or more single-layer sheets combined together ("plural-layer sheet"); two or more layers co-extruded together ("co-extruded sheet"); two or more co-extruded sheets combined together; a combination of at least one single-layer sheet and at least one co-extruded sheet; and a combination of at least one plural-layer sheet and at least one co-extruded sheet. In various embodiments of the present invention, a multilayered interlayer comprises at least two polymer layers (e.g., a single layer or multiple layers co-extruded) disposed in direct contact with each other, wherein each layer comprises a polymer resin. The term "resin," as utilized herein refers to the polymeric component (e.g., PVB) removed from the mixture that results from the acid catalysis and subsequent neutralization of polymeric precursors. Generally, plasticizer, such as those discussed more above, will be added to the resins to result in a plasticized polymer. Additionally, resins may have other components in addition to the polymer and plasticizer including; e.g., acetates, salts, and alcohols.

**[0051]** Although the above described steps of the PVB recovery process 100 can be performed to yield recovered PVB with at least some inherent plasticizer (e.g., 3 to 5 phr of plasticizer, 10 to 20 phr of plasticizer, 35 to 45 phr of plasticizer, or various other amounts of plasticizer), embodiments may require additional plasticizer to be added to the PVB before the PVB is used to form a polymer interlayer and/or a laminated glass panel comprising a polymer interlayer. For example, in some embodiments, an additional amount of from 25 to 50 phr, 25 to 45 phr, 30 to 40 phr, or 33 to 35 phr may be added to the resulting PVB before the polymer interlayer and/or the laminated glass panel is manufactured. In other embodiments, an additional amount of less than 25 phr, or less than 20 phr or less, or greater than 50 phr, or greater than 55 phr, or greater than 60 phr, or greater than 65 phr, or greater than 70 phr or more may be added to the resulting PVB before the polymer interlayer and/or the laminated glass panel is manufactured. Higher or lower amounts of plasticizer may be added as desired, depending on the desired properties and application. The recovered PVB resin (or resins) typically has a molecular weight of greater than 30,000 Daltons, or less than 500,000 Daltons, or from about 30,000 to about 500,000 Daltons, or from about 100,000 to about 400,000 Daltons, or from 100,000 to about 175,000 Daltons, or from about 200,000 to about 250,000 Daltons or from about 250,000 to about 350,000 Daltons.

**[0052]** Once a sufficient amount of plasticizer is added to the recovered PVB, it is contemplated that polymer interlayer sheets may be produced by any suitable process known to one of ordinary skill in the art of producing polymer interlayer sheets that are capable of being used in a multiple layer panel (such as a glass laminate). For example, it is

contemplated that the polymer interlayer sheets may be formed through solution casting, compression molding, injection molding, melt extrusion, melt blowing or any other procedures for the production and manufacturing of a polymer interlayer sheet known to those of ordinary skill in the art. Further, in embodiments where multiple polymer interlayers are utilized, it is contemplated that these multiple polymer interlayers may be formed through co-extrusion, blown film, dip coating, solution coating, blade, paddle, air-knife, printing, powder coating, spray coating or other processes known to those of ordinary skill in the art. While all methods for the production of polymer interlayer sheets known to one of ordinary skill in the art are contemplated as possible methods for producing the polymer interlayer sheets described herein, this application will focus on polymer interlayer sheets produced through extrusion and/or co-extrusion processes.

**[0053]** In the extrusion process, thermoplastic resin and plasticizers, including any of those resins and plasticizers described above, are generally pre-mixed and fed into an extruder device. Additives such as colorants and UV inhibitors (in liquid, powder, or pellet form) may be used and can be mixed into the thermoplastic resin or plasticizer prior to arriving in the extruder device. These additives are incorporated into the thermoplastic polymer resin, and by extension the resultant polymer interlayer sheet, to enhance certain properties of the polymer interlayer sheet and its performance in the final multiple layer glass panel product.

**[0054]** In the extruder device, the particles of the thermoplastic raw material and plasticizers, including any of those resins, plasticizers, and other additives described above, are further mixed and melted, resulting in a melt that is generally uniform in temperature and composition. Embodiments of the present invention may provide for the melt temperature to be approximately 200° C. Once the melt reaches the end of the extruder device, the melt is propelled into the extruder die. The extruder die is the component of the extruder device which gives the final polymer interlayer sheet product its profile. The die will generally have an opening, defined by a lip, that is substantially greater in one dimension than in a perpendicular dimension. Generally, the die is designed such that the melt evenly flows from a cylindrical profile coming out of the die and into the product's end profile shape. A plurality of shapes can be imparted to the end polymer interlayer sheet by the die so long as a continuous profile is present. Generally, in its most basic sense, extrusion is a process used to create objects of a fixed cross-sectional profile. This is accomplished by pushing or drawing a material through a die of the desired cross-section for the end product.

**[0055]** In some embodiments, a co-extrusion process may be utilized. Co-extrusion is a process by which multiple layers of polymer material are extruded simultaneously. Generally, this type of extrusion utilizes two or more extruders to melt and deliver a steady volume throughput of different thermoplastic melts of different viscosities or other properties through a co-extrusion die into the desired final form. For example, the multiple layer interlayers of the present invention (e.g., in the form of a trilayer interlayer) may be preferably co-extruded using a multiple manifold co-extrusion device which includes a first die manifold, a second die manifold, and a third die manifold. The co-extrusion device may operate by simultaneously extruding polymer melts from each manifold through a die and out of

an opening, where the multiple layer interlayer is extruded as a composite of three individual polymer layers. The polymer melts may flow through the die such that the core layer is positioned between the skin layers, so as to result in the manufacture of a trilayer interlayer with the core layer sandwiched between the skin layers. The die opening may include a pair of lips positioned on either side of the opening. Given the positional orientation of the polymer melts, the skin layers may come into contact with the lips. Regardless, the interlayer thickness can be varied by adjusting the distance between die lips located at the die opening.

**[0056]** Often, polymer interlayers having three layers will be used in the manufacture of a laminated glass panel. For example, in some embodiments of this application, the increased acoustic attenuation properties of soft layers are combined with the mechanical strength of stiff/rigid layers to create a multilayered interlayer. In these embodiments, a central soft layer is sandwiched between two stiff/rigid outer layers. This configuration of (stiff)/(soft)/(stiff) creates a multilayered interlayer that is easily handled, can be used in conventional lamination methods and that can be constructed with layers that are relatively thin and light. The soft, core layer is generally characterized by a lower residual hydroxyl content, a higher plasticizer content, and/or a lower glass transition temperature than the relatively stiffer, skin layers.

**[0057]** The following offers a simplified description of the manner in which multiple layer glass panels are generally produced in combination with the interlayers formed according to the processes described above. First, as discussed above, a multiple layer interlayer may be co-extruded using a multiple manifold co-extrusion device. The device operates by simultaneously extruding polymer melts from each manifold toward an extrusion opening. Properties of the layers can be varied by adjusting attributes (e.g., temperature and/or opening dimensions) of the die lips at the extrusion opening. Once formed, the interlayer sheet can be placed between two glass substrates and any excess interlayer is trimmed from the edges, creating an assembly. It is not uncommon for multiple polymer interlayer sheets or a polymer interlayer sheet with multiple layers (or a combination of both) to be placed within the two glass substrates creating a multiple layer glass panel with multiple polymer interlayers. Then, air is removed from the assembly by an applicable process or method known to one of skill in the art; e.g., through nip rollers, vacuum bag or another deairing mechanism. Additionally, the interlayer is partially press-bonded to the substrates by any method known to one of ordinary skill in the art. In a last step, in order to form a final unitary structure, this preliminary bonding is rendered more permanent by a high temperature and pressure lamination process, or any other method known to one of ordinary skill in the art such as, but not limited to, autoclaving.

**[0058]** In view of the above, multiple layer panels may be comprised of two sheets of glass, or other applicable substrates, with a polymer interlayer sheet or sheets sandwiched therebetween. Multiple layer panels are generally produced by placing at least one polymer interlayer sheet between two substrates to create an assembly. FIG. 3 illustrates a multiple layer panel 50 comprising a pair of glass sheets 52 with a multilayered interlayer sandwiched therebetween. The multilayered interlayer is configured as an acoustic trilayer interlayer having three individual polymer interlayer sheets, including a soft core layer 54 and two relatively stiffer skin

layers **56** positioned on either side of the core layer **54**. Such glass panels that incorporate such a trilayer may, as discussed above, have superior acoustic properties due to the sound dampening provided by the soft, core layer.

[0059] In some embodiments, the interlayer (e.g., the core layer **54** and the skin layers **56**) will have a generally constant or uniform thickness about the length of the interlayer (see, e.g., FIG. 3). However, in alternative embodiments, as shown in a multiple layer panel **60** of FIG. 4, the interlayer may have at least one region of non-uniform thickness. For example, the interlayer, comprised of the core layer **54** and skin layers **56**, may be wedge-shaped, such that the thickness of the interlayer changes (e.g., linearly) about the length of the interlayer. In some such embodiments, the thickness of the interlayer may change due to a thickness change in the core layer **54** (i.e., with the skin layers **56** having a generally constant thickness). Alternatively, the thickness of the interlayer may change due to a thickness change in the skin layers **56** (i.e., with the core layer **54** having a generally constant thickness). In further alternatives, the thickness of the interlayer may change due to a thickness change in both the core layer **54** and the skin layers **56**. In further embodiments (not shown), one or more layers may increase in thickness in the cross machine direction of the interlayer while one or more layers are concurrently reduced in thickness, while maintaining an interlayer having at least one region of non-uniform thickness, such as a wedge-shaped interlayer. Such glass panels that incorporate such a trilayer may, as discussed above, have superior acoustic properties due to the sound dampening provided by the soft, core layer. In addition, due to the non-uniform thickness of the trilayer, the glass panels may provide beneficial characteristics for use in heads-up displays (“HUDs”) by reducing unwanted image projection defects (e.g., reducing ghost images).

[0060] Beneficially, laminated glass panels formed with at least one polymer layer/interlayer containing the recovered PVB, obtained from the PVB recovery processes **100** and/or **100'** discussed above, may have superior optical qualities. Such qualities are generally due to the recovered PVB being equilibrated so as to have a uniform residual PVOH content. For example, when the recovered PVB (i.e., the equilibrated PVB-containing composition) is mixed with 20 phr of plasticizer to form a PVB resin, and the PVB resin is formed into a 0.772 mm PVB layer and positioned between two 2.3 mm sheets of glass to form a laminated glass panel, the laminated glass panel may have a haze of less than 5%, less than 2%, less than 1%, less than 0.5%, or less than 0.25%. Haze is a percentage of transmitted light that is scattered so that its direction deviates more than a specified angle from the direction of the incident beam. Haze may be measured using a haze meter or a spectrophotometer, known to one of skill in the art, and in accordance with ASTM D1003-Procedure B using Illuminant C, at an observer angle of 2 degrees.

[0061] However, it can be difficult to produce polymer layers/interlayers with the necessary residual PVOH content to be formed into specific layers of polymer interlayers. For instance, as was noted above, for acoustic trilayer interlayers, the soft, core layer is generally characterized by a lower residual PVOH content, whereas the relatively stiffer, skin layers are generally characterized by a higher residual PVOH content. Given that the PVB feed composition used in the PVB recovery processes **100** and/or **100'** may com-

prise multiple different PVB-containing compositions, the recovered PVB (after being recycled through the PVB recovery processes **100** and/or **100'** described above) may be formed with various levels of residual PVOH content, which may not be suitable for use as skin layers or as core layers in a trilayer interlayer. However, the inventors have discovered that by controlling the amount of butyraldehyde used in the PVB recovery processes **100** and/or **100'**, the residual PVOH content of the recovered PVB can be precisely controlled. For example, by increasing the amount of butyraldehyde used in the PVB recovery processes **100** and/or **100'**, the residual PVOH content of the recovered PVB can be reduced, whereas by decreasing the amount of butyraldehyde used in the PVB recovery processes **100** and/or **100'**, the residual PVOH content of the recovered PVB can be increased.

[0062] In view of the above, as illustrated in FIG. 5, embodiments of the present invention include a further PVB recovery process **200** for controlling the amount of residual PVOH content of recovered PVB. The process **200** comprises an initial step **S210** of mixing a PVB feed composition with a solvent, a catalyst, and, optionally, butyraldehyde to produce an equilibrated PVB-containing composition. In some embodiments, the PVB feed composition comprises at least two different PVB-containing compositions. An additional step **S220** includes determining a residual PVOH content of the equilibrated PVB-containing composition. A further step **S230** includes adjusting the amount of butyraldehyde used in the mixing step **S210** based on the determined residual PVOH content.

[0063] In more detail, the PVB feed composition may be mixed, as provided in step **210**, with the solvent, catalyst, and, optionally, butyraldehyde within the mixing tank **12** and/or the holding tank **14** of the reclamation system **10**, as previously described with respect to the PVB recovery processes **100** and/or **100'**, to produce an equilibrated PVB-containing composition. As was also previously noted, the PVB feed composition may comprise at least two different PVB-containing compositions, such as a first PVB-containing composition having a different residual PVOH content than a second PVB-containing composition. Nevertheless, once the PVB feed composition has been equilibrated, the residual PVOH content of the equilibrated PVB-containing composition may be uniform and may be determined, as provided in step **S220**.

[0064] In some embodiments, the residual PVOH content may be determined when the equilibrated PVB-containing composition is in solution form, such as when the equilibrated PVB-containing composition is within the holding tank **14**, the filtering tank **16**, and/or the precipitation tank **22**. In some embodiments, the residual PVOH content of the equilibrated PVB-containing composition in solution form may be tested by cloud point measurement. Such cloud point measurement may be performed on samples of the equilibrated PVB-containing composition solution extracted from the holding tank **14**, the filtering tank **16**, and/or the precipitation tank **22**. Alternatively, the cloud point measurement may be performed directly on the equilibrated PVB-containing composition solution within the holding tank **14**, the filtering tank **16**, and/or the precipitation tank **22**.

[0065] In other embodiments, the residual PVOH content may be determined when the equilibrated PVB-containing composition is in solid or resin form, such as when solid PVB has been obtained from the equilibrated PVB reaction

mixture within the solvent column 18, within the precipitation tank 22 (e.g., after precipitation), within the wash tank 24 (e.g., after evaporation), within the drying tank 26, and/or otherwise after the recovered PVB has been formed into a resin for introduction into the extruder 20. In some embodiments, the residual PVOH content of the equilibrated PVB-containing composition in solid or resin form may be tested by an infrared sensor and/or by titration. Such testing may be performed on samples of the equilibrated PVB-containing composition (in solid or resin form) extracted from the solvent column 18, from the precipitation tank 22, from the wash tank 24 from the drying tank 26, and/or otherwise prior to introduction into the extruder 20. Alternatively, the testing may be performed directly on the equilibrated PVB-containing composition (in solid or resin form) within the solvent column 18, within the precipitation tank 22, within the wash tank 24, within the drying tank 26, and/or within feed lines supplying the PVB resin to the extruder 20.

**[0066]** Based on the measured residual PVOH content of the equilibrated PVB, embodiments provide for the residual PVOH content to be adjusted. As noted above, the inventors have found that by increasing the amount of butyraldehyde used in the PVB recovery processes 100 and/or 100', the residual PVOH content of the recovered PVB can be reduced, whereas by decreasing the amount of butyraldehyde used in the PVB recovery processes 100 and/or 100', the residual PVOH content of the recovered PVB can be increased. Thus, a target residual PVOH content of the equilibrated PVB can be established, and the amount of butyraldehyde used in the PVB recovery processes 100 and/or 100' can be adjusted to achieve such target.

**[0067]** In some embodiments, the target residual PVOH content of the equilibrated PVB may be set from 14 to 50 wt. %, from 15 to 25 wt. %, from 16 to 22 wt. %, or from 18.0 to 20.0 wt. %. Such a target may be used, for example, for embodiments in which the equilibrated PVB is to be formed into a resin used in skin layers of an acoustic trilayer. In other embodiments, the target residual PVOH content of the equilibrated PVB may be set from 5 to 15 wt. %, from 7 to 13 wt. %, or from 9 to 11 wt. %. Such a target may be used, for example, for embodiments in which the equilibrated PVB is to be formed into a resin used in a core layer of an acoustic trilayer.

**[0068]** The amount of butyraldehyde used in the PVB recovery process 100 can be adjusted by modifying the amount of butyraldehyde used in the mixing tank 12. For example, in situations where the residual PVOH content of the equilibrated PVB, e.g., as recovered from the PVB recovery process 100, is greater than the target, the amount of butyraldehyde used in the mixing tank 12 may be increased (i.e., butyraldehyde is added to the mixing tank 12 from the solvent column 18 or from a fresh butyraldehyde source). In contrast, in situations where the residual PVOH content of the equilibrated PVB, e.g., as recovered from the PVB recovery processes 100 and/or 100', is less than the target, the amount of butyraldehyde used in the mixing tank 12 may be decreased (i.e., butyraldehyde may be extracted from the mixing tank 12).

**[0069]** In some embodiments, one or more of (i) the determination of residual PVOH content of the equilibrated PVB, (ii) the comparison with the target residual PVOH content, and (iii) the adjustment of the amount butyraldehyde used in the PVB recovery process may be automated. For example, embodiments may include a control system

(e.g., including memory elements, processing elements, and/or input/output elements) for automatically (i) determining the residual PVOH content of the equilibrated PVB within the PVB recovery process, (ii) comparing the determined residual PVOH content with a target residual PVOH content, and (iii) automatically adjusting the amount of butyraldehyde amount used in the PVB recovery process. As such, the control system may be part of the reclamation systems 10 and/or 10', and may be in communication with testing equipment (e.g., sensors) that perform the testing (e.g., cloud point, infrared, titration testing) of the residual PVOH content of the equilibrated PVB within the systems 10 and/or 10'. Upon receiving data indicative of the residual PVOH content of the equilibrated PVB, the control system may compare such data with the target residual PVOH content that is established and stored within the control system. Based on such a comparison, the control system may automatically adjust the amount of butyraldehyde used in the PVB recovery process (e.g., the amount used in the mixing tank 12 of the systems 10 and/or 10'). The control system may perform such steps based on the processing elements performing computer-implemented instructions stored on the memory elements (e.g., non-transitory computer readable storage media) of the control system.

**[0070]** In view of the above, embodiments provide another process 300, as illustrated in FIG. 6, of recovering PVB. The process 300 comprises a step 310 of determining a target residual PVOH content. An additional step 320 includes combining a PVB feed composition with a solvent, a catalyst, and, optionally, butyraldehyde to produce an equilibrated PVB-containing composition. The PVB feed composition may comprises at least two different PVB-containing compositions. An additional step 330 includes measuring a residual PVOH content of the equilibrated PVB-containing composition. A further step 340 includes adjusting the amount of butyraldehyde used in the combining step 310 based on a comparison between the target residual PVOH content and the measured residual PVOH content.

**[0071]** As a further benefit, the recovered PVB obtained from the PVB recovery processes 100 and/or 100' discussed above, may be formed with preferred molecular weight values for use in acoustic trilayer interlayers. In more detail, the inventors have found that recovered PVB obtained from the PVB recovery processes 100 and/or 100' can have a reduced molecular weight with respect to the PVB feed composition fed into the process. In some instances, the PVB feed composition may comprise recycle PVB formed from acoustic trilayer interlayers. Such acoustic trilayer interlayers may comprise a combination of skin layers and core layers, with the core layers generally having higher molecular weights than the skin layers. If the recovered PVB is to be used in the skin layers of new acoustic trilayer interlayers, then the molecular weight of the recovered PVB is generally required to be less than the PVB feed composition. Beneficially, the PVB recovery processes 100 and/or 100' provides for the recovered PVB to have a reduced molecular weight with respect to the PVB feed composition due to the steps of the PVB recovery process 100 unwinding, dissolving, and/or breaking the interchain linkages of the PVB feed composition. A further benefit of the recovered PVB having a reduced molecular weight (with respect to the PVB feed composition) is that the recovered PVB also has a lower viscosity (with respect to the PVB feed composi-

tion), which improves flow of the PVB resin through the extruder and/or the autoclave.

[0072] In view of the above, embodiments of the present invention provide another process 400, as illustrated in FIG. 7, for recovering PVB. The process 400 includes an initial step S410 of mixing a PVB feed composition with a solvent, a catalyst, and, optionally, butyraldehyde to produce an equilibrated PVB-containing composition, with the PVB feed composition having a molecular weight greater than 200,000 (200K) Daltons. In other embodiments, the PVB feed composition may have a molecular weight of greater than 225K, greater than 250K, greater than 275K, and/or greater than 300K Daltons. In addition, the PVB feed composition may have a viscosity of greater than 230 cps. An additional step S420 may comprise producing a PVB resin from the equilibrated PVB-containing composition, with the PVB resin having a molecular weight that is at least 25K less than the molecular weight of the PVB feed composition. In other embodiments, the PVB resin may have a molecular weight that is at least 30K, at least 40K, at least 50K, at least 60K, at least 70K, at least 80K, at least 90K, or at least 100K Daltons less than the molecular weight of the PVB feed composition. Furthermore, the PVB resin may have a viscosity at least 10 cps, at least 15 cps, and/or at least 20 cps less than the viscosity of the PVB feed composition. Alternatively, or in addition, the PVB resin may have a viscosity at least 10%, at least 15%, at least 20%, or between 10 and 20% less than the viscosity of the PVB feed composition. For example, the PVB resin may have a viscosity of less than 230 cps, less than 220 cps, less than 210 cps, and/or less than 200 cps.

[0073] Finally, embodiments of the present invention may provide another process 500, as illustrated in FIG. 8, for recovering PVB. The process 500 includes an initial step S510 of combining and heating a PVB feed composition with a solvent, a catalyst, and, optionally, butyraldehyde to produce an equilibrated PVB-containing composition, with the PVB feed composition having a molecular weight greater than 200K Daltons. An additional step S520 may comprise precipitating the equilibrated PVB-containing composition to produce a PVB resin, with the PVB resin having a molecular weight that is at least 25K less than the molecular weight of the PVB feed composition.

#### Example 1

[0074] A mixture of 850 parts of SD29 alcohol (190 proof ethanol), 150 parts of PVB resin having a PVOH content of 10.50 wt. % (referred to herein as the “starting PVB resin”), 3 parts water, and 0.4 parts sulfuric acid were charged within a one liter three-neck jacketed glass reactor. The mixture was stirred and heated to 78° C., where the mixture was held for four hours. The resulting mixture was cooled to 65° C. and neutralized with 0.46 part of KOH until the mixture reached a pH of 6. The resulting cloudy mixture was further mixed with 8 equivalents of water in a high intensity mixer to form a PVB slurry. Alcohol and residual butyraldehyde were removed from the PVB slurry with fill and drain washing with deionized water, and the resulting slurry was filtered and dried. After drying, 134 parts of “recovered PVB resin” was obtained. The recovered PVB resin had a PVOH content of 18.38 wt. %, as measured using a Bomem MB160 FT-(near infrared) NIR Analyzer. It is noted that the Bomem

MB160 FT-NIR Analyzer was calibrated using a sample PVB resin with a known residual PVOH content determined using titration.

[0075] In addition, each of the starting PVB resin and the recovered PVB resin underwent viscosity measurements according to the following “viscosity test.” To begin, 50 ml of 92 wt. % isopropanol in deionized water at 20° C. was pipetted into a 4 oz. test bottle. A sample of the resin, measuring 1.9084 gm, was added to the test bottle and shaken vigorously for 30 minutes. The test bottle was placed in a 45° C. bath for at least 60 minutes. The test bottle was removed from the bath and was again shaken until complete solution was achieved. The test bottle was then placed in a 20° C. bath for 60 minutes. A viscometer tube was placed in the 20° C. bath for approximately 5 minutes to equilibrate. 10 ml of the solution was transferred from the test bottle to the viscometer tube (e.g., Cannon Fenske Viscometer No. 400) using a fast flow pipette. A pressure bulb was used to press the fluid to the upper mark of the viscometer. The pressure was released to allow the liquid level to flow past the upper mark of the viscometer. A timer was used to calculate the time for the liquid in the viscometer to pass between the upper and a lower mark. Viscosity, in centistokes, was determined by multiplying the measured time by a tube factor associated with the viscometer. Viscosity, in centipoise, was determined by multiplying the viscosity, in centistokes, by the solution density in gm/cc. Using the viscosity test, the starting PVB resin was determined to have a viscosity of 234 cps, while the viscosity of the recovered PVB resin was found to have been reduced to 213 cps.

[0076] Furthermore, the molecular weights of the starting PVB resin and the recovered PVB resin were measured using the “molecular weight test” described below. It was found that the starting PVB resin had a molecular weight of 273,031 Daltons, while the viscosity of the recovered PVB resin had reduced a molecular weight of 245,203 Daltons. In more detail, the molecular weight test comprises gel permeation chromatography (“GPC”), whereby hexafluoroisopropanol AR from Biosolve was used as the GPC mobile phase (0.8 ml/min). Each sample was prepared by weighing approximately 20 mg of the PVB resin into a 25 ml flask, and adding 10 ml of the mobile phase. The flasks were then placed in an automated shaking device until the PVB resin was fully dissolved. Analyses were performed on a three-detector system consisting of a Viscotek GPCmax (autosampler, pump, and degasser), a Viscotek triple detector TDA302 (RALL/LALLS, Viscometer, and DRI combination) with column oven. The separation was performed by three Viscotek mixed bed columns type I-MB (low, medium and high range molecular weight) maintained at 45° C. The complete detector set-up was calibrated using a narrow PMMA standard from Viscotek with a reported molecular weight of 64,368 Daltons, an intrinsic viscosity of 0.615 and a dn/dc value of 0.189. The refractive index of the mobile phase was determined to be 1,2649 g/ml through prior experimentation.

#### Example 2

[0077] A mixture of 800 parts of SD29 alcohol (190 proof ethanol), 200 parts of recycle PVB resin formed from acoustic trilayer interlayers (having skin layers with a PVOH content of 10.50 wt. % and core layers with a PVOH content of 18.70 wt. %), 3 parts water, and 0.5 parts sulfuric acid were charged within a one liter three-neck jacketed

glass reactor. The mixture was stirred and heated to 78° C., where the mixture was held for four hours. The resulting mixture was cooled to 65° C. and neutralized with 0.46 part of KOH until the mixture reached a pH of 6. The resulting cloudy mixture was filtered to yield a clear viscous solution. The solution was cast into a film and allowed to dry to result in a clear film. The film was pressed into a 0.772 mm thick film of recovered PVB resin. The recovered PVB resin was found to have a PVOH content of 23.08 wt. % using the Bomem MB160 FT-NIR Analyzer described above.

### Example 3

**[0078]** A mixture of 800 parts of SD29 alcohol, 200 parts of recycle PVB formed from acoustic trilayer interlayers (having skin layers with a PVOH content of 10.50 wt. % and core layers with a PVOH content of 18.70 wt. %), 15 parts butyraldehyde, 3 parts water, and 0.5 parts sulfuric acid were charged within a one liter three-neck jacketed glass reactor. The mixture was stirred and heated to 78° C., where the mixture was held for four hours. The resulting mixture was cooled to 65° C. and neutralized with 0.46 part of KOH until the mixture reached a pH of 6. The resulting cloudy mixture was filtered to yield a clear viscous solution. The solution was cast into a film and allowed to dry to result in a clear film. The film was pressed into a 0.772 mm thick film of recovered PVB resin. The recovered PVB resin was found to have a PVOH content of 18.77 wt. % using the Bomem MB160 FT-NIR Analyzer described above.

**[0079]** While the invention has been disclosed in conjunction with a description of certain embodiments, including those that are currently believed to be the preferred embodiments, the detailed description is intended to be illustrative and should not be understood to limit the scope of the present disclosure. As would be understood by one of ordinary skill in the art, embodiments other than those described in detail herein are encompassed by the present invention. Modifications and variations of the described embodiments may be made without departing from the spirit and scope of the invention.

**[0080]** It will further be understood that any of the ranges, values, or characteristics given for any single component of the present disclosure can be used interchangeably with any ranges, values or characteristics given for any of the other components of the disclosure, where compatible, to form an embodiment having defined values for each of the components, as given herein throughout. For example, a polymer layer can be formed comprising plasticizer content in any of the ranges given in addition to any of the ranges given for residual hydroxyl content, where appropriate, to form many permutations that are within the scope of the present invention but that would be cumbersome to list.

**[0081]** The present invention and its preferred embodiments is now further described in terms of numbered Items 1 to 114.

**[0082]** Item 1. A process for recovering poly(vinyl butyral) (PVB), said process comprising the following steps:

**[0083]** (a) mixing a PVB feed composition with a solvent, a catalyst, and, optionally, butyraldehyde to produce an equilibrated PVB-containing composition, wherein the PVB feed composition comprises at least two different PVB-containing compositions;

**[0084]** (b) determining a residual polyvinyl alcohol (PVOH) content of the equilibrated PVB-containing composition; and

**[0085]** adjusting the amount of butyraldehyde used in said mixing of step (a) based on the residual PVOH content determined in step (b)

**[0086]** Item 2. The process of Item 1, further including the step of setting a target residual PVOH content for the equilibrated PVB-containing composition

**[0087]** Item 3. The process of Item 2, wherein if the residual PVOH content determined in step (b) is greater than the target residual PVOH content, said adjusting of step (c) includes increasing the amount of butyraldehyde used in said mixing of step (a).

**[0088]** Item 4. The process of Item 2, wherein if the residual PVOH content determined in step (b) is less than the target residual PVOH content, said adjusting of step (c) includes decreasing the amount of butyraldehyde used in said mixing of step (a).

**[0089]** Item 5. The process of Item 4, wherein decreasing the amount of butyraldehyde used in said mixing of step (a) includes extracting butyraldehyde.

**[0090]** Item 6. The process of Item 2, wherein the target residual PVOH content for the equilibrated PVB-containing composition is from 14 to 50%, from 15 to 25%, from 16 to 22%, from 18.0 to 19.0%, or about 18.7%.

**[0091]** Item 7. The process of Item 1, wherein the equilibrated PVB-containing composition is a solution and said determining of step (b) is performed by cloud point measurement.

**[0092]** Item 8. The process of Item 1, wherein the equilibrated PVB-containing composition is a PVB resin and said determining of step (b) is performed using an infrared sensor.

**[0093]** Item 9. The process of Item 1, wherein the equilibrated PVB-containing composition is a PVB resin and said determining of step (b) is performed by titration.

**[0094]** Item 10. The process of Item 1, wherein the PVB feed composition comprises post-consumer recycle PVB.

**[0095]** Item 11. The process of Item 1, wherein the PVB feed composition comprises post-industrial recycle PVB.

**[0096]** Item 12. The process of Item 1, wherein the PVB feed composition comprises virgin PVB.

**[0097]** Item 13. The process of Item 1, wherein the at least two different PVB-containing compositions include a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different PVOH content than the second PVB-containing composition.

**[0098]** Item 14. The process of Item 13, wherein a relative difference in the residual PVOH content of the first PVB-containing composition and the residual PVOH content of the second PVB-containing composition is between 2 and 10 wt. %.

**[0099]** Item 15. The process of Item 1, wherein the at least two different PVB-containing compositions include a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different aldehyde content than the second PVB-containing composition.

**[0100]** Item 16. The process of Item 1, wherein the at least two different PVB-containing compositions include a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different vinyl acetate content than the second PVB-containing composition.

[0101] Item 17. The process of Item 1, wherein the at least two different PVB-containing compositions include a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different plasticizer content than the second PVB-containing composition.

[0102] Item 18. The process of Item 1, wherein the at least two different PVB-containing compositions include a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different plasticizer type than the second PVB-containing composition.

[0103] Item 19. The process of Item 1, wherein the at least two different PVB-containing compositions include a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different molecular weight than the second PVB-containing composition.

[0104] Item 20. The process of Item 1, wherein the at least two different PVB-containing compositions include a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having different additives than the second PVB-containing composition, wherein the additives are selected from one or more of the following: adhesion control agents, UV stabilizers, anti-oxidants, IR absorbers, and colorants.

[0105] Item 21. The process of Item 1, wherein when the equilibrated PVB-containing composition is mixed with 20 phr of plasticizer to form a PVB resin, and the PVB resin formed into a 0.772 mm PVB layer and positioned between two 2.3 mm sheets of glass to form a laminated glass panel, the laminated glass panel has a haze of less than 5%, less than 2%, less than 1%, less than 0.5%, or less than 0.25%.

[0106] Item 22. The process of Item 1, wherein the solvent comprises alcohol.

[0107] Item 23. The process of Item 1, wherein the catalyst comprises sulfuric acid.

[0108] Item 24. The process of Item 1, wherein said mixing of step (a) includes heating the PVB feed composition, the solvent, the catalyst, and/or the butyraldehyde.

[0109] Item 25. The process of Item 1, wherein said mixing of step (a) includes agitating the PVB feed composition, the solvent, the catalyst, and/or the butyraldehyde.

[0110] Item 26. The process of Item 1, further including the step of filtering the equilibrated PVB-containing composition to remove solids.

[0111] Item 27. The process of Item 1, further including the step of neutralizing the equilibrated PVB-containing composition by adding a base.

[0112] Item 28. The process of Item 27, wherein the base comprises potassium hydroxide.

[0113] Item 29. The process of Item 1, further including the step of precipitating the equilibrated PVB-containing composition to obtain a PVB resin.

[0114] Item 30. The process of Item 29, further including the step of drying the PVB resin.

[0115] Item 31. The process of Item 30, further including the step of washing the PVB resin to remove salt impurities.

[0116] Item 32. A process for recovering poly(vinyl butyral) (PVB), said process comprising the following steps:

[0117] (a) establishing a target residual polyvinyl alcohol (PVOH) content;

[0118] (b) combining a PVB feed composition with a solvent, a catalyst, and, optionally, butyraldehyde to

produce an equilibrated PVB-containing composition, wherein the PVB feed composition comprises at least two different PVB-containing compositions;

[0119] (c) measuring a residual PVOH content of the equilibrated PVB-containing composition; and

[0120] (d) adjusting the amount of butyraldehyde used in said combining of step (b) based on a comparison between the target residual PVOH content and the residual PVOH content measured in step (c).

[0121] Item 33. The process of Item 32, wherein if the residual PVOH content measured in step (c) is greater than the target residual PVOH content established in step (a), said adjusting of step (d) includes increasing the amount of butyraldehyde used in said combining of step (b).

[0122] Item 34. The process of Item 32, wherein if the residual PVOH content measured in step (c) is less than the target residual PVOH content established in step (a), said adjusting of step (d) includes decreasing the amount of butyraldehyde used in said combining of step (b).

[0123] Item 35. The process of Item 34, wherein decreasing the amount of butyraldehyde used in said combining of step (b) includes extracting butyraldehyde.

[0124] Item 36. The process of Item 32, wherein the target residual PVOH content is from 14 to 50 wt. %, from 15 to 25 wt. %, from 16 to 22 wt. %, from 18.0 to 19.0 wt. %, or about 18.7 wt. %.

[0125] Item 37. The process of Item 32, wherein the equilibrated PVB-containing composition is a solution and said measuring of step (c) is performed by cloud point measurement.

[0126] Item 38. The process of Item 32, wherein the equilibrated PVB-containing composition is a PVB resin and said measuring of step (c) is performed using an infrared sensor.

[0127] Item 39. The process of Item 32, wherein the equilibrated PVB-containing composition is a PVB resin and said measuring of step (c) is performed by titration.

[0128] Item 40. The process of Item 32, wherein the PVB feed composition comprises post-consumer recycle PVB.

[0129] Item 41. The process of Item 32, wherein the PVB feed composition comprises post-industrial recycle PVB.

[0130] Item 42. The process of Item 32, wherein the PVB feed composition comprises virgin PVB.

[0131] Item 43. The process of Item 32, wherein the at least two different PVB-containing compositions include a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different PVOH content than the second PVB-containing composition.

[0132] Item 44. The process of Item 43, wherein a relative difference in the residual PVOH content of the first PVB-containing composition and the residual PVOH content of the second PVB-containing composition is between 2 and 10 wt. %.

[0133] Item 45. The process of Item 32, wherein the at least two different PVB-containing compositions include a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different PVB content than the second PVB-containing composition.

[0134] Item 46. The process of Item 32, wherein the at least two different PVB-containing compositions include a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing compo-

sition having a different vinyl acetate content than the second PVB-containing composition.

[0135] Item 47. The process of Item 32, wherein the at least two different PVB-containing compositions include a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different plasticizer content than the second PVB-containing composition.

[0136] Item 48. The process of Item 32, wherein the at least two different PVB-containing compositions include a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different plasticizer type than the second PVB-containing composition.

[0137] Item 49. The process of Item 32, wherein the at least two different PVB-containing compositions include a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different molecular weight than the second PVB-containing composition.

[0138] Item 50. The process of Item 32, wherein the at least two different PVB-containing compositions include a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having different additives than the second PVB-containing composition, wherein the additives are selected from one or more of the following: adhesion control agents, UV stabilizers, anti-oxidants, IR absorbers, and colorants.

[0139] Item 51. The process of Item 32, wherein when the equilibrated PVB-containing composition is mixed with 20 phr of plasticizer to form a PVB resin, and the PVB resin formed into a 0.772 mm PVB layer and positioned between two 2.3 mm sheets of glass to form a laminated glass panel, the laminated glass panel has a haze of less than 5%, less than 2%, less than 1%, less than 0.5%, or less than 0.25%.

[0140] Item 52. The process of Item 32, wherein the solvent comprises alcohol.

[0141] Item 53. The process of Item 32, wherein the catalyst comprises sulfuric acid.

[0142] Item 54. The process of Item 32, wherein said combining of step (b) includes heating the PVB feed composition, the solvent, the catalyst, and/or the butyraldehyde.

[0143] Item 55. The process of Item 32, wherein said combining of step (b) includes agitating the PVB feed composition, the solvent, the catalyst, and/or the butyraldehyde.

[0144] Item 56. The process of Item 32, further including the step of filtering the equilibrated PVB-containing composition to remove solids.

[0145] Item 57. The process of Item 32, further including the step of neutralizing the equilibrated PVB-containing composition by adding a base.

[0146] Item 58. The process of Item 57, wherein the base comprises potassium hydroxide.

[0147] Item 59. The process of Item 32, further including the step of precipitating the equilibrated PVB-containing composition to obtain a PVB resin.

[0148] Item 60. The process of Item 59, further including the step of drying the PVB resin.

[0149] Item 61. The process of Item 60, further including the step of washing the PVB resin to remove salt impurities.

[0150] Item 62. A process for recovering poly(vinyl butyral) (PVB), said process comprising the following steps:

[0151] (a) mixing a PVB feed composition with a solvent, a catalyst, and, optionally, butyraldehyde to produce an equilibrated PVB-containing composition, wherein the PVB feed composition has a molecular weight greater than 200K Daltons;

[0152] (b) producing a PVB resin from the equilibrated PVB-containing composition, wherein the PVB resin has a molecular weight that is at least 25K Daltons less than the molecular weight of the PVB feed composition.

[0153] Item 63. The process of Item 62, wherein the PVB feed composition has a molecular weight greater than 225K, greater than 250K, greater than 275K, or greater than 300K.

[0154] Item 64. The process of Item 62, wherein the PVB resin has a molecular weight that is at least 30K, at least 40K, at least 50K, at least 60K, at least 70K, at least 80K, at least 90K, or at least 100K Daltons less than the molecular weight of the PVB feed composition.

[0155] Item 65. The process of Item 62, wherein the PVB resin has a viscosity of less than 230 cps, less than 220 cps, less than 210 cps, and/or less than 200 cps.

[0156] Item 66. The process of Item 62, wherein the PVB resin has a percent residual polyvinyl alcohol (PVOH) content from 14 to 50%, from 15 to 25%, from 16 to 22%, from 18.0 to 19.0%, or about 18.7%.

[0157] Item 67. The process of Item 62, wherein the PVB feed composition comprises post-consumer recycle PVB.

[0158] Item 68. The process of Item 62, wherein the PVB feed composition comprises recycle trim PVB.

[0159] Item 69. The process of Item 62, wherein the PVB feed composition comprises virgin PVB.

[0160] Item 70. The process of Item 62, wherein the PVB feed composition comprises at least two different PVB-containing compositions including a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different PVB content than the second PVB-containing composition.

[0161] Item 71. The process of Item 62, wherein the PVB feed composition comprises at least two different PVB-containing compositions including a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different PVOH content than the second PVB-containing composition.

[0162] Item 72. The process of Item 62, wherein the PVB feed composition comprises at least two different PVB-containing compositions including a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different vinyl acetate content than the second PVB-containing composition.

[0163] Item 73. The process of Item 62, wherein the PVB feed composition comprises at least two different PVB-containing compositions including a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different plasticizer content than the second PVB-containing composition.

[0164] Item 74. The process of Item 62, wherein the PVB feed composition comprises at least two different PVB-containing compositions including a first PVB-containing composition and a second PVB-containing composition,



with the first PVB-containing composition having a different plasticizer type than the second PVB-containing composition.

**[0165]** Item 75. The process of Item 62, wherein the PVB feed composition comprises at least two different PVB-containing compositions including a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different molecular weight than the second PVB-containing composition.

**[0166]** Item 76. The process of Item 62, wherein the PVB feed composition comprises at least two different PVB-containing compositions including a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having different additives than the second PVB-containing composition, wherein the additives are selected from one or more of the following: adhesion control agents, UV stabilizers, antioxidants, IR absorbers, and colorants.

**[0167]** Item 77. The process of Item 62, wherein when the PVB resin is formed into a 0.772 mm PVB layer and positioned between two 2.3 mm sheets of glass to form a laminated glass panel, the laminated glass panel has a haze of less than 5%, less than 2%, less than 1%, less than 0.5%, or less than 0.25%.

**[0168]** Item 78. The process of Item 62, wherein the solvent comprises alcohol.

**[0169]** Item 79. The process of Item 62, wherein the catalyst comprises sulfuric acid.

**[0170]** Item 80. The process of Item 62, wherein said mixing of step (a) includes heating the PVB feed composition, the solvent, the catalyst, and/or the butyraldehyde.

**[0171]** Item 81. The process of Item 62, wherein said mixing of step (a) includes agitating the PVB feed composition, the solvent, the catalyst, and/or the butyraldehyde.

**[0172]** Item 82. The process of 62, further including the step of filtering the equilibrated PVB-containing composition to remove solids.

**[0173]** Item 83. The process of Item 62, further including the step of neutralizing the equilibrated PVB-containing composition by adding a base, and wherein the base comprises potassium hydroxide.

**[0174]** Item 84. The process of Item 62, wherein said producing of step (b) includes precipitating the equilibrated PVB-containing composition to obtain the PVB resin.

**[0175]** Item 85. The process of Item 62, further including the step of drying the PVB resin.

**[0176]** Item 86. The process of Item 62, further including the step of washing the PVB resin to remove salt impurities.

**[0177]** Item 87. A process for recovering poly(vinyl butyral) (PVB), said process comprising the following steps:

**[0178]** (a) combining and heating a PVB feed composition, a solvent, a catalyst, and, optionally, butyraldehyde to produce an equilibrated PVB-containing composition, wherein the PVB feed composition has a molecular weight greater than 200K Daltons;

**[0179]** (b) precipitating the equilibrated PVB-containing composition to produce a PVB resin, wherein the PVB resin has a molecular weight that is at least 25K less than the molecular weight of the PVB feed composition.

**[0180]** Item 88. The process of Item 87, wherein the PVB feed composition has a molecular weight greater than 225K, greater than 250K, greater than 275K, or greater than 300K Daltons.

**[0181]** Item 89. The process of Item 87, wherein the PVB resin has a molecular weight that is at least 30K, at least 40K, at least 50K, at least 60K, at least 70K, at least 80K, at least 90K, or at least 100K Daltons less than the molecular weight of the PVB feed composition.

**[0182]** Item 90. The process of Item 87, wherein the PVB resin has a viscosity less than 230 cps, less than 220 cps, less than 210 cps, and/or less than 200 cps.

**[0183]** Item 91. The process of Item 87, wherein the PVB resin has a percent residual polyvinyl alcohol (PVOH) content from 14 to 50%, from 15 to 25%, from 16 to 22%, from 18.0 to 19.0%, or about 18.7%.

**[0184]** Item 92. The process of Item 87, wherein the PVB feed composition comprises post-consumer recycle PVB.

**[0185]** Item 93. The process of Item 87, wherein the PVB feed composition comprises recycle trim PVB.

**[0186]** Item 94. The process of Item 87, wherein the PVB feed composition comprises virgin PVB.

**[0187]** Item 95. The process of Item 87, wherein the PVB feed composition comprises at least two different PVB-containing compositions including a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different PVB content than the second PVB-containing composition.

**[0188]** Item 96. The process of Item 87, wherein the PVB feed composition comprises at least two different PVB-containing compositions including a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different PVOH content than the second PVB-containing composition.

**[0189]** Item 97. The process of Item 87, wherein the PVB feed composition comprises at least two different PVB-containing compositions including a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different vinyl acetate content than the second PVB-containing composition.

**[0190]** Item 98. The process of Item 87, wherein the PVB feed composition comprises at least two different PVB-containing compositions including a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different plasticizer content than the second PVB-containing composition.

**[0191]** Item 99. The process of Item 87, wherein the PVB feed composition comprises at least two different PVB-containing compositions including a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different plasticizer type than the second PVB-containing composition.

**[0192]** Item 100. The process of Item 87, wherein the PVB feed composition comprises at least two different PVB-containing compositions including a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different molecular weight than the second PVB-containing composition.

**[0193]** Item 101. The process of Item 87, wherein the PVB feed composition comprises at least two different PVB-containing compositions including a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having different additives than the second PVB-containing composition, wherein the additives are selected from one or more of the following: adhesion control agents, UV stabilizers, anti-oxidants, IR absorbers, and colorants.

**[0194]** Item 102. The process of Item 87, wherein when PVB resin is formed into a 0.772 mm PVB layer and positioned between two 2.3 mm sheets of glass to form a laminated glass panel, the laminated glass panel has a haze of less than 5%, less than 2%, less than 1%, less than 0.5%, or less than 0.25%.

**[0195]** Item 103. The process of Item 87, wherein the solvent comprises alcohol.

**[0196]** Item 104. The process of Item 87, wherein the catalyst comprises sulfuric acid.

**[0197]** Item 105. The process of Item 87, wherein said combining and heating of step (a) includes agitating the PVB feed composition, the solvent, the catalyst, and/or the butyraldehyde.

**[0198]** Item 106. The process of Item 87, further including the step of filtering the equilibrated PVB-containing composition to remove solids.

**[0199]** Item 107. The process of Item 87, further including the step of neutralizing the equilibrated PVB-containing composition by adding a base, and wherein the base comprises potassium hydroxide.

**[0200]** Item 108. The process of Item 87, further including the step of drying the PVB resin.

**[0201]** Item 109. The process of Item 87, further including the step of washing the PVB resin to remove salt impurities.

**[0202]** Item 110. The recycled PVB formed by the method of any of Items 1 to 109.

**[0203]** Item 111. A resin layer comprising the recycled PVB of Item 110.

**[0204]** Item 112. An interlayer comprising the resin layer of Item 111.

**[0205]** Item 113. The interlayer of Item 112, further comprising a second resin layer, or wherein the resin layer is a core layer, and the interlayer further comprises a second resin layer and a third resin layer, wherein the core layer is between the second resin layer and the third resin layer.

**[0206]** Item 114. A laminate comprising a first substrate, the interlayer of any of Items 112 to 113, and a second substrate, wherein the interlayer is between the first substrate and the second substrate.

1. A process for recovering poly(vinyl butyral) (PVB), said process comprising the following steps:

- (a) mixing a PVB feed composition with a solvent, a catalyst, and, optionally, butyraldehyde to produce an equilibrated PVB-containing composition, wherein the PVB feed composition comprises at least two different PVB-containing compositions;
- (b) determining a residual polyvinyl alcohol (PVOH) content of the equilibrated PVB-containing composition; and
- (a) adjusting the amount of butyraldehyde used in said mixing of step (a) based on the residual PVOH content determined in step (b).

2. The process of claim 1, further including the step of setting a target residual PVOH content for the equilibrated PVB-containing composition.

3. The process of claim 2, wherein if the residual PVOH content determined in step (b) is greater than the target residual PVOH content, said adjusting of step (c) includes increasing the amount of butyraldehyde used in said mixing of step (a), or wherein if the residual PVOH content determined in step (b) is less than the target residual PVOH content, said adjusting of step (c) includes decreasing the amount of butyraldehyde used in said mixing of step (a), and wherein decreasing the amount of butyraldehyde used in said mixing of step (a) includes extracting butyraldehyde.

4. The process of claim 1, wherein the equilibrated PVB-containing composition is a solution and said determining of step (b) is performed by cloud point measurement, or the equilibrated PVB-containing composition is a PVB resin and said determining of step (b) is performed using an infrared sensor or by titration.

5. The process of claim 1, wherein said mixing of step (a) includes heating the PVB feed composition, the solvent, the catalyst, and/or the butyraldehyde.

6. A process for recovering poly(vinyl butyral) (PVB), said process comprising the following steps:

- (a) establishing a target residual polyvinyl alcohol (PVOH) content;
- (b) combining a PVB feed composition with a solvent, a catalyst, and, optionally, butyraldehyde to produce an equilibrated PVB-containing composition, wherein the PVB feed composition comprises at least two different PVB-containing compositions;
- (c) measuring a residual PVOH content of the equilibrated PVB-containing composition; and
- (d) adjusting the amount of butyraldehyde used in said combining of step (b) based on a comparison between the target residual PVOH content and the residual PVOH content measured in step (c).

7. The process of claim 6, wherein if the residual PVOH content measured in step (c) is greater than the target residual PVOH content established in step (a), said adjusting of step (d) includes increasing the amount of butyraldehyde used in said combining of step (b), or wherein if the residual PVOH content measured in step (c) is less than the target residual PVOH content established in step (a), said adjusting of step (d) includes decreasing the amount of butyraldehyde used in said combining of step (b), and wherein decreasing the amount of butyraldehyde used in said combining of step (b) includes extracting butyraldehyde.

8. The process of claim 6, wherein the equilibrated PVB-containing composition is a solution and said measuring of step (c) is performed by cloud point measurement, or wherein the equilibrated PVB-containing composition is a PVB resin and said measuring of step (c) is performed using an infrared sensor, or wherein the equilibrated PVB-containing composition is a PVB resin and said measuring of step (c) is performed by titration.

9. The process of claim 6, wherein said combining of step (b) includes heating the PVB feed composition, the solvent, the catalyst, and/or the butyraldehyde.

10. The process of claim 6, wherein the target residual PVOH content is from 14 to 50 wt. %.

11. The process of claim 6, wherein the at least two different PVB-containing compositions include a first PVB-containing composition and a second PVB-containing com-

position, with the first PVB-containing composition having a different PVOH content than the second PVB-containing composition, and wherein a relative difference in the residual PVOH content of the first PVB-containing composition and the residual PVOH content of the second PVB-containing composition is between 2 and 10 wt. %.

**12.** The process of claim **6**, wherein when the equilibrated PVB-containing composition is mixed with 20 phr of plasticizer to form a PVB resin, and the PVB resin formed into a 0.772 mm PVB layer and positioned between two 2.3 mm sheets of glass to form a laminated glass panel, the laminated glass panel has a haze of less than 5%, less than 2%, less than 1%, less than 0.5%, or less than 0.25%.

**13.** A process for recovering poly(vinyl butyral) (PVB), said process comprising the following steps:

- (a) mixing a PVB feed composition with a solvent, a catalyst, and, optionally, butyraldehyde to produce an equilibrated PVB-containing composition, wherein the PVB feed composition has a molecular weight greater than 200K Daltons; and
- (b) producing a PVB resin from the equilibrated PVB-containing composition, wherein the PVB resin has a molecular weight that is at least 25K Daltons less than the molecular weight of the PVB feed composition.

**14.** The process of claim **13**, wherein said mixing of step (a) includes heating the PVB feed composition, the solvent, the catalyst, and/or the butyraldehyde.

**15.** A process for recovering poly(vinyl butyral) (PVB), said process comprising the following steps:

- (a) combining and heating a PVB feed composition, a solvent, a catalyst, and, optionally, butyraldehyde to produce an equilibrated PVB-containing composition, wherein the PVB feed composition has a molecular weight greater than 200K Daltons; and
- (b) precipitating the equilibrated PVB-containing composition to produce a PVB resin, wherein the PVB resin

has a molecular weight that is at least 25K less than the molecular weight of the PVB feed composition.

**16.** The process of claim **15**, wherein said combining and heating of step (a) includes agitating the PVB feed composition, the solvent, the catalyst, and/or the butyraldehyde.

**17.** The process of claim **15**, wherein the PVB resin has a molecular weight that is at least 50K Daltons less than the molecular weight of the PVB feed composition.

**18.** The process of claim **15**, wherein the PVB feed composition comprises at least two different PVB-containing compositions including a first PVB-containing composition and a second PVB-containing composition, with the first PVB-containing composition having a different vinyl acetate content than the second PVB-containing composition.

**19.** The process of claim **1**, wherein the PVB feed composition comprises one or more of post-consumer recycle PVB, post-industrial recycle PVB, or recycle trim PVB.

**20.** The process of claim **1**, further including a step of neutralizing the equilibrated PVB-containing composition by adding a base, wherein the base comprises potassium hydroxide.

**21.** The recycled PVB formed by the method of claim **1**.

**22.** A resin layer comprising the recycled PVB of claim **21**.

**23.** An interlayer comprising the resin layer of claim **22**.

**24.** The interlayer of claim **23**, wherein the resin layer is a core layer, and the interlayer further comprises a second resin layer and a third resin layer, wherein the core layer is between the second resin layer and the third resin layer.

**25.** A laminate comprising a first substrate, the interlayer of claim **24**, and a second substrate, wherein the interlayer is between the first substrate and the second substrate.

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