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PLIABLE, ABSORBENT, BONDED PAPER LAMINATES

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The present invention relates to multi-ply paper laminates formed from tissue weight paper plies and to a process for their manufacture. More particularly, the present invention is concerned with tissue weight papers and their transformation into multi-ply paper laminates having two to four plies which, while substantially retaining the absorbency, porosity and pliability of the original tissue weight paper plies are endowed with wet and dry tensile strength and toughness. Furthermore, the process of this invention, while concerned with the manufacture of laminated sheet materials from plies of tissue weight paper, results in multi-ply tissue weight paper laminates exhibiting to a substantial degree the strength and toughness characteristics of woven textiles.

Specifically this invention provides for a process wherein the fibers of tissue weight paper plies and the plies themselves are bonded together with about 8% to about 14%, preferably about 11% to about 13%, based on the weight of the dry multi-ply paper laminate, of regenerated cellulose, applied to the adjoining surfaces of the tissue weight paper plies as a viscose foam.

Sheeted structures have been produced in the prior art by forming fleeces of carded textile fibers as described in U.S. 2,705,687 and U.S. 2,705,688, issued on Apr. 5, 1955, to De Witt R. Petterson et al. and I. S. Ness et al., respectively. Further examples of bonded fibrous sheets are disclosed in U.S. 2,039,312, issued on May 5, 1936, to Joshua H. Goldman, and in U.S. 1,745,557 and U.S. 1,856,114, issued respectively on Feb. 4, 1930, and May 3, 1932, to George A. Richter et al.

The prior art processes have obtained a fabric-like quality in bonded fibrous sheets by the employment of one or more manufacturing steps or raw material selections to enhance pliability. For example, U.S. 2,039,312 referred to above and U.S. 3,009,822, issued on Nov. 21, 1961, to Arthur H. Drelich, disclose "spot bondings" in printed patterns which, in conjunction with non-bonded areas in a sheet predominately composed of fibers longer than papermaking fibers, enhance pliability. Again, the products described in U.S. 2,705,687 and U.S. 2,705,688, above, achieve hand and drape by selecting a carded raw material having directionally oriented textile length fibers as opposed to the random oriented fibers in a wet-laid sheet composed of papermaking length fibers.

The disclosures of U.S. 1,745,557 and U.S. 1,856,114, above, illustrate methods of using viscose in the overall bonding of a wet-laid paper towel with a small amount, about 2%, of regenerated cellulose. The amount of viscose employed in these disclosures is insufficient to block the fiber interstices when applied as a solution, but develops only a limited amount of fiber bonding.

Applicants have discovered that the necessity of limiting the use of regenerated cellulose binder to spot bonding, of choosing carded substrates composed of textile length fibers for bonding and of employing a minimal amount of regenerated cellulose binder to avoid stiffness and a harsh surface feel can be overcome in multi-ply tissue paper laminates. The foregoing detrimental features are overcome by applying a selected amount of cellulose binder as a viscose foam to the adjoining surfaces of two to four tissue weight paper plies prior to laminating them into multi-ply paper laminates and regenerating the celluose. This procedure develops superior wet and dry

strength in the multi-ply paper laminates while retaining, or enhancing, the pliability, hand, absorbency, porosity and other fabric-like qualities of the individual tissue weight paper plies. U.S. 2,338,960, granted to C. L. Nottebohm on Jan. 11, 1944, discloses the use of foams in connection with the binding of "fleeces," oriented, carded, pretreated fabrics such as those used in U.S. 2,705,687 and 2,705,688. In contrast applicants have discovered that viscose foams, prepared in a certain manner and applied in the aforementioned selected amounts, develop superior wet and dry strength characteristics in multi-ply tissue weight paper laminates having two to four plies while substantially retaining the desirable porosity, pliability, absorbency and other fabric-like qualities of tissue weight paper plies composed of papermaking length fibers.

It is, therefore, an object of this invention to provide a process for producing a pliable, porous and absorbent multi-ply paper laminate from two to four tissue weight paper plies.

A further object of this invention is to provide a process for producing a multi-ply paper laminate having two to four plies and exhibiting enhanced wet and dry strength while retaining porosity, absorbency and pliability characteristics substantially like those of the individual tissue weight paper plies.

Broadly stated, the present process is carried out by selecting two to four plies of tissue weight paper to serve as substrate material in a multi-ply paper laminate. One or more of the adjoining, or interior, surfaces of the subsequent multi-ply paper laminate are then coated with viscose foam, and the tissue weight paper plies are brought together and adhesively impregnated with the viscose foam in a pressure zone. The pressure zone can be conveniently provided by the nip of two pressure rolls which can also emboss a pattern in the multi-ply paper laminate as it is formed. The use of pressure rolls is not essential, however, and any convenient method of bringing the tissue weight paper plies into contact in a pressure zone can be employed.

More precisely, the process of the present invention is carried out by selecting two to four tissue weight paper plies, each having a basis weight of about 6 pounds to about 20 pounds per 3000 square feet to serve as substrates for the viscose foam in a multi-ply tissue weight paper laminate. Although the process is illustrated hereinbelow with tissue weight paper plies of equal basis weight, the selection of equal basis weight plies is not a necessary condition. The use of more than four tissue weight paper plies, or plies of heavier weight than those stated, results in an undesirably stiff and less porous multi-ply paper laminate, while individual tissue sheets coated with viscose foam lack the requisite tensile strength.

Having selected the tissue weight paper plies, a viscose is prepared which contains about 8% to about 10% by weight of cellulose. It is noted that the foregoing percentages and all further percentages and parts stated in the remainder of the disclosure and claims are by weight, except where otherwise specifically stated.

Applicants prefer to use viscose containing the greatest amount of cellulose possible without adversely affecting the handle and porosity of the multi-ply paper laminates. While practice of the invention has shown that the viscosity, cellulose content, sodium hydroxide and carbon bisulfide content of the viscose used herein can be varied over the normal range found in commercial viscoses, studies have revealed preferable viscose compositions. Preferable viscose compositions contain about 9% to about 10% cellulose, about 2.0% to about 2.5% sulfur originally added as carbon bisulfide and about 4% to about 6% sodium hydroxide with the remaining portion of the compositions being comprised of water. The prefer-

able viscoses are further characterized by exhibiting a viscosity of about 100 to about 150 poises.

Viscose for use in the present process does not require filtration processing to remove the gels and the particulate matter normally present in unfiltered commercial viscose. In fact, the use of unfiltered viscose may provide added adhesive qualities desirable in the present multi-ply paper laminates.

Although viscose foam of any composition providing the stated amounts of regenerated cellulose binder at the foamed volumes stated hereinbelow can be used to obtain the benefits of the process, applicants have found it desirable to use a viscose foam containing about 40% to about 80% viscose together with water and the necessary amount of foaming surfactant to attain the degree of foaming stated below. One preferred viscose foam contains about 60% viscose together with about 2% foaming surfactant and water. The preference is based upon the properties of these mixtures to result in viscose foams exhibiting desirable manipulation charactertistics and resulting in desirable combinations of multi-ply paper laminate qualities.

The ingredients of the viscose foam can be combined in any order. A preferred procedure for preparing the viscose foam is to pre-foam the surfactant and water mixture by using agitation. In this manner, air is introduced into the surfactant and water mixture until a thick fine-bubbled lather is obtained. The viscose is then added to and dispersed throughout the pre-foamed surfactant and water mixture while continuing the agitation to form the viscose foam. The viscose foam, when ready for use in the instant process has a volume of about 2 to about 6 times, preferably about 4 times, the unfoamed volume of its combined ingredients, including the viscose, water and surfactant.

Control of the viscose foam volume provides a means for controlling the amount of viscose, and therefore regenerated cellulose, added to the tissue weight paper plies in the multi-ply paper laminates. Assuming a fixed cellulose composition by weight in the viscose foam together with a given volume of foam depoisted on an area of tissue weight paper ply, more viscose to become regenerated cellulose adhesive per multi-ply paper laminate area will result with a decreased degree of foaming. An increase in the degree of foaming volume will result in less regenerated cellulose per given area of multi-ply paper laminate.

Applicants have discovered that anionic surfactants, for example sodium alkyl benezene sulfonates derived from polypropylene, and sodium alkyl sulfates derived from coconut alcohols are more satisfactory as foaming surfactants for the present viscose foam system than are the cationic or nonionic types of surfactants. Applicants prefer to use sodium alkyl benzene sulfonate foaming surfactants wherein the alkyl groups contain a range of about 9 carbon atoms to about 15 carbon atoms, although any surfactant which will result in the stated range of viscose foam volume can be employed.

Having selected the tissue weight paper plies and prepared the viscose foam, the viscose foam is applied in a uniform continuous coating to one or more adjoining surfaces of the tissue weight paper plies in amounts to result in the percentages of regenerated cellulose binder stated hereinabove. The uniform continuous coating of viscose foam can be applied by a doctor blade or any conventional equipment adapted to deposit and uniformly spread regulated quantities of viscous materials.

The tissue weight paper plies are then brought together to form a multi-pay paper laminate by any conventional means of bringing their surfaces into contact, for example, by passing them through the nip of two rubber-covered pressure rolls. The tissue weight paper plies in their laminated, or combined, state are then passed through a bath to regenerate the cellulose in the viscose.

The composition of the cellulose regeneration bath can be like any of those baths conventionally in use for the regeneration of the cellulose in viscose to produce viscose rayon fibers or cellophane. Applicants have used an aqueous regenerating bath containing about 5% sulfuric acid together with sodium sulfate at ambient temperature; the amount of sodium sulfate in the regenerating bath at equilibrium is determined by the sodium hydroxide content of the viscose. The use of regeneration baths containing sodium sulfate and, in some instances, zinc salts in addition to sulfuric acid will also give satisfactory results.

After regeneration of the cellulose in the viscose, the multi-ply paper laminate can be subjected to any of the conventional purification and treatment steps given to cellulose regenerated from viscose. These conventional purification and treatment steps include washing with water to an acid free state, desulfuring, bleaching with hypochlorite, softening and drying and are all quite well known in the viscose industry. Softening, for example, can be accomplished by passing the laminated tissue sheet through an aqueous solution containing a humectant such as glycerine or diethylene glycol. Drying, for example, can be carried out by any of the conventional methods used in the drying of paper; including, for example, steam can drying, festoon drying and tunnel drying.

The use of various finishing operations in connection with the production of viscose films and fibers is not, per se, a part of the present invention, which, as stated hereinbefore, is concerned with a novel process for manufacturing multi-ply paper laminates from tissue weight paper plies. While it is true that the various conventional finishing steps, for example embossing, creping, calendering, perforating, or possible combinations thereof, may enhance the fabric-like qualities of the present multi-ply paper laminate, the finishing procedure can be varied widely, dependent upon the specific characteristics desired in the finished product.

The wet and dry tensile strengths of the multi-ply paper laminates of this invention were determined for comparison with multi-ply laminates containing greater or lesser amounts of regenerated cellulose. The breaking loads (lbs. per inch width) reported in the examples below were determined on an Instron tester (Model No. TM) using a one inch wide sample with a zero-span between the tester jaws. The breaking load is the force required to rupture the test strip. The total work to break (inch lbs./sq. inch) was determined on the Instron tester by pulling apart a one inch wide sample with a span of 4 inches between the tester jaws. The total work to break is the area under the stress-strain curve plotted by the Instron tester as the sample was stretched and finally ruptured. The dry test results were determined from air-dry samples, while the wet tests were determined from saturated samples.

The following examples illustrate specific embodiments of the process for manufacturing multi-ply paper laminates from tissue weight paper plies, as described herein, and illustrations of the effect of operation outside the disclosed limits on the multi-ply paper laminate product are also provided. The examples are intended to illustrate the invention, but are not intended to limit it to any greater extent than do the appended claims.

Example I

Two tissue weight paper plies, each weighing 7 lbs. per 3000 sq. ft., were selected for use in a multi-ply paper laminate.

A viscose containing 10% cellulose, 6% sodium hydroxide, 2.5% sulfur originally added as carbon bisulfide and having a viscosity of 100 poises together with a salt index of 6 was then prepared.

Twenty parts of water were then placed in a container and 25 parts of a solution containing 10% of sodium alkyl benzene sulfonate wherein the alkyl groups were

predominately those containing 12 carbon atoms was added thereto. A high speed agitator was used to foam the water containing the alkyl benzene sulfonate mixture to 4 times its liquid volume.

Seventy-five parts of the viscose was thereafter added to the foamed water solution with continued agitation to attain a uniform blend of the viscose in a foamed state and to maintain the system at 4 times its liquid volume. The viscose foam contained about 63% viscose, about 2% alkyl benzene sulfonate and about 35% water.

The two tissue weight paper plies were then coated on the faces to be brought into contact with the above viscose foam in amounts to result in the deposition of 12.4% regenerated cellulose in the 2-ply paper laminate on a dry basis prior to addition of the glycerine softener. About 2 cubic feet of foam were applied per 3000 square feet of laminate.

The viscose foam coated, tissue weight paper plies were then brought together by rubber-covered pressure rolls and pressed to form the 2-ply paper laminate. The cellulose in the 2-ply paper laminate was next regenerated by passing the 2-ply paper laminate into an aqueous solution containing 5% sulfuric acid together with equilibrium sodium sulfate at 72° F.

Following the regeneration of the cellulose from the viscose foam between the plies of the laminate, the laminate was washed to an acid free condition in water and softened by passing it through an aqueous solution containing 15% glycerine.

The regenerated, washed and softened laminate was then oven dried. The product of the process of Example I had a dry and wet breaking load, respectively of 18.2 pounds and 6.5 pounds per inch of laminate width. The total work to break the product of Example I in a dry and wet condition was respectively 0.640 inch-pound per square inch and 0.374 inch-pound per square inch, and the product was suitable for use as a hand towel.

Additional multi-ply paper laminates containing greater and lesser quantities of regenerated cellulose binder were prepared in the manner of Example I. The toughness (total work to break wet and dry expressed in inch lbs./sq. inch) and strength (breaking load expressed in lbs./inch width) of all the multi-ply paper laminates, including that of Example I are reported in Table I below together with equivalent values for a blank made of two plies of the same tissue weight paper with no cellulose binder regenerated from viscose foam. Illustrations I and II showing the effect of operating outside of the herein disclosed range of regenerated cellulose binder addition are also provided. For close comparison, although the 2-ply paper laminates weighed approximately 1 oz./sq. yard, all of the strength results are given on the basis of a ratio calculated 2-ply paper laminate weigh of exactly 1 oz./sq. yard.

process. Strength properties are maximized at about 11% to about 13% regenerated cellulose binder. The porosity, absorbency and pliability of Examples I–VI was maintained. At binder contents below 8%, as in Illustration I, strength properties decrease (wet delamination of the plies occurs). When the binder content is increased above about 14%, as in Illustration II with 18% binder content, the product loses wet strength and becomes increasingly stiff like the regenerated cellulose binder instead of the tissue weight paper plies.

All of the products of the present invention as shown in the examples above containing about 8% to about 14% of cellulose binder regenerated from viscose foam were soft and porous, and they exhibited toughness and high wet and dry strength together with a desirable pliability.

A multi-ply paper laminate prepared according to the process of Example I but having four plies and prepared with a viscose foam at 2 times liquid volume was porous, absorbent and pliable. A multi-ply paper laminate prepared according to the process of Example I but having two plies each weighing 20 pounds per 3000 sq. ft. and wherein the viscose foam, containing 8% cellulose in the viscose, is foamed to 6 times its liquid volume will also be porous, absorbent and pliable.

The 2-ply paper laminate products of Examples II, III, IV, V and VI were useful as disposable handkerchiefs, bed sheets, table cloths, hand towels and cleaning cloths.

Having thus described the invention, what is claimed is:

1. A process for the manufacture of a multi-ply paper laminate having two to four tissue weight paper plies and exhibiting the qualities of pliability, absorbency, porosity, wet and dry tensile strength and toughness which comprises the steps of (1) selecting the appropriate number of tissue weight paper plies each having a basis weight of about 6 pounds per 3000 square feet to about 20 pounds per 3000 square feet, (2) preparing a viscose foam containing about 40% to about 80% of viscose from viscose which contains about 8% to about 10% cellulose, foaming surfactant and water and foaming said viscose foam to about 2 times to about 6 times its liquid volume, (3) applying the viscose foam of step 2 as a uniform continuous coating between adjoining surfaces of said tissue weight paper plies in an amount to result in the presence of about 8% to about 14% of regenerated cellulose in the dried multi-ply paper laminate, (4) laminating the tissue weight paper plies by bringing their adjoining surfaces into contact in a pressure zone, (5) passing the multi-ply paper laminate through a cellulose regenerating bath, and (6) washing and drying the resultant multi-ply paper laminate.

2. The process for the manufacture of a multi-ply paper laminate described in claim 1 wherein the tissue weight paper plies are laminated in step 4 by passing them through a pressure zone formed by the nip of two rubber-covered pressure rolls and the multi-ply paper laminate is passed through an aqueous bath containing diethylene glycol as a softener, immediately prior to drying in step 6.

TABLE I

Example	Laminate Weight, oz./sq. yd.	Regenerated Cellulose, percent dry product basis	Breaking Load, lbs./inch width		Total Work to Break, inch-lbs./sq. inch		Laminate properties
			Dry	Wet	Dry	Wet	
Illustration I	0.93	7.0	15.8	6.0	.383	----------	Delaminated in wet total-work-to-break test.
II	0.95	8.3	15.5	5.9	.487	.264	Absorbent, porous and pliable.
III	1.00	10.2	15.7	6.2	.531	.341	Do.
IV	1.00	11.1	17.1	6.2	.553	.324	Do.
I	1.02	12.4	18.2	6.5	.640	.374	Do.
V	1.04	13.0	17.9	7.4	.585	.356	Do.
VI	1.05	14.5	16.6	6.2	.546	.348	Do.
Illustration II	1.16	18.0	14.2	5.0	.610	.270	Reduced pliability, porosity and strength.
Blank	0.84	0.0	9.7	2.8	.324	.105	Low-strength.

The data in Table I above show that amounts of regenerated cellulose binder in the range of about 8% to about 14% are effective in the practice of the present

3. A process for the manufacture of a multi-ply paper laminate having two to four tissue weight paper plies and exhibiting the qualities of pliability, absorbency, porosity, wet and dry tensile strength and toughness which comprises the steps of (1) selecting the appropriate number of tissue weight paper plies each having a basis weight of about 6 pounds per 3000 square feet to about 20 pounds per 3000 square feet, (2) preparing a viscose foam from a mixture containing about 40% to about 80% of viscose, foaming surfactant and water, wherein said viscose has a viscosity of about 100 to about 150 poises and contains about 9% to about 10% cellulose, about 2.0% to about 2.5% sulfur originally added as carbon bisulfied and about 4% to about 6% sodium hydroxide and foaming said viscose foam to about 2 times to about 6 times its liquid volume, (3) applying the viscose foam of step 2 as a uniform continuous coating between adjoining surfaces of said tissue weight paper plies in and amount to result in the presence of about 11% to about 13% of regenerated cellulose in the dried multi-ply paper laminate, (4) laminating the tissue weight paper plies by bringing their adjoining surfaces into contact in a pressure zone, (5) passing the multi-ply paper laminate through a cellulose regenerating bath, and (6) washing and drying the resultant multi-ply paper laminate.

4. A process for the manufacture of a multi-ply paper laminate having two to four tissue weight paper plies and exhibiting the qualities of pliability, absorbency, porosity, wet and dry tensile strength and toughness which comprises the steps of (1) selecting the appropiate number of tissue weight paper plies each having a basis weight of about 6 pounds per 3000 square feet to about 20 pounds per 3000 square feet, (2) preparing a viscose foam containing about 60% viscose, about 2% foaming surfactant and water, wherein said viscose has a viscosity of about 100 poises to about 150 poises and contains about 8% to about 10% cellulose, about 2.5% sulfur originally added as carbon bisulfide and about 6% sodium hydroxide, and foaming said viscose foam to about 2 times to about 6 times its liquid volume, (3) applying the viscose foam of step 2 as a uniform continuous coating between the adjoining surfaces of said tissue weight paper plies in an amount to result in the presence of about 11% to about 13% of regenerated cellulose in the dried multi-ply paper laminate, (4) laminating the tissue weight paper plies by bringing their adjoining surfaces into contact in a pressure zone, (5) passing the multi-ply paper laminate through a cellulose regenerating bath, and (6) washing and drying the resultant multi-ply paper laminate.

5. A process for the manufacture of a multi-ply paper laminate having two tissue weight paper plies and exhibiting the qualities of pliability, absorbency, porosity, wet and dry tensile strength and toughness which comprises the steps of (1) selecting two tissue weight paper plies each having a basis weight of about 7 pounds per 3000 square feet, (2) preparing a viscose foam containing about 63% viscose, about 2% foaming surfactant and about 35% water, wherein said viscose has a viscosity of about 100 poises and contains about 10% cellulose, about 2.5% sulfur originally added as carbon bisulfide and about 6% sodium hydroxide and wherein said foaming surfactant is a sodium alkyl benzene sulfonate surfactant whose alkyl groups contain about 9 to about 15 carbon atoms, by first foaming the foaming surfactant and water to about 4 times their liquid volume by agitation and thereafter adding the viscose while maintaining the foamed volume by continued agitation, (3) applyng the viscose foam of step 2 as a uniform continuous coating between the adjoining surfaces of said tissue weight paper plies in an amount to result in the presence of about 12% of regenerated cellulose in the dried multi-ply paper laminate, (4) laminating the tissue weight paper plies by bringing their adjoining surfaces into contact in a pressure zone formed by the nip of two rubber-covered rolls, (5) passing the multi-ply paper laminate through a cellulose regenerating bath containing about 5% sulfuric acid, and (6) washing the multi-ply paper laminate to an acid free condition in water, passing it through an aqueous solution containing about 15% by weight of glycerine and drying the resultant multi-ply paper laminate.

6. A multi-ply paper laminate having two to four tissue weight paper plies each having a basis weight of about 6 pounds to about 20 pounds per 3000 square feet and exhibiting the qualities of pliability, absorbency, porosity, wet and dry tensile strength and toughness, the plies of which are bonded together by about 8% to about 14% in the multi-ply paper laminate of cellulose regenerated from a viscose foam prepared from viscose containing about 8% to about 10% cellulose, foaming surfactant and water and said viscose foam is foamed to about 2 times to about 6 times its liquid volume.

7. A multi-ply paper laminate having two to four tissues weight paper plies each having a basis weight of about 6 pounds per 3000 square feet to about 20 pounds per 3000 square feet and exhibiting the qualities of pliability, absorbency, porosity, wet and dry tensile strength and toughness, the plies of which are bonded together by about 11% to about 13% in the multi-ply paper laminate of cellulose regenerated from a viscose foam prepared from viscose, foaming surfactant and water, wherein said viscose has a viscosity of about 100 to about 150 poises and contains about 9% to about 10% cellulose, about 2.0% to about 2.5% sulfur originally added as carbon bisulfide and about 4% to about 6% sodium hydroxide and said viscose foam is foamed to about 2 times to about 6 times its liquid volume.

8. A multi-ply paper laminate having two to four tissue weight paper plies each having a basis weight of about 6 pounds per 3000 square feet to about 20 pounds per 3000 square feet and exhibiting the qualities of pliability, absorbency, porosity, wet and dry tensile strength and toughness, the plies of which are bonded together by about 11% to about 13% in the multi-ply paper laminate of cellulose regenerated from a viscose foam containing about 63% viscose, about 2% foaming surfactant and about 35% water, wherein said viscose has a viscosity of about 100 poises to about 150 poises and contains about 8% to about 10% cellulose, about 2.5% sulfur added as carbon bisulfide and about 6% sodium hydroxide and said viscose foam is foamed to about 2 to about 6 times its liquid volume.

9. A multi-ply paper laminate having two tissue weight paper plies each having a basis weight of about 7 pounds per 3000 square feet and exhibiting the qualities of pliability, absorbency, porosity, wet and dry tensile strength and toughness, the two plies of which are bonded together by about 12% in the multi-ply paper laminate of cellulose regenerated from a viscose foam containing about 63% viscose, about 2% foaming surfactant and about 35% water, wherein said viscose has a viscosity of about 100 poises and contains about 10% cellulose, about 2.5% sulfur originally added as carbon bisulfide and about 6% sodium hydroxide and wherein said foaming surfactant is a sodium alkyl benzene sulfonate surfactant whose alkyl groups contain about 9 to about 15 carbon atoms and the viscose foam is prepared by first foaming the foaming surfactant and water to about 4 times their liquid volume by agitation and thereafter adding the viscose while maintaining the foamed volume by continued agitation.

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