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SYNTHETIC FIBER PAPER AND PROCESS FOR PRODUCING THE SAME

Hanns F. Arledter, Stockbridge, Mass., assignor, by mesne assignments, to Hurlbut Paper Company, a corporation of Ohio

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The present invention relates to paper and to a process of producing the same, and more particularly to a paper having a fibrous content of synthetic fibers only and to a process of producing the same.

Heretofore synthetic resin fibers and metallic fibers have been used in paper only in conjunction with other fibers, such as papermaker fibers or cellulosic fibers. Although synthetic resin fibers or metallic fibers have been woven so as to form cloth-like materials, or have been air deposited to form so-called non-woven cloth, it has not been possible heretofore to prepare a sufficiently strong sheet-like paper material containing a very high percentage, approximately 100%, of synthetic resin fibers or metallic fibers which are arranged heterogeneously as in the case of ordinary paper containing organic fibrous materials and made by a wet process on a paper machine.

The production of a synthetic fiber paper product having a fibrous content consisting essentially of synthetic resin fibers or metallic fibers has been fraught with many difficulties. Synthetic fibers in general are characterized by their smoothness, inability to hydrate when beaten in a beater, and their lack of a tendency to cling to one another when an aqueous mixture thereof is spread upon a continuously moving screen and dried as is done in the papermaking industry. It has also been exceedingly difficult to disperse synthetic resin fibers and metallic fibers in water. Furthermore, conventional papermaking equipment has not proven to be suitable for the production of fibrous synthetic resin paper or metallic fiber paper wherein the synthetic resin fibers have a very small or very large diameter.

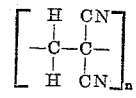
Accordingly, it is an object of the present invention to provide a strong paper-like material having a fibrous content consisting of approximately 100% synthetic fibers of the nature of synthetic resin fibers, metallic fibers, or synthetic inorganic fibers, such as quartz. Moreover, it is a further object of the invention to provide a process of producing such papers.

The synthetic fiber paper of the invention is useful as a filter for acid and alkali solutions, since it is acid and alkali resistant. The paper may also be employed as a battery separator for acid and alkali batteries. Furthermore, in the case of paper containing Teflon, the paper is useful in the electrical field and for high temperature applications wherein the temperature may range from about 200° C. to about 300° C. The synthetic fiber paper containing metallic fibers is useful for capacitors, provided thin fibers of the nature of tantalum or aluminum are employed and for battery plates, if, for example, nickel, iron, lead or zinc are employed. The paper can be employed also in laminates for heating panels, heat dissipating surfaces, radiation shielding, catalytic effects, and as resistors.

The process of the invention for preparing a synthetic fiber paper comprises forming a paper web of from about 70% to about 90% of slightly acid soluble synthetic

fibers and from about 30% to about 10% respectively of acid soluble fibers. The slightly acid soluble synthetic fibers are synthetic resin fibers and metallic fibers which upon exposure to sulfuric acid or hydrofluoric acid of a concentration of about 90% for 5 minutes show a weight loss of less than 50%. This amount of dissolution of synthetic fibers will still leave fibers having sufficient strength and thickness to be suitable in the process of the invention. The acid soluble fibers are cellulosic fibers and glass fibers which dissolve completely in sulfuric acid and hydrofluoric acid respectively under such conditions. A discontinuous non-penetrating coating of from about 5% to about 10% solids by weight of a resin binder insoluble in sulfuric acid and also in hydrofluoric acid may be applied onto the fibers of the paper web, if desired. The paper web, either with or without the resin binder, is treated with sulfuric acid when it contains cellulosic fibers or with hydrofluoric acid when it contains glass fibers to remove these acid soluble fibers therefrom.

Any synthetic resin fibers may be employed in the process as long as they are only slightly acid soluble or insoluble in the glass or cellulose solvent employed. Typical examples of such fibers include polytetrafluoroethylene (Teflon), polyvinylchloride, polyacrylonitrile (Orlon), copolymer of 60% vinylchloride and 40% acrylonitrile (Dynel), copolymer of vinylchloride and vinylacetate (Vinyon), and dinitrile fibers having the following base molecule:



where n represents an integer other than zero. The molecular weight of the synthetic resin fibers may vary in any practical range producible, e.g., 30,000 to about 3,000,000. The diameter of the synthetic resin fibers may be about 6 to 30 microns, although fibers of other diameters may be employed if desired. It was found that mixtures of precut synthetic resin fibers having a length in the range from about 3 mm. to about 10 mm. permitted the manufacture of paper with from two to tenfold increase in strength in regard to fold, tear, and wet strength and with a higher overall strength in regard to burst strength and tensile strength as compared with paper wherein the synthetic resin fibers were cut to random papermaker lengths with a conventional beater.

Any suitable metallic fibers, e.g., silver, aluminum, gold, lead, tantalum, nickel, steel, and copper may be employed in the paper and method according to this invention, provided they are insoluble or only slightly soluble in sulfuric acid as well as in hydrofluoric acid.

Stainless steel or steel fibers, for example, do not dissolve to an appreciable extent in concentrated sulfuric acid owing to the formation of a black deposit on the surface of the metal which protects the metal from further action. Thus steel fibers exposed to sulfuric acid of various concentrations for 5 minutes, this time being the maximum exposure period necessary in the process of the invention, showed the following weight losses:

Concentration of Sulfuric Acid	Weight Loss of Steel Fibers
Percent	*Percent*
90	40
80	35
70	31

The action of sulfuric acid on very pure metals is very small due to the absence of galvanic couples. Zinc, which

has been carefully purified, has almost no reaction with sulfuric acid. The comparative passivity of metals in strong sulfuric acid or hydrofluoric acid originates in part also from the presence of gaseous or solid films on the surfaces of the metallic fibers which prevent further free access of the acid. Cellulosic fibers, on the other hand, dissolve rather quickly, within 30–120 seconds, in sulfuric acid and during this short time period the attack of sulfuric acid on steel fibers can be sufficiently small to make this process feasible.

The acid soluble fibers may be cellulosic fibers, for example, rag stock, alpha cellulose, and cellulose of low alpha content for better solubility, or they may be glass fibers. These acid soluble fibers serve as dispersing aids for the synthetic fibers and also enable the synthetic fibers of the paper web to cling to one another while the web is being formed upon a continuously moving screen and dried on the dryers and further processed until the final bonding is achieved.

Any suitable resin binder insoluble in sulfuric acid as well as in hydrofluoric acid may be used in the process. The following resins are typical of those which can be used: polyvinylchloride, copolymers of vinylchloride and vinylacetate, copolymers of vinylidene chloride and vinylchloride, phenolic resins, synthetic rubber compounds, and a polymer of trifluorochloroethylene. The resin binder may be employed to bind the synthetic fibers together in the final paper product.

The synthetic fibers in the paper web can be bonded in any of the following ways also.

Calendering of the formed paper structure at 1,000–18,000 p.s.i., at room or elevated temperatures densifies the paper structure and deforms the synthetic fibers so that an entanglement between the synthetic fibers takes place. For certain synthetic resin fibers for example, polyvinylchloride fibers fusion of the fibers at the crossing point is obtained. Entanglement or fusion of the synthetic fibers gives equally sufficient physical adhesion for a self-supporting fiber structure after the cellulosic or glass carrier fibers are removed by acid treatment.

Synthetic thermoplastic resin fibers of low fusion point, for example, Vinyon fibers, may be added in the conventional manner to the paper structure. The paper structure is then calendered with heat and pressure whereupon the thermoplastic fibers fuse with the non-thermoplastic synthetic fibers in the structure, giving the final paper good strength after removal of the cellulosic or glass fibers by acid treatment.

In the case of metallic fiber paper structures, the bonding can take place either with the aid of pressure or with sintering techniques at elevated temperatures and under vacuum, neutral nitrogen or reducing hydrogen atmospheres.

Any suitable solvent may be used which will dissolve the acid soluble fibers in the paper web, thereby leaving a paper web containing synthetic resin fibers or metallic fibers only, with or without a resin binder. Sulfuric acid may be used, for example, in dissolving cellulosic fibers, while hydrofluoric acid may be used in dissolving glass fibers which may be added as the dispersing aid. It will be appreciated that other solvents may be used in lieu of sulfuric acid or hydrofluoric acid. Cellulosic fibers may be dissolved, for example, with Cellosolve (cupriethylenediamine).

Further details of the process are as follows. Cellulosic fibers or glass fibers which serve as a dispersing aid and as an aid in preventing slipping of the synthetic fibers in the paper web during processing are beaten in a conventional beater. This fiber stock is diluted with water and synthetic fibers, preferably precut, added thereto and well distributed therein by the brushing action of the beater roll, or other mechanical devices, such as refiners. The fiber slurry is then further diluted with water and the resulting paper furnish formed into a paper web on a conventional papermaking machine.

During the manufacture of the paper a resin binder may be added in the beater or headbox with any of the methods known in the art of papermaking. The resin binder may also be added by spraying on the paper machine or in the size press. As noted above the resin binder is applied in such a manner that a discontinuous non-penetrating coating of all of the fibers of the paper web is achieved. It is essential that the coating be discontinuous and non-penetrating in order that the subsequent acid treatment will be capable of dissolving the acid soluble fibers leaving behind only the slightly acid soluble synthetic fibers and the acid insoluble resin binder. If the fibers in the wet paper web were coated with a uniform nonpermeable film, then the acid subsequently applied thereto would be unable to attack the acid soluble fibers and dissolve them. In addition, the resin binder must not penetrate the acid soluble fibers so as to render them insoluble. Accordingly, in order to achieve a discontinuous non-penetrating coating of the fibers of the paper web by the resin binder, the paper web is preferably sprayed with an emulsion or precipitate resin system so as to apply from about 5% to about 10% solids by weight of the resin binder. True water or solvent solutions of the resin binder are only feasible if the same do not penetrate into the fibers or have a tendency to form a bond at the junction of the fibers thereby leaving the fibers free to attack by the subsequent acid treatment.

After the resin binder, if any, has been applied to the fibers of the paper web, the finished paper is calendered with or without heat to the wanted specific gravity and final paper strength. The paper web is then treated with sulfuric acid or hydrofluoric acid to remove the acid soluble fibers therefrom. While the paper web may be treated with the aicid by many conventional methods, such as spraying, it is preferred to pass the paper web through the acid bath and remove the surplus acid with a press roll. After the acid soluble fibers have been dissolved from the paper web, the paper is then washed in succession with water, ammonia, and water. The neutral paper is then dried and heat calendered to the finished product.

The paper produced by the above-described process consists essentially of an interfelted web of randomly distributed synthetic fibers bonded together with a resin binder or with a physical fiber-to-fiber bond. The synthetic resin fibers or metallic fibers in the paper product are accordingly arranged as in a standard paper product, i.e., in a heterogeneous or random distribution. The synthetic fibers have no major orientation or are oriented in the way obtainable with a conventional sheet-forming machine. The fibrous content of the paper consists entirely of synthetic fibers. The resin binder content, if any, of the paper product may vary from about 2% to about 30% by weight. The strength of the paper product will vary with the nature of the resin binder, the amount of resin binder, the heat and calendering pressure applied, the density of the paper, and length of the fibers. It is preferred that the resin binder content lies within the range of about 5% to about 7% by weight. Accordingly, the synthetic fiber paper containing a resin binder comprises from about 98% to about 70% and preferably from about 95% to about 93%, of synthetic fibers and from about 2% to about 30%, and preferably from about 5% to about 7%, respectively of a resin binder, the amounts being by weight.

The process and product of the invention will be further described in connection with the following examples. Examples 1–4 show the modifications of the process and produce wherein no resin binder is utilized.

Example 1 shows the use of synthetic resin fibers and cellulosic fibers.

Example 1

25% cellulose fibers were beaten in a beater together with 60% fibers of a copolymer of vinylchloride and acrylonitrile (Dynel) and 15% fibers of a copolymer of vinylchloride and vinylacetate (Vinyon) until a papermaking fiber length was obtained. A paper web was formed and processed in the conventional manner on a paper machine at a dryer temperature of 220° F. The dried and finished paper was calendered at 175° F. at 12,000 p.s.i. to bond the paper internally by the Vinyon fibers which have a softening point of 160° F. and a melting point of approximately 230° F. The paper web was led through a sulfuric acid bath of 66% concentration to dissolve the cellulose fibers, pressed to remove excess acid, and rewound while wet. After 20 minutes the paper was washed successively with water, ammonia, and water; rewound; and then calendered again. The paper consisted essentially of Dynel and Vinyon fibers only.

The use of synthetic resin fibers and glass fibers is illustrated in Example 2.

Example 2

One pound (44%) of Vinyon HH fibers (copolymer of vinylchloride and vinylacetate) in 20 gallons of water was beaten to papermaker length. One fourth pound (12%) of glass fibers of 0.75 to 1 micron diameter was added to the Vinyon fiber slurry, well dispersed and evenly distributed. One pound (44%) of precut Dynel fibers (copolymer of vinylchloride and acrylonitrile) of ⅛ to ¼ inch length was mixed into the fiber stock. The stock was diluted with 40 gallons of water, bringing the volume to 60 gallons, and the paper web was formed on the wire of a paper machine. The paper machine speed and dryed temperature of 170–220° F. was adjusted in such a way that the Vinyon fibers did not shrink excessively. The paper was heat calendered at a temperature of 175° F. with a pressure of 3,000 p.s.i. close to the end of the dryer section of the paper machine. The calendering fused the Vinyon fibers with each other and with the Dynel fibers. The glass fibers were removed from the finished paper web by passing the paper web through a hydrofluoric acid bath. The paper was washed successively with water, ammonia, and water; rewound; and calendered again. The paper consisted essentially of Dynel and Vinyon fibers only.

In Example 3 metallic fibers and cellulosic fibers were used.

Example 3

Twenty-five parts of rag fibers were beaten to a beating degree of 45° S.R. at 6% stock consistency. The rag stock was diluted with 2,000 parts water, and seventy-five parts aluminum fibers were added and processed in the beater to a papermaking length of 1 to 6 mm. A paper web was formed on the wire of a paper machine in a conventional way. The dry paper was calendered to a specific gravity of 1.5 g./cc. The cellulosic fibers were removed from the paper by passing it through a sulfuric acid bath at room temperature. The paper was washed successively with water, ammonia, and water; dried and calendered. The paper consisted essentially of aluminum fibers only.

Example 4 shows the use of metallic fibers and glass fibers.

Example 4

Fifteen parts of glass fibers of 0.75 micron average diameter and 3,000 parts of water were processed in a beater and eighty-five parts of precut copper fibers of 3–6 mm. length were dispersed in the fiber slurry. The paper sheets were formed with a sheet mold and the paper was calendered in two steps in the wet state and after drying, with a pressure of 6,000 p.s.i. The glass fibers were removed by passing the paper web through a hydrofluoric acid bath. The paper was then washed successively with ammonia, water, and ammonia; dried; and calendered. The paper product consisted essentially of copper fibers.

Examples 5–8 show the use of a resin binder, Example 5 using synthetic resin fibers, cellulosic fibers and a resin binder.

Example 5

Twenty-five pounds of alpha cellulose were beaten to a beating degree of 46° S.R. at a stock consistency of 5%. The stock was then diluted with water to a stock consistency of 1.0% and seventy-five pounds of polyvinylchloride fibers, precut with a fiber cutter to a length of 2, 4, and 6 mm. lengths were well distributed in the stock under the brushing action of the beater roll, but without any beating or fiber shortening of the polyvinylchloride fibers. The fiber slurry was then diluted with water to a stock consistency of approximately 0.5–1%. This furnish was then formed into a paper web of eighty pounds—24 x 36—500—on the wire of a conventional papermaking machine. A 5% polyvinylchloride emulsion having an emulsion particle size of 0.5–2 microns was then sprayed onto the wet paper web on the wire of the papermaking machine, thereby forming a discontinuous, non-penetrating coating of the polyvinylchloride resin binder upon the fibers of the paper web. The paper web was then calendered to a specific gravity of 0.6–0.8. The resin coated paper web was passed through a sulfuric acid bath having a concentration of 90–98% and a temperature of 40–50° C. to dissolve the alpha cellulose fibers in the paper. The paper web was passed through the acid bath for a period of about 2 minutes and pressed between rolls to remove excess acid. The paper was then washed with water, neutralized with ammonia, and again washed with water. The paper was then dried and calendered and wound upon a receiving drum. The paper product produced thereby consisted essentially of 95% polyvinylchloride fibers bound together with 5% of a polyvinylchloride resin binder.

Example 6 illustrates the process using synthetic resin fibers, glass fibers, and a resin binder.

Example 6

20% of glass fibers having a diameter of about 1 micron were beaten in a beater at a stock consistency of 5%. The stock was then diluted with water to a stock consistency of 1% and eighty pounds (80%) of polytetrafluoroethylene fibers precut with a fiber cutter to a length in the range from 2 mm. to 6 mm. were well distributed in the fibrous glass stock under the brushing action of the beater roll, but without any beating or fiber shortening of the polytetrafluoroethylene fibers. The fiber slurry was diluted with water to a stock consistency of about 0.5–1%. This furnish was formed into a paper web of 180 pounds—24 x 36—500—on the wire of a conventional papermaking machine. A 5% emulsion of a copolymer of vinylchloride and vinylacetate was added to the stock in the headbox of the paper machine and there flocculated with alum. The finished paper was calendered to a specific gravity of about 0.7. The paper web was passed through a hydrofluoric acid bath having a temperature of about 30° C. to dissolve the glass fibers in the paper web, the paper web being exposed to the hydrofluoric acid bath for about 20 minutes. The paper was then washed in succession with hydrofluoric acid, water, ammonia, and water. The neutral paper was then dried and heat calendered and wound upon a receiving drum. This paper product consisted essentially of 95% polytetrafluoroethylene fibers bound together with 5% of a copolymer of vinylchloride and vinylacetate.

In Example 7 metallic fibers, cellulosic fibers, and a resin binder were utilized.

Example 7

Thirty parts of rag fibers were beaten to a beating

degree of 50° S.R. in 2,000 parts of water. Seventy-five parts of aluminum fibers were added and processed in the beater to obtain a fiber slurry with fibers of papermaker length. Care was taken so that the aluminum fibers were kept as long as possible. Fourteen parts of phenol-formaldehyde resin emulsion containing 40% solids with particle size of 1–4 microns were added and the fiber-resin slurry was flocculated with alum. The sheet forming was done with a papermaker wire with a stock consistency of approximately 0.1%. The paper was calendered and dried at a temperature of 165° C. until the phenol-formaldehyde resin was well cured. The cellulosic material in the paper was then removed by passing the paper through a sulfuric acid bath. The paper consisted essentially of 94% of aluminum fibers and 6% phenol-formaldehyde resin binder.

Example 8 illustrates the process using metallic fibers, glass fibers, and a resin binder.

Example 8

Eighty-five parts of steel fibers cut to ¼ inch length were mixed and brushed in the beater with fifteen parts of glass fibers of 0.75 micron diameter in approximately 5,000 parts of water until a smooth uniform fiber slurry was obtained. Four parts of polyvinylchloride emulsion was added and flocculated with acid. The stock was then diluted with 15,000 parts of water and the paper formed on the wire of a paper machine. A paper of 400 pounds (24 x 36—500) basis weight and 40 mil thickness was produced with this fiber slurry. The paper was placed on a plastic screen and hydrofluoric acid filtered through the paper until all the glass fibers were removed. The paper was washed successively with water, ammonia, and water; dried; and calendered to a specific gravity of 1.5. The paper consisted essentially of 95% steel fibers and 5% polyvinylchloride resin binder.

Various modifications and changes may be made in the process and product of this invention without departing from the spirit thereof, and accordingly the invention is to be limited only within the scope of the appended claims.

This application is a continuation-in-part of copending application Serial No. 397,467, filed December 10, 1953, now abandoned.

I claim:

1. The process of preparing a synthetic fiber paper comprising forming a paper web of from about 70% to about 90% of slightly acid soluble synthetic fibers selected from the group consisting of synthetic resin fibers and metallic fibers and from about 30% to about 10% respectively of acid soluble fibers selected from the group consisting of cellulosic fibers and glass fibers and treating the paper web with an acid in which said acid soluble fibers are soluble to remove the acid soluble fibers therefrom.

2. The process of preparing a synthetic fiber paper comprising forming a paper web of from about 70% to about 90% of synthetic resin fibers slightly soluble in sulfuric acid and from about 30% to about 10% respectively of cellulose fibers soluble in sulfuric acid and treating the paper web with sulfuric acid to remove the cellulosic fibers therefrom.

3. The process of preparing a synthetic fiber paper comprising forming a paper web of from about 70% to about 90% of synthetic resin fibers slightly soluble in hydrofluoric acid and from about 30% to about 10% respectively of glass fibers soluble in hydrofluoric acid and treating the paper web with hydrofluoric acid to remove the glass fibers therefrom.

4. The process of preparing a synthetic fiber paper comprising forming a paper web of from about 70% to about 90% of metallic fibers slightly soluble in sulfuric acid and from about 30% to about 10% respectively of cellulosic fibers soluble in sulfuric acid and treating the paper web with sulfuric acid to remove the cellulosic fibers therefrom.

5. The process of preparing a synthetic fiber paper comprising forming a paper web of from about 70% to about 90% of metallic fibers slightly soluble in hydrofluoric acid and from about 30% to about 10% respectively of glass fibers soluble in hydrofluoric acid and treating the paper web with hydrofluoric acid to remove the glass fibers therefrom.

6. The process of preparing a synthetic fiber paper comprising forming a paper web of from about 70% to about 90% of synthetic resin fibers slightly soluble in sulfuric acid and from about 30% to about 10% respectively of cellulosic fibers soluble in sulfuric acid, applying a discontinuous non-penetrating coating of from about 5% to about 10% solids of a resin binder insoluble in sulfuric acid onto the fibers of the paper web, and treating the paper web with sulfuric acid to remove the cellulosic fibers therefrom.

7. The process of preparing a synthetic fiber paper comprising forming a paper web of from about 70% to about 90% of synthetic resin fibers slightly soluble in hydrofluoric acid and from about 30% to about 10% repectively of glass fibers soluble in hydrofluoric acid, applying a discontinuous non-penetrating coating of from about 5% to about 10% solids of a resin binder insoluble in hydrofluoric acid onto the fibers of the paper web, and treating the paper web with hydrofluoric acid to remove the glass fibers therefrom.

8. The process of preparing a synthetic fiber paper comprising forming a paper web of from about 70% to about 90% of metallic fibers slightly soluble in sulfuric acid and from about 30% to about 10% respectively of cellulosic fibers soluble in sulfuric acid, applying a discontinuous non-penetrating coating of from about 5% to about 10% solids of a resin binder insoluble in sulfuric acid onto the fibers of the paper web, and treating the paper web with sulfuric acid to remove the cellulosic fibers therefrom.

9. The process of preparing a synthetic fiber paper comprising forming a paper web of from about 70% to about 90% of metallic fibers slightly soluble in hydrofluoric acid and from about 30% to about 10% respectively of glass fibers soluble in hydrofluoric acid, applying a discontinuous non-penetrating coating of from about 5% to about 10% solids of a resin binder insoluble in hydrofluoric acid onto the fibers of the paper web, and treating the paper web with hydrofluoric acid to remove the glass fibers therefrom.

10. A water-laid synthetic fiber paper consisting of an interfelted web of randomly distributed synthetic fibers slightly soluble in sulfuric acid and hydrofluoric acid selected from the group consisting of synthetic resin fibers and metallic fibers; said web having voids therein caused by the dissolution from the web of fibers soluble in one of said acids.

11. A synthetic fiber paper consisting of an interfelted web of randomly distributed synthetic resin fibers slightly soluble in sulfuric acid and hydrofluoric acid; said web having voids therein caused by the dissolution from the web of fibers soluble in one of said acids.

12. A water-laid synthetic fiber paper consisting essentially of an interfelted web of randomly distributed metallic fibers slightly soluble in sulfuric acid and hydrofluoric acid; said web having voids therein caused by the dissolution from the web of fibers soluble in one of said acids.

13. A water-laid synthetic fiber paper consisting essentially of from about 70% to about 98% of an interfelted web of randomly distributed metallic fibers slightly soluble in sulfuric acid and hydrofluoric acid bonded together with from about 30% to about 2% respectively of a resin binder; said web having voids therein caused

by the dissolution from the web of fibers soluble in one of said acids.

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