

12

**EUROPEAN PATENT APPLICATION**

21 Application number: 79300077.9

51 Int. Cl.<sup>2</sup>: **D 04 H 1/64**  
**A 61 L 15/00, A 47 K 10/16**

22 Date of filing: 17.01.79

30 Priority: 18.01.78 US 870551  
18.01.78 US 870552

43 Date of publication of application:  
25.07.79 Bulletin 79/15

84 Designated contracting states:  
DE FR GB IT NL

71 Applicant: **AIR PRODUCTS AND CHEMICALS, INC.**  
P.O. Box 538  
Allentown, Pennsylvania 18105(US)

72 Inventor: **Davidowich, George**  
602 Madison Avenue  
Dunellen New Jersey 08812(US)

72 Inventor: **Miller, Gerald Donald**  
RD3 Box 253  
Bell Mead New Jersey 08502(US)

72 Inventor: **Daniels, Wiley Edgar**  
3760, Baldwin Drive  
Easton Pennsylvania 18042(US)

74 Representative: **Lucas, Brian Ronald**  
c/o Air Products Limited Coombe House St. George's  
Square  
New Malden, Surrey(GB)

54 Moist packaged towelette and method of making same.

57 A moist packaged towelette comprises a sheet of nonwoven fabric provided with a binder comprising polyvinyl alcohol in contact with an aqueous solution in a container. The aqueous solution contains a sufficient concentration of a compound which prevents the polyvinyl alcohol dissolving in the aqueous solution but which, when dissolved in excess water, allows the polyvinyl alcohol to dissolve in the excess water thereby reducing the structural integrity of the towelette. Suitable compounds include boric acid and sodium sulphate. The binder may consist of pure polyvinyl alcohol or a mixture of polyvinyl alcohol and polyvinyl acetate. A mixture of polyvinyl alcohol, vinyl acetate-ethylene copolymers and, if desired, polyvinyl acetate, can also be used as a binder.

This invention relates to a moist packaged towelette and to a method of making the same.

Moist packaged towelettes are generally made by coating a sheet of nonwoven fabric, usually absorbent paper, with a binder and storing the coated fabric in contact with an aqueous solution in a container. The aqueous solution typically contains alcohol and, for example, a perfume or deodorant. The binder increases the structural integrity of the nonwoven fabric and prevents it disintegrating in the aqueous solution. (For the avoidance of doubt the term "nonwoven fabric" as used herein includes fabrics comprising carded or randomly orientated or cross-laid fibres. The fibres may comprise, for example, natural or regenerated cellulose, other synthetic or proteinaceous fibres of biodegrade materials, or mixtures of these).

Various binders have been proposed but all which are known to us have the disadvantage that the coated fabric maintains a high structural integrity even after prolonged exposure to water. This can result in blocked drains.

One known binder for nonwoven fabrics which are not intended to be exposed to moisture is polyvinyl alcohol. Whilst nonwoven fabrics provided with this binder have excellent structural integrity in the dry they disintegrate rapidly when immersed in water.

We have discovered that a nonwoven fabric provided with a binder comprising polyvinyl alcohol will retain a high structural integrity in contact with an aqueous solution provided that the aqueous solution contains a sufficient concentration of a compound to prevent the polyvinyl alcohol dissolving in the aqueous solution. When the solution is diluted the polyvinyl alcohol dissolves thereby weakening the structural integrity of the towelette.

Accordingly, the present invention provides a moist packaged towelette comprising a sheet of nonwoven fabric

provided with a binder and maintained in contact with an aqueous solution in a container characterized in that said binder is polyvinyl alcohol and said aqueous solution contains a compound which is present in said solution at a sufficient concentration to prevent said polyvinyl alcohol dissolving in said aqueous solution but which, when diluted in excess water, permits said polyvinyl alcohol to dissolve in said excess water thereby reducing the structural integrity of said towelette.

Compounds which will prevent polyvinyl alcohol dissolving in aqueous solutions are well known and examples are listed in "Polyvinyl Alcohol", 1973; Finch C.A., John Wiley & Sons Ltd., Table 2.3 at page 40. Substantially all of these compounds (which comprise boric acid and water soluble salts) will, when dissolved in excess water, permit the polyvinyl alcohol to dissolve thereby reducing the structural integrity of the nonwoven fabric. It is, of course, a simple procedure to check if a particular compound is suitable.

Of the available compounds boric acid is presently preferred and preferably comprises at least 1% (by weight) of the aqueous solution with 3% to 5% (by weight) being preferred and 4% to 5% (by weight) being more preferred.

One of the problems associated with using a water soluble salt as the compound is that the required concentration in the aqueous solution is so high that, in many cases, the salt crystallizes on the skin when the towelette is used. We strongly recommend that the salts which are used should be those which need be present to no more than 40% (by weight) of the aqueous solution and, more preferably, to no more than 20% (by weight) of the aqueous solution.

If a salt is to be used then we recommend sodium sulphate at a concentration of between 3% and 20% (by weight) of the aqueous solution and more preferably between 7% and 20% (by weight) of the aqueous solution.

Polyvinyl alcohol is generally produced by the hydrolysis of polyvinyl acetate. Pure polyvinyl alcohol (i.e. 100% hydrolysed polyvinyl acetate) is relatively insoluble in water at room temperature when compared with 80% to 99% hydrolysed polyvinyl acetate. Accordingly, the binder preferably comprises a mixture of polyvinyl alcohol and polyvinyl acetate. If desired the polyvinyl alcohol may comprise as little as 1% (by weight) of such a mixture although it preferably comprises between 80% and 95% thereof.

10 Mixtures (emulsions) comprising vinyl acetate-ethylene copolymers and polyvinyl alcohol may also be used as a binder. In such a case the polyvinyl alcohol preferably comprises from 1% to 10% (by weight) of the binder and probably acts as a protective colloid. If desired the binder may  
15 also contain polyvinyl acetate and preferably between 5 and 25% (by weight) thereof. One particularly preferred range of binders comprises (by weight) 1 to 10% of 80 - 90% hydrolysed polyvinyl acetate and the balance vinyl acetate-ethylene copolymers. Preferably the vinyl acetate-ethylene copoly-  
20 mers contain (by weight) not more than 45% vinyl acetate and not more than 60% ethylene.

In all the above cases the weight of the binder is preferably between 5% and 50% of the weight of the untreated non-woven fabric.

25 The present invention also provides a method for making a moist packaged towelette which method comprises the step of wetting a sheet of nonwoven fabric provided with a binder by bringing said sheet into contact with an aqueous solution characterized in that said binder comprises polyvinyl alcohol  
30 and said aqueous solution contains a sufficient concentration of a compound which prevents the polyvinyl alcohol dissolving in the aqueous solution but which, when dissolved in excess water, allows the polyvinyl alcohol to dissolve in the water thereby reducing the structural integrity of the towelette.

The present invention also provides a method for making a moist packaged towelette which method comprises taking a sheet of nonwoven fabric which has been provided with a binder and packaging said sheet of treated material in contact with an aqueous solution in a container characterized in that said binder comprises polyvinyl alcohol and said aqueous solution contains a sufficient concentration of a compound which prevents the binder from dissolving in the aqueous solution but which, when dissolved in excess water, allows the binder to dissolve in the water thereby reducing the structural integrity of the towelette.

Preferably said compound is boric acid and advantageously said aqueous solution comprises at least 1% (by weight) of said compound with 3% to 5% (by weight) being preferred and 4% to 5% (by weight) being more preferred.

The container should preferably be impermeable to all the components of the aqueous solution. However, for economic reasons a container need only be sufficiently impermeable to the components of the aqueous solution for a limited period of time, for example, the anticipated time delay between manufacture and use. The container itself may be in the form of a sachet for accommodating a single towelette or a bag or box for accommodating a plurality of towelettes. In the latter cases the bags or boxes are preferably resealable to minimise evaporation of the aqueous solutions.

The binder may conveniently be applied to the nonwoven fabric by making an aqueous solution (or emulsion) of the binder and applying it to the fabric by, for example, a roller or a spray gun. Alternatively, the nonwoven fabric may simply be dipped in the aqueous solution (or emulsion).

Once treated the nonwoven fabric is preferably dried, and is then cut and, if desired, folded. The nonwoven fabric may then either be wetted by the aqueous solution and inserted in a container or inserted in a container and wetted. It should be understood that it is not essential to

dry the nonwoven fabric after the application of the binder  
although drying is preferred for ease of handling.

5

10

15

20

25

30

35

0003186

For a better understanding of the invention reference will now be made to the following non-limiting examples.

EXAMPLE 1

5 A sheet of 24 pound (25 x 38 inch - 500  
ream) (10.9 Kg - 27.7 96.5 cm) high ground-  
wood, unsized paper was immersed in water for  
two minutes. The wet sheet was found to have a  
tensile strength of approximately 0.59 pounds  
(0.27 Kg).

10 EXAMPLE 2

A sheet of the same paper used in Example  
1 was impregnated with a solution of VINOL  
(Trade Mark) 205 polyvinyl alcohol (PVOH) to  
the extent of 4 pounds (1.80 Kg) dry add-on and  
15 dried in a 120°C forced air oven. After imm-  
ersion in water for two minutes the wet sheet  
was found to have a tensile strength of 0.59  
pounds (0.27 Kg), i.e. approximately equal to  
the wet sheet in Example 1. (VINOL 205 is 87%  
20 to 89% hydrolysed polyvinyl acetate of low vis-  
cosity (4-6 cps) marketed by Applicants).

EXAMPLE 3

Two sheets of paper were prepared and  
dried as in Example 2. However, instead of  
25 immersion in water both sheets were immersed in  
an aqueous solution containing 5% (by weight)  
boric acid at room temperature.

After immersion for two minutes one wet  
sheet was tested and found to have a tensile  
30 strength of 1.6 pounds (0.73 Kg), i.e. nearly 3  
times the tensile strength in Example 1 and 2.

The other wet sheet was then immersed in a  
large quantity of water for a further two min-  
utes and when tested was found to have a wet

35

tensile strength of less than 0.8 pounds (0.365 Kg).

EXAMPLE 4

5 A sheet of paper was prepared and dried as  
in Example 2. The sheet was then immersed in  
an aqueous solution containing 5% (by weight)  
boric acid at room temperature for 1 year. On  
removal from the solution there was no detect-  
able reduction in tensile strength as measured  
10 by finger pull.

EXAMPLE 5

In order to determine the probable shelf  
life of the packaged towelette films of 15 ml.  
(0.038 cm) wet thickness were separately cast  
15 from VINOL 205 and VINOL 540 PVOH and dried at  
room temperature. Strips of the films of  
1 x 6 inches (2.54 x 15.24 cm) were then imm-  
ersed in an aqueous solution containing 5% (by  
weight) boric acid at various temperatures.  
20 The probable shelf life of the packaged towel-  
ette at various temperatures is indicated in  
Table 1.

TABLE 1

25	80°F (26.7°C)	130°F (54.4°C)	160°F (71.1°C)
VINOL 205	300 days	30 days	16 hours
VINOL 540	300 days	30 days	3 days

30 In contrast all the films dissolved with-  
in 5 minutes when immersed in ordinary water.  
(VINOL 540 is 87% to 89% hydrolysed polyvinyl  
acetate of high viscosity (40 - 50 cps) market-  
ed by Applicants). 80% hydrolysed PVOH is  
35 commonly known to have reverse solubility,



i.e. is insoluble in water above 20°C but is soluble at room temperature. For this reason towelettes should advantageously be coated with this material for use in high temperature atmospheres.

#### EXAMPLE 6

A high groundwood stock paper substrate (24 pound/3300 ft.<sup>2</sup> = 10.9 Kg/307 sq. meters) was treated with a 15% aqueous solution of VINOL 205 PVOH applied with a No. 10 Mayer rod separately to each side of the paper and dried at 250°F ( 120°C) for 30 seconds. The coated first side was dried before applying the coating to the other side.

The dried paper was then immersed for two minutes in a 5% boric acid solution and its wet tensile strength determined by Instron (C) and compared with that of the base stock (A) and the coated sheet without boric acid (B). The results are reported in Table 2 below. The resolubility was demonstrated by further immersion of the boric acid treated sheet in plain water for two minutes (D).

TABLE 2

		Instron Wet Strength (pounds/kgs)
A.	Base stock after immersion in water (untreated)	0.55/0.25
B.	Treated with PVOH and then immersed in water	0.59/0.27
C.	Treated with PVOH, then immersed in aqueous boric acid (5% molten)	1.73/0.79
D.	Reimmersion in excess water after C.	0.70/0.32

EXAMPLE 7

Further studies were carried out to determine the effect of boric acid concentration on the wet tensile strength of PVOH in pregated papers. These studies were made on paper sheets of a 42 pound/3300 sq. ft. stock (19 kg/307 sq. meters) each respectively immersed in boric acid solution of successively increasing concentrations. It was found that the wet tensile strength increased almost linearly with concentration from 0.72 pounds (~ .33 kg) at zero boric acid to 1.41 pounds (~ 0.64 kg) at 5% boric acid.

EXAMPLE 8

Papers treated with other grades of polyvinyl alcohol were tested to determine the effect of boric acid in inhibiting disintegration. These included commercial grades identified as:

	<u>% Hydrolysis</u>	<u>Viscosity (cps)</u>
VINOL 540	87-90	40-50
VINOL 605	80	4.4-5.2
VINOL 650	80	40-60
VINOL 107	98-98.8	5-7

Each of these VINOL compositions were applied to a 24 lb./3300 ft.<sup>2</sup> (10.9 Kg/307 sq. meters) base stock and dried at 250°F (120°C) for 30-90 seconds, as required. The amount of PVOH add-on varied due to viscosity differences so that the measured wet tensile values are not directly relatable between the grades.

All of these PVOH treated sheets exhibited wet tensile improvement with 5% boric acid

immersion versus water immersion and all showed resolubility in plain water after short immersion in boric acid solution, as shown in Table 3.

5

TABLE 3Wet Strength (lbs)

				Boric Acid then Water
	<u>% Add on</u>	<u>Water</u>	<u>Boric Acid</u>	
10	VINOL 205	17	0.60	1.73
	VINOL 540	31	0.90	>2.0
	VINOL 605	11	0.64	1.56
	VINOL 650	27	0.80	>2.0
15	VINOL 107	--	1.27	>2.0

EXAMPLE 9

The water soluble salts listed in Table 4 below are believed to be suitable substitutes in place of boric acid, (which is also listed for comparison) at concentrations of up to 20% (by weight) in the articles of this invention. Table 4 shows the minimum concentration causing precipitation of the compound dissolved in a 5% solution of polyvinyl alcohol (98-99% hydrolysed, degree of polymerization 1700-1800).

30

35

TABLE 4\*

	<u>Compound</u>	Minimum concentration for salting out (g/l)
5	$(\text{NH}_4)_2\text{SO}_4$	66
	$\text{Na}_2\text{SO}_4$	50
	$\text{K}_2\text{SO}_4$	61
	$\text{FeSO}_4$	105
	$\text{MgSO}_4$	60
10	$\text{Al}_2(\text{SO}_4)_3$	57
	$\text{KAl}(\text{SO}_4)_2$	58
	Potassium citrate	38
	$\text{H}_3\text{BO}_3$	16.5

15 \*Data on the soluble salts of Table 4 were taken from Finch, C.A., POLYVINYL ALCOHOL, 1973; John Wiley & Sons, Ltd., Table 2.3 at page 40.

EXAMPLE 10

20 Cast films of VINOL 205 PVOH (1" x 6" = 2.5 x 15.24 cm) were separately tested to determine solubility respectively in boric acid solutions and in sodium sulphate solutions at different concentrations. The results are reported in Table 5.

TABLE 5

	<u>Solute</u> <u>g/100 cc water</u>	<u>Film</u> <u>Description</u>
25	<u>Sodium Sulphate</u>	
	5	Soluble; 30 seconds
	10	Slimy
30	15	Slimy
	20	Insoluble; transparent film
	30	Insoluble; transparent film
	<u>Boric Acid</u>	
35	1	Soluble; 2 minutes
	3	Stringy
	5	Insoluble; turned white opaque in 2 minutes

From the foregoing tests it appears that while the soluble salts listed in Table 4 above, such as sodium sulphate, can be employed to retard solubilization of polyvinyl alcohol films, greater concentrations i.e. about 7% to about 20%,  
5 are required than when using boric acid.

As projected from the data set forth in Tables 4 and 5, potassium citrate appears to be even more efficient than sodium sulphate.

The specific behaviour of boric acid in retaining solub-  
10 ilization of PVOH film is not attributable to the pH of the boric acid solution. Whereas a VINOL 205 film was insoluble in 5% boric acid solution, such film was readily dissolved respectively, in 5% aqueous solution of citric and phosphoric acid and a 0.7% solution of fumaric acid.

15 EXAMPLE 11

A 60% vinyl acetate-40% ethylene copolymer emulsion containing 4% PVOH (75% VINOL 205 and 25% VINOL 523) (by weight) of the copolymer, and containing a total of 52% solids was  
20 cast to form a film of 15 mil wet thickness and air dried. While the film retained its definition when immersed in water, it exhibited practically no wet tensile strength as evidenced by the fact that it could not suspend its  
25 own weight.

When immersed in a 5% boric acid solution, the film exhibited surprisingly good wet tensile strength and was highly elastic. However, this film removed from the boric acid solution  
30 was redispersed in plain water in less than two minutes.

The treated film in contact with boric acid solution retained wet tensile strength for more than 30 days at 130°F (54.4°C). At  
35 160°F (71.1°C) the film retained wet tensile

strength for 3 days indicating excellent film stability and shelf life at the elevated temperature that may be experienced under storage conditions.

5

EXAMPLE 12

The same emulsion as employed in Example 11 was diluted and applied to a paper substrate.

10

The emulsion was diluted with water to a 25% total solids content and applied to both sides of a 42 pound/3300 square foot (19 Kg/307 mg/square meters) paper substrate, and the treated paper dried at 120°C in a forced air oven. The pick-up was 3.5 pounds (1.59 Kg) dry emulsion.

15

A sample of the dried emulsion treated paper, as determined by conventional Instron test, showed a wet tensile strength after immersion in water, of 1.08 pounds (0.49 Kg) as compound to the untreated stock which showed a wet tensile of 0.72 pounds (0.33 Kg).

20

A duplicate sample of the dried emulsion treated paper immersed in 5% boric acid solution for 2 minutes when tested by Instron exhibited a tensile of 1.41 pounds (0.64 Kg). When reimmersed in plain water for 2 minutes, the paper returned to about its initial wet strength, 1.09 pounds (0.49 Kg).

25

Another duplicate sample of the dried emulsion treated paper was immersed in 5% boric acid solution for 30 minutes maintained about the same tensile strength as that previously

30

35

shown for the boric acid treatment while the water value on reimmersion decreased to 0.91 pounds (0.41 Kg).

It should be noted that the paper in the foregoing example had a relatively low dried emulsion add-on. At higher add-on levels or lower basis weight substrate greater relative increase in tensile strength may be realized.

EXAMPLE 13

While in Example 10 and 11 boric acid is employed as the agent for increasing the wet strength of the nonwoven fiber sheet during storage and use, certain soluble salts known to react with polyvinyl alcohol to effect precipitation or gelling thereof, may be employed. These are less preferred than boric acid, however, since larger concentrations of these are required for the desired purpose. Examples of such salts are set out in Table 4 hereinbefore.

EXAMPLE 14

Cast films of the same emulsion as employed in Example 11 (1" x 6" = 2.5 x 15.24 cm) were separately tested to determine solubility respectively in boric acid solutions and in sodium sulphate solutions at different concentrations. The results are reported in Table 2.

TABLE 2

	<u>Solute</u> <u>g/100 cc water</u>	<u>Film</u> <u>description</u>
	<u>Sodium sulphate</u>	
5	0	Weak film
	5	Some film strength develop- ment
	20	Stronger film
	<u>Boric acid</u>	
10	0	Weak film
	1	Some film strength develop- ment
	3	Stronger film
	5	Optimum film strength

15        From the foregoing results, it appears that while the soluble salts, such as sodium sulphate, can be employed to retard solubilization of polyvinyl acetate films, somewhat greater concentrations, i.e. about 3% to about 20%, are required than when using boric acid.

20

25

30

35



CLAIMS

1. A moist packaged towelette comprising a sheet of non-woven fabric provided with a binder and maintained in contact with an aqueous solution in a container characterized in that  
5 said binder is polyvinyl alcohol and said aqueous solution contains a compound which is present in said solution at a sufficient concentration to prevent said polyvinyl alcohol dissolving in said aqueous solution but which, when diluted in excess water, permits said polyvinyl alcohol to dissolve  
10 in said excess water thereby reducing the structural integrity of said towelette.
2. A moist packaged towelette according to Claim 1, characterized in that said compound is boric acid.
3. A moist packaged towelette according to Claim 2, characterized in that said boric acid comprises at least 1% (by  
15 weight) of said aqueous solution.
4. A moist packaged towelette according to Claim 3, characterized in that said boric acid comprises 3% to 4% (by weight) of said aqueous solution.
- 20 5. A moist packaged towelette according to Claim 4, characterized in that said boric acid solution comprises 4% to 5% (by weight) of said aqueous solution.
6. A moist packaged towelette according to any preceding Claim, characterized in that said compound comprises a water  
25 soluble salt and said aqueous solution contains from 3% to 20% (by weight) of said salt.
7. A moist packaged towelette according to Claim 6, characterized in that said salt is sodium sulphate.
8. A moist packaged towelette according to any preceding  
30 Claim, characterized in that said binder comprises polyvinyl acetate.
9. A moist packaged towelette according to Claim 8, characterized in that said binder comprises between 80% and 99% (by weight) polyvinyl alcohol.

10. A moist packaged towelette according to Claim 8, characterized in that said binder comprises vinyl acetate-ethylene copolymer.
11. A moist packaged towelette according to Claim 10, characterized in that said vinyl acetate-ethylene copolymers  
5 comprises from 10% to 40% (by weight) vinyl acetate.
12. A moist packaged towelette according to Claim 10 or 11 when appended to Claim 8, characterized in that said binder comprises from 1% to 10% (by weight) polyvinyl alcohol.
- 10 13. A method for making a moist packaged towelette which method comprises the step of wetting a sheet of nonwoven fabric provided with a binder by bringing said sheet into contact with an aqueous solution characterized in that said binder comprises polyvinyl alcohol and said aqueous solution  
15 contains a sufficient concentration of a compound which prevents the polyvinyl alcohol dissolving in the aqueous solution but which, when dissolved in excess water, allows the polyvinyl alcohol to dissolve in the water thereby reducing the structural integrity of the towelette.
- 20 14. A method for making a moist packaged towelette which method comprises taking a sheet of nonwoven fabric which has been provided with a binder, and packaging said sheets of treated material in contact with an aqueous solution in a container characterized in that said binder comprises poly-  
25 vinyl alcohol and said aqueous solution contains a sufficient concentration of a compound which prevents the binder from dissolving in the aqueous solution but which, when dissolved in excess water, allows the binder to dissolve in the water thereby reducing the structural integrity of the towelette.
- 30 15. A method according to Claim 13 or 14, characterized in that said compound is boric acid.



DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (Int. Cl. <sup>2</sup> )
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	
A	<u>GB - A - 1 510 667</u> (DEXTER) * Page 2, lines 1-4; claim 1 *	1	D 04 H 1/64 A 61 L 15/00 A 47 K 10/16
	--		
	<u>US - A - 3 692 725</u> (D.V.DUCHANE) * Column 1, lines 46-50; column 6, lines 14-23; claim 1 *	1,2	
	--		
	<u>US - A - 3 886 112</u> (J.J. WATSON et al.) * Column 2, lines 24-36; claim 1 *		TECHNICAL FIELDS SEARCHED (Int.Cl. <sup>2</sup> )  D 04 H 1/64 A 61 L 15/00 A 47 K 10/16
	----		CATEGORY OF CITED DOCUMENTS  X: particularly relevant A: technological background O: non-written disclosure P: intermediate document T: theory or principle underlying the invention E: conflicting application D: document cited in the application L: citation for other reasons
<input checked="" type="checkbox"/>	The present search report has been drawn up for all claims		&: member of the same patent family, corresponding document
Place of search  The Hague		Date of completion of the search  03-04-1979	Examiner  ELSEN-DROUOT