Kirk-Othmer Encyclopedia of Chemical Technology.

FIBERS, POLY(VINYL ALCOHOL)

The principal fiber types that fall under the category of vinyl fibers are fibers that contain at least 85% by weight of vinyl chloride, known generically as vinyon fibers, and those that are composed of at least 50% by weight of vinyl alcohol and are referred to as vinal fibers. The latter are by far larger volume commercial products. Other fibers in this category are based on vinylidene chloride or tetrafluoroethylene (see Vinyl polymers, vinyl chloride polymers; Vinylidene chloride monomer and polymers; Fluorine compounds, organic–polytetrafluoroethylene).

Vinal fibers, or poly(vinyl alcohol) fibers, are not made in the United States, but the fiber is produced commercially in Japan, Korea, and China where the generic name vinylon is used. These materials are the subject of this article (see also Vinyl polymers, vinyl alcohol polymers).

1. History of Poly(vinyl alcohol) Fiber

Vinyl alcohol does not exist as a monomer, but Herrmann and Haehnel (1) were able to obtain the desired product poly(vinyl alcohol) [9002-89-5] (PVA), by polymerizing vinyl acetate and then hydrolyzing the resultant poly(vinyl acetate). This process is employed for the commercial production of PVA even now. The principal concern of the discoverers was development of a suture for surgical operations; the fiber then obtained was not suited for clothing use (2).

In Germany (3), the UK (4), the United States (5), and France (6) study began to improve the water resistance of the fiber so that it could be used for general-purpose items including clothing. In Japan, Sakurada and his co-researchers succeeded in obtaining a water-insoluble PVA fiber in 1939, by wet spinning an aqueous PVA solution into a coagulating bath of concentrated aqueous sodium sulfate, followed by formalization of the fiber thus spun (7). At about the same time, Kanegafuchi Spinning Co. published a process for producing a PVA fiber by wet spinning an aqueous PVA solution into a specific coagulating bath (8).

In 1942, Kyoto University, with government support, constructed a pilot plant to promote research and development of PVA fiber; during World War II they continued developing techniques for the industrial production of PVA fiber. After the war, Kurashiki Rayon Co., Ltd. (now Kuraray Co., Ltd.) constructed a pilot plant and started commercial production in 1950. Many chemical companies and fiber manufacturers in Japan, such as Dainippon Spinning Co., Ltd. (now Unitika Co., Ltd.) and The Nippon Synthetic Chemical Industry Co., Ltd. subsequently began production of PVA materials including fibers. PVA fiber was given the generic name of vinylon in Japan in 1948, and vinal in the United States.

Commercial production of PVA fiber was thus started in Japan, at as early a period as that for nylon. However, compared with various other synthetic fibers which appeared after that period, the properties of which have continuously been improved, PVA fiber is not very well suited for clothing and interior uses because of its characteristic properties. The fiber, however, is widely used in the world because of unique features such as high affinity for water due to the $-OH$ groups present in PVA, excellent mechanical properties because of high crystallinity, and high resistance to chemicals including alkali and natural conditions.

The People's Republic of China introduced Kuraray technology and started production of PVA fiber by a wet spinning process in 1965. Its annual capacity reached 165,000 tons in 1986 (9). The Democratic People's Republic of Korea produce PVA and reportedly have an annual production capacity of 50,000 tons (9).

2. Manufacture of Fiber

2.1. Raw Material

PVA is synthesized from acetylene [74-86-2] or ethylene [74-85-1] by reaction with acetic acid (and oxygen in the case of ethylene), in the presence of a catalyst such as zinc acetate, to form vinyl acetate [108-05-4] which is then polymerized in methanol. The polymer obtained is subjected to methanolysis with sodium hydroxide, whereby PVA precipitates from the methanol solution.

PVA used for the manufacture of fiber generally has a degree of polymerization of about 1700 and, for general-purpose fiber, a high degree of hydrolysis of vinyl acetate units of at least 99 mol %.

Pure PVA dissolves in water but does not fluidize by melting. Commercial production of PVA fiber is therefore carried out by wet spinning or dry spinning, utilizing aqueous PVA solution. In either case, purified PVA is dissolved in hot water and the solution is extruded through fine holes of a spinneret; the extruded streams are coagulated to form continuous filaments, which are then heat-treated to have adequate mechanical properties.

Since PVA fiber as spun is soluble in water, it is necessary to improve the water resistance of the as-spun fiber (10). Heat treatment followed by acetalization is a classic method to provide high water resistance.

Tows and staples, as well as short cut chips, are being manufactured by wet spinning and filament yarns by both dry and wet spinning.

2.2. Wet Spinning Process

Figure 1 is a flow diagram for producing PVA fiber by means of the wet spinning process utilizing a coagulating bath of an aqueous sodium sulfate solution. After PVA has been washed to remove impurities, it is dissolved in hot water to yield an aqueous solution with a prescribed concentration and viscosity. A dissolution-improving agent such as calcium chloride may be added (11) or, instead of water, organic solvents capable of dissolving PVA can be used (12, 13). The solution is, after filtration and deaeration, sent to a spinning machine. In the spinning machine, the aqueous PVA solution is extruded through spinnerets into a coagulating bath of an aqueous sodium sulfate solution with high concentration and coagulated therein; the continuous filaments formed are withdrawn from the bath.

The spinning machine is, generally, of the vertical type because of its stable operation and compactness (14). The filaments as spun are slightly drawn with guide rolls, then drawn in the coagulating bath in a ratio of 2:3 and dried. The filaments are then heat-drawn and heat-treated at high temperatures to obtain necessary mechanical properties and dimensional stability. The sodium sulfate deposited on the filaments from the coagulating bath does not hinder heat processing. The heat-treated filaments are provided with hot water resistance by acetalization with formaldehyde or the like.

For staple, the heat-treated filaments in the form of tow are cut to prescribed lengths and then acetalized to develop crimp on individual cut fibers. A suitable finish is selected from conventional ones and applied to the acetalized fibers to improve their spinnability and other properties required for the intended use.

Filament yarn is produced basically by the same process except that the spinning machine is so constructed as to prevent individual yarns leaving spinnerets from contacting each other.

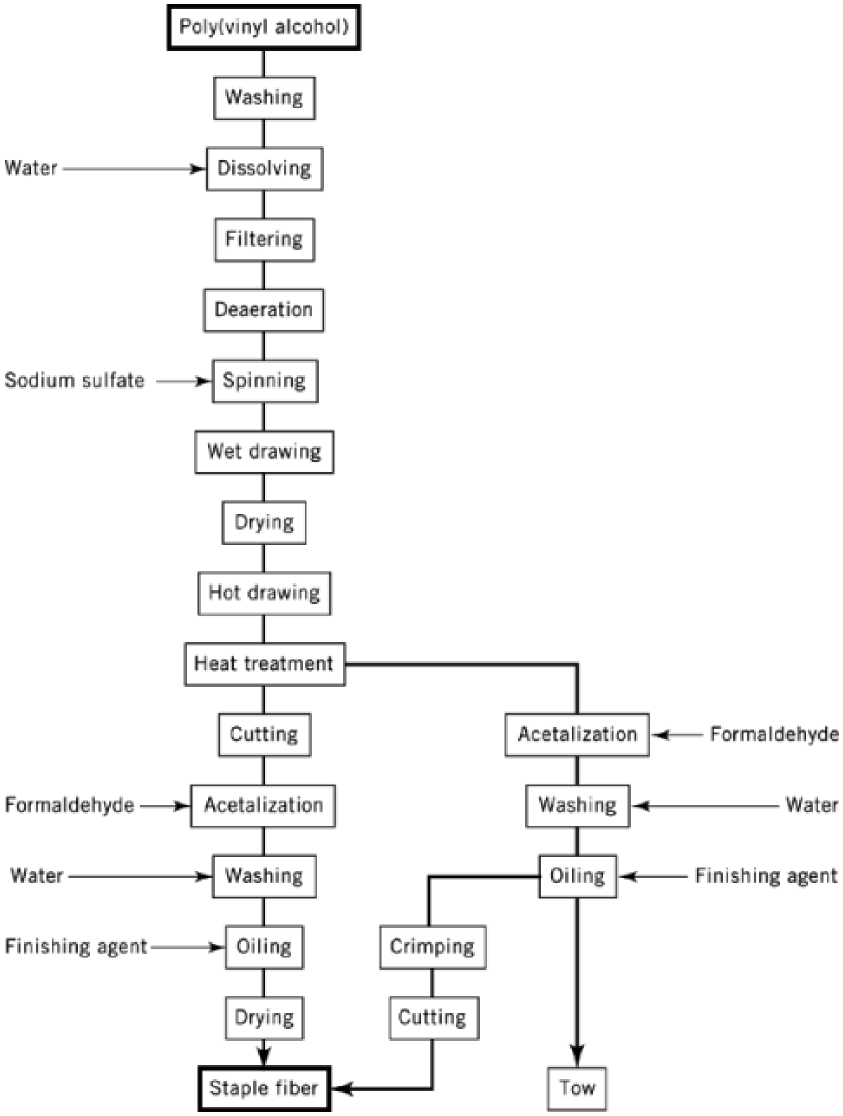

Fig. 1. Flow diagram for the manufacture of vinal by wet spinning.

2.2.1. Mechanism of Fiber Formation

After extrusion of a spinning dope through the orifices of a spinneret into a coagulating bath, the extruded streams undergo dehydration and coagulation by action of the salt contained in the bath. Network structure forms in each of the streams as a result of secondary bonding between the molecules of PVA. The fiber structure that forms is fixed depending on the stage during the coagulation and to what extent the network structure is formed. For instance, with salt in the bath, dehydration from the outer surface of each of the extruded streams proceeds rapidly just after extrusion and the outer surface of each of the extruded streams proceeds rapidly just after extrusion and the outer surface becomes a dense skin layer. The inner portion of the fiber solidifies only insufficiently because of the skin suppressing outward diffusion of interior water. As a result, the finished

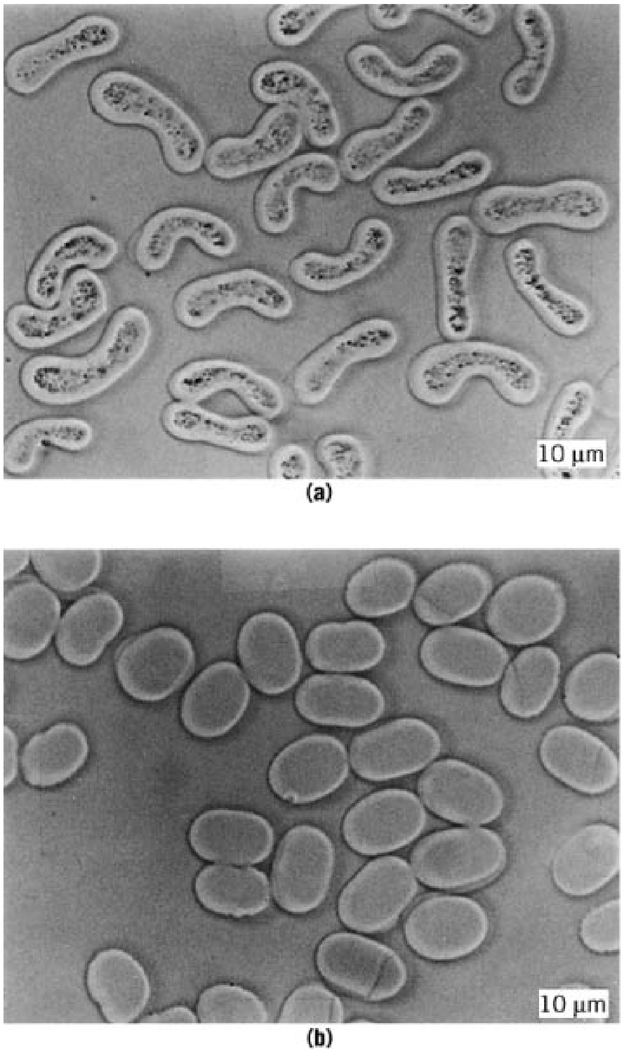

Fig. 2. Photograph of the cross sections of PVA fiber manufactured by wet spinning with a coagulating bath of sodium sulfate (**a**) and sodium hydroxide (**b**).

fiber has a two-layer (skin/core) structure as shown in the cross section in Figure 2**a.** On the other hand, in what is known as alkali spinning using a bath of sodium hydroxide solution or the like, gelation is considered to precede dehydration and the fiber cross section becomes circular and uniform (Fig. 2**b**).

The cross-sectional shape and uniformity of as-spun fiber influence the drawability in the succeeding heat-drawing process. As-spun fiber from dehydration–coagulation spinning is difficult to draw to a high draw ratio so that the finished fiber is limited in tensile strength and like properties. However, finished fiber from alkali spinning has high strength and low elongation as a result of the circular, uniform cross section.

An aqueous PVA solution containing a small amount of boric acid may be extruded into an aqueous alkaline salt solution to form a gel-like fiber (15, 16). In this process, sodium hydroxide penetrates rapidly into the aqueous PVA solution extruded through orifices to make it alkaline, whereby boric acid cross-links PVA

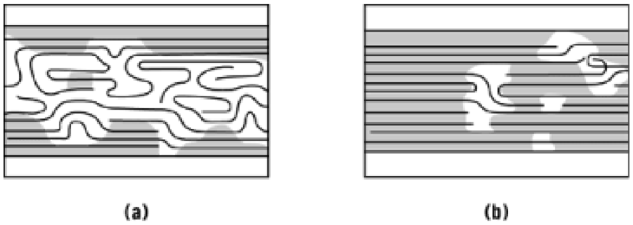

Fig. 3. Schematic comparison of the structures of PVA fibers formed by salt coagulation and alkali coagulation. (**a**) Low orientation, low crystallinity; (**b**) high orientation, high crystallinity.

molecules with each other. The resulting fiber is provided with sufficient strength to withstand transportation to the next process step and its cross section does not show a distinct skin/core structure.

After the washing, the fiber is dried, and then is heat-drawn in the same manner as in the case of dehydration–coagulation with salt but to a much higher draw ratio. As a result the finished fiber has high strength and modulus and is, without acetalization, sufficiently resistant to boiling water. Figure 3 shows schematic fiber structures (17).

Boric acid/alkali spinning has been commercialized in Kuraray Co. and Unitika Co. in Japan, and is reportedly under research and development also in the People's Republic of China as a process for producing high strength PVA fiber to be used for replacing asbestos (9).

Fine adjusting and optimization of each step of this process is still underway, and a PVA fiber having a single fiber strength as high as 2 N/tex (21 gf/dtex), which is close to that of aramid fiber, has been reported (18).

2.3. Dry Spinning

This process comprises extruding a high concentration aqueous PVA solution into conditioned air and drying the extruded streams to obtain fibers. Details of this process have been given (19, 20). In dry spinning, the extruded streams coagulate by cooling, rather than by evaporation of water. Figure 4 shows a flow diagram of the manufacture of filament yarn by dry spinning.

This process starts with preparation of raw material PVA chips. Chip preparation is conducted for the purposes of securing prescribed water content and improving operation efficiency during extrusion dissolution. Ordinary PVA, containing sodium acetate, is washed with cool water to reduce the sodium acetate content. After washing and squeezing off of excess water, the PVA is fed to a kneader, where it is kneaded to give a prescribed water content and then formed into chips having a prescribed size through a granulator.

The chips are fed to an extruder, where they are dissolved and the air contained therein is mostly removed by backflow. The concentrated solution leaving the extruder is filtered and then extruded through orifices into a vertical, several meter long spinning tube, where part of the water evaporates and the extruded filaments obtain the strength to withstand their own weight. The fluidity of the spinning solution is an important factor in fiber formation in spinning tubes, and a number of rheological studies have been conducted (21–24). One determination (25) of the relationship between the apparent viscosity upon passage through an orifice and the shearing force indicates that higher shearing force is favorable insofar as it decreases apparent viscosity, but it increases ballooning (die swell) of the extruded solution, which damages spinnability.

After being dried through a drier, the fiber is heat-drawn at a draw ratio of around 10, to obtain a serviceable strength. Various modifications have been applied to this process, to produce a variety of filament yarns including a monofilament having a diameter of 200 to 700 μm (for seaweed cultivation material and for reinforcing cement) and water-soluble yarns (26). Water-soluble yarns mostly use PVAs with low degree of

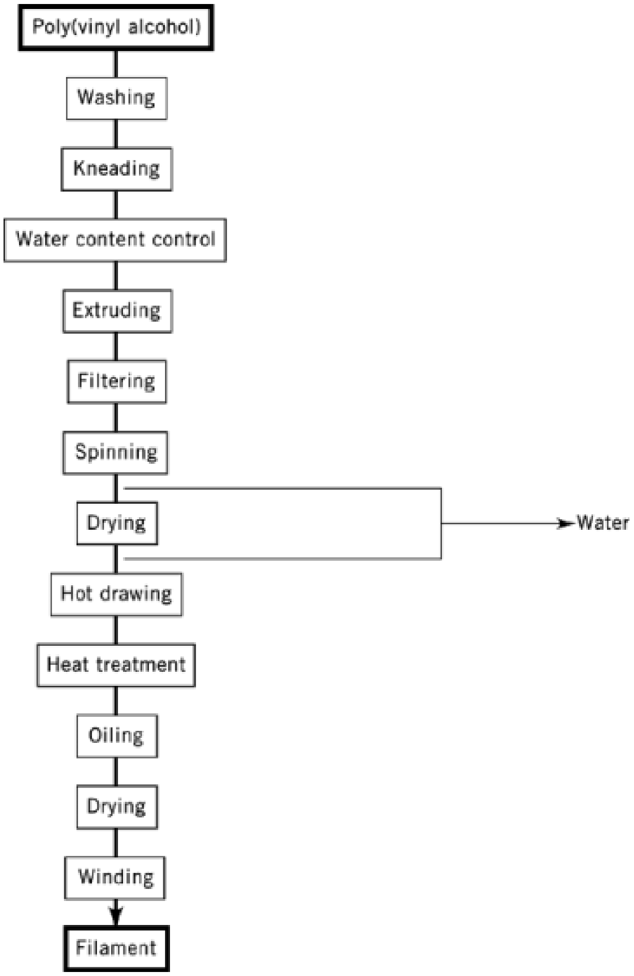

Fig. 4. Flow diagram for the manufacture of vinal by dry spinning.

polymerization, and the dissolution temperatures are adjusted by controlling the degree of hydrolysis of the raw material PVA and/or the heat-treatment conditions of the yarns (27).

2.4. Other Spinning Processes

The following examples are of scientific interest but have not been employed on an industrial scale.

2.4.1. Dimethyl Sulfoxide (DMSO) Solvent

The spinning solution is a 13–15% solution in DMSO, and the coagulating bath comprises acetone, methanol, toluene, or mixtures of the foregoing with DMSO (28). The fiber obtained as-spun can be drawn in a drawing ratio of 20 at 200°C, to give a finished fiber having a strength of 0.88 N/tex (9 gf/dtex) and a Young's modulus of 44 N/tex (450 gf/dtex).

Table 1. Mechanical Properties of Vinylon[a, b]

	Staple fiber		Filament yarn	
Property	Regular	High strength	Regular	High strength
tensile strength, N/tex[c]				
dry	0.35–0.58	0.6–0.93	0.3–0.35	0.84–1.20
wet	0.27–0.46	0.47–0.79	0.19–0.28	0.40–0.93
dry/wet strength ratio, %	72–85	78–85	70–80	75–90
loop strength, N/tex[c]	0.28–0.46	0.44–0.51	0.40–0.53	0.62–1.15
knot strength, N/tex[c]	0.22–0.35	0.40–0.46	0.2–0.26	0.24–0–4
elongation, %				
dry	12–26	9–17	17–22	6–22
wet	12–26	9–17	17–25	8–26
elastic recovery,[d] %	70–85	72–85	70–90	70–90
initial modulus, N/tex[c]	2.3–6.2	6.2–22	5.3–7.9	10.6–35.3
apparent Young's modulus, MPa[e]	2,940–7,850	7,850–28,440	6,865–9,320	13,240–45,100
moisture regain, %				
20°C, 65% rh[f]	4.5–5.0	3.5–4.5	3.5–4.5	2.5–4.5
20°C, 20% rh	regular 1.2–1.8; high strength 1.0–1.5			
20°C, 95% rh	regular 10.0–12.0; high strength 8.0–10.0			

[a] Specific gravity = $1.26 - 1.30$; official moisture regain = 5%.

[b] Ref. 33.

[c] To convert N/tex to gf/dtex, multiply by 10.2.

[d] At 3% elongation.

[e] To convert MPa to kgf/mm^2, multiply by 0.102.

[f] Standard conditions.

2.4.2. Solvent of Ethylene Glycol and Other Polyols

Highly crystalline PVAs are obtained using a solvent of ethylene glycol (EG), diethylene glycol (DEG), triethylene glycol, or glycerol, which are called single-crystal solvents (29, 30).

2.4.3. Dry-Jet Wet Spinning (Gel Spinning)

This is a new spinning process that comprises dissolving a PVA with a high degree of polymerization, extruding the solution into air, immediately thereafter introducing the extruded streams into an aqueous salt solution or an organic solvent to permit them to gel therein, and drawing the gelled filaments in a high draw ratio (31, 32). Fibers having high strength and modulus not hitherto achieved are reportedly obtained.

3. Fiber Mechanical Properties

The mechanical properties of PVA fiber vary depending on the conditions of fiber manufacture such as spinning process, drawing process, and acetalization conditions, and the manufacture conditions of raw material PVA. Table 1 shows the mechanical properties of PVA fibers commercially available in Japan under the name of vinylon (33). As apparent from the table, PVA fibers are characterized by high strength, low elongation, and high modulus. In addition to general-purpose types, high strength types with strength of at least 1.47 N/tex (15 gf/dtex) are produced by alkali spinning (34); material with a yarn strength of nearly 1.77 N/tex (18 gf/dtex) has become available (35).

4. Physical and Chemical Properties

4.1. Moisture Absorbency

PVA fiber is more hygroscopic than any other synthetic fiber. The hygroscopicity varies depending on how the fiber is processed after spinning, ie, in heat-drawing, heat-treatment, acetalization, and the like.

4.2. Dimensional Stability

The wet heat resistance of PVA fiber is indicated by the wet softening temperature (WTS) at which the fiber shrinks to a specified ratio. At one time, the WTS was not more than 95°C for nonacetalized PVA fiber, but improvement of WTS has been achieved by improvement in heat-drawing and -treating techniques; other methods proposed include suppression of polymerization temperature of vinyl acetate (36) and employment of alkali spinning (37).

On the other hand, water-soluble PVA fibers are available on the market. They are stable in cool water but shrink in warm water and dissolve at 40 to 90°C. The dissolution temperature is controlled by the degree of polymerization and hydrolysis of PVA, heat-treatment conditions after spinning, etc.

PVA fiber is better in dimensional stability under dry heat than other synthetic fibers.

4.3. Thermal Resistance and Flammability

Thermal analysis of PVA filament yarn shows an endothermic curve that starts rising at around 220°C; the endothermic peak (melting point) is 240°C, varying a little depending on manufacture conditions. When exposed to temperatures exceeding 220°C, the fiber properties change irreversibly.

With respect to flammability, PVA fiber has a limiting oxygen index (LOI) of 20, the same as that of polyester and polyamide fibers, and does not drip even when burnt, a feature which is highly valued. A flame-retardant-grade PVA fiber is available; it is obtained by mix-spinning with poly(vinyl chloride). This fiber, having an LOI of 30–34 and high strength, is used for bedding and workclothes.

4.4. Weather Resistance

It has been shown for over 40 years that PVA fiber has excellent resistance against exposure to sunlight. The resistance to uv light is also excellent, which has been proven with the tents used in Antarctic expeditions and by Himalayas' climbing parties. Nets for golf practice ranges maintained 90% of their initial strength after actual exposure for seven years (38). A PVA nonwoven fabric used for reinforcing an asphalt roofing maintained, after 10 years of actual use, 100% of the initial fiber strength (39).

4.5. Chemical Resistance

Table 2 shows the chemical resistance of PVA fiber (40). The fiber exhibits markedly high resistance to organic solvents, oils, salts, and alkali. In particular, the fiber has unique resistance to alkali, and is hence widely used in the form of a paper principally comprising it and as reinforcing material for cement as a replacement of asbestos.

5. Application

In the 1950s and 1960s PVA fiber had been used for clothing and interiors, but after commercialization of polyester fiber and acrylic fiber, PVA fiber gradually lost its market share in these fields because of problems

Table 2. Comparison of Various Fibers' Resistance to Chemicals

	Testing conditions			Retention of strength[a], %			
Chemical	Concen-tration, %	Temper-ature, °C	Time, h	Kuralon[b]	Cotton	Nylon	Poly-ester
acid							
sulfuric	10	20	10	100	51	56	100
hydrochloric	10	20	10	100	70	77	95
alkali							
sodium hydroxide	40	20	10	100	84	82	97
salt							
sodium carbonate	1	100	10	97	75	99	95
sodium chloride	3	100	10	100	90	86	98
organic solvent							
benzene	100	20	1000	100	100	88	98
carbon tetrachloride	100	20	1000	100	90	82	87
miscellaneous							
mineral oil	100	100	10	100	70	100	100
cottonseed oil	100	20	1000	100	100	93	100
lard	100	20	1000	100	100	76	100

[a] Control showed 100% retention.
[b] Kuralon is the registered name of Kuraray's PVA fiber.

in elastic recovery, dye-ability, cost, etc. On the other hand, the fiber has found uses in a variety of industrial fields thanks to its superior mechanical properties of high strength and modulus and low elongation, resistance to chemicals, in particular to alkali, high durability against uv light, etc.

5.1. Rubber Industry

This is the oldest industrial area in which PVA fiber has been successfully applied. The fiber is used for reinforcement for belts, hoses, and the like, where its high strength and modulus and low elongation are highly appreciated. The fiber is also easy to treat with resorcinol–formaldehyde latex and has high adhesiveness.

PVA fiber is best fit for reinforcement of oil brake hoses for cars that require high reliability, because of excellent mechanical properties and good chemical resistance to pressure–transmission liquid contained in the hose.

5.2. Agricultural Materials

PVA fiber is used in this field mostly in the form of shade cloth. The mesh cloth protects plants and vegetables not only from vermin but also from frost or excessive sunlight. In some cases the mesh cloth, by reflecting far infrared light, is used for warmth keeping purposes. In these applications the high weather resistance of the fiber plays an important role, in addition to high strength and excellent dimensional stability.

5.3. Fishing Materials

Since the late 1960s, the fiber has been losing its market share in the field of fishing nets, because of other fiber materials superior in water-separatability and resistance to waves. At present, the fiber is used, because of chemical and physical uniqueness of the fiber surface, for nets for cultivating seaweed, an important food in Japan and Korea. Long lines for fishing tuna also use PVA fiber.

PVA fiber ropes are widely used in fishing and on ships, because of excellent weather resistance, coiling property, ease of handling, twist stability, etc. For this purpose spun yarns obtained directly from tow by the Perlok spinning system are used.

5.4. Sewing Thread

Spun yarns and filament yarns of PVA fiber with their characteristics of low elongation and high strength are used as industrial sewing threads for leather materials such as shoes and bags and for similar items. In Japan, the PVA fiber threads are also used for sewing tatami mattress.

5.5. Nonwoven Fabric

Crimped PVA staple is being used for the manufacture of dry-laid nonwoven. Also, as an example utilizing the uniqueness of the fiber, a soft sheet is prepared by shrinking and partly dissolving in hot water a nonwoven from water-soluble PVA fiber and then insolubilizing the fabric by acetalization or similar processes. This sheet is used as car wipers, wipers for high grade furniture, and for similar purposes.

Slivers obtained by directly stretch-breaking tow through the Perlok spinning system are formed into a nonwoven by a random web process. The nonwoven is impregnated with asphalt (qv) and this composite is used as roofing material. This usage makes the most of the excellent asphalt-impregnability, as well as the high strength and modulus, of PVA fiber.

5.6. Paper

Both warm-water-soluble and water-insoluble PVA fibers are used as raw materials for paper, the former as a fiber-shaped binder and the latter as a main, structural-member fiber singly or in combination with other fibrous materials, to yield unique species of paper. The insoluble type fiber is used, for example, in combination with wood pulp to improve paper strength or in combination with the water-soluble type to give an alkali-resistant paper. Paper containing PVA fiber is excellent in folding characteristics and has markedly high tensile strength. Because its porosity is readily controllable and it has high alkali resistance, the paper is widely used in the world as separator for alkali dry battery.

5.7. Reinforcement

PVA fiber in the form of short cut chips having a length of several millimeters to several tens of millimeters is widely used as raw material for paper and for reinforcing plastics, cement, and the like, and has been acquiring more and more significance.

5.7.1. Fiber-Reinforced Plastics

PVA fiber, with its high strength and toughness, has become widely used as reinforcement fiber for FRP. Strength as high as 1.8 N/tex (18 gf/dtex) has been reported (32, 41). Penetration in this field will therefore become more active.

5.7.2. Fiber-Reinforced Cementitious Material

Use of asbestos (qv) has been legally restricted in Europe and the United States as being hazardous to health. In asbestos cement, which had consumed 70–80% of total asbestos, PVA fiber has been used in large amount as a replacement for asbestos. PVA fiber has a strength of at least 0.88 N/tex (9 gf/dtex) and can therefore provide the necessary reinforcement for cement; the fiber has excellent adhesiveness to cement (qv) and alkali resistance, and is not a health hazard.

Based on the technology developed for using PVA fiber as a replacement for asbestos in cement products, Kuraray has been developing thick fibers for reinforcing concrete (42). Super-thick fibers with a thickness of 39 tex (350 den) (200 μm in diameter) to 444 tex (4000 den) (660 μm in diameter) are now available; the 39 tex material is used for reinforcing various mortar-based cement products and the 444 tex material for reinforcing concrete in civil engineering works such as tunnels, roads, harbors, and bays.

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Related Articles

Vinyl polymers, poly(vinyl alcohol); Fluorine compounds, organic, polytetrafluoroethylene; Nonwoven fabrics, Fibers, survey