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# PATENT SPECIFICATION

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## COMPLETE SPECIFICATION

### Polyolefine-Coated, Reticulated Polyurethane Foams

We, SCOTT PAPER COMPANY, a Corporation organized under the laws of the State of Pennsylvania, United States of America, of International Airport, Philadelphia 13, State of Pennsylvania, United States of America, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention pertains to novel structures and their method of making, more particularly, this invention pertains to a structure at least partially formed of a polyolefin material.

The novel structure of the present invention may have a reticulated polyurethane foam polymer core intimately surrounded by a completely covering outer envelope of a polyolefin material such as polyethylene or polypropylene, which is attached to said reticulated polyurethane foam material by first wetting the polyurethane structure with a liquid, preferably water, and then depositing on the wetted structure a polyolefin powder which is subsequently melted in a heat zone to form said envelope. The polyurethane core may be removed, if desired. Novel products of this invention are characterized by the substantial absence of membranous material.

Prior art fully-reticulated foam structures have generally been made from polyurethanes, although polyvinylchloride foams of acceptable permeability have recently been obtained in reticulated form by novel explosion techniques.

The term "reticulated" is well known in the foam art and defines a net-like product consisting of nexae and strands joined together at spaced apart points to form a continuous structure. Generally, a prerequisite for foam reticulation is either an open-celled structure (intercommunicating cells) for explosion and chemical reticulation or a structure susceptible to partial chemical attack resulting in cell-wall removal. For the last process open-celled structure is also a desideratum. For explosion

reticulation open-celled foams are needed to allow the gas to be introduced into the interior thereof.

Polyurethane structures suffer from poor chemical resistance to acid and alkali attack and are subject to considerable swelling when in contact with most organic solvents, while polyvinylchloride foams allow little pore size and pore uniformity control.

Most polyolefin foams such as polyethylene and polypropylene foams are close-celled; i.e., there is little if any intercommunication. This phenomenon, therefore, prevents the close-reticulated polyolefin foams from being explosively reticulated.

It is also well known that polyolefins are extremely resistant to acid or alkali attack and do not swell in most solvents. Because of this resistance, reticulated polyolefin structures have always been a long-sought goal in acid- or alkali-filtering applications. Various processes have been used to obtain suitable filtering devices made of polyethylene. For example, polyethylene shreds or floss have been made into a filter element. Other processes have been directed to sintering polyethylene particles into a cohesive filter. Even though the polyolefin foams have been made and even if they could be reticulated (presently believed to be impossible), pore size control of the polyolefin foams in the range desired in filtering operations is practically non-existent when compared to the pore size control possible with polyurethane foams.

Prior art processes for coating articles of various kinds are known. For example, a molten polyethylene may be used to coat a pre-heated article. However, the viscosity of the molten mass is so high (at the temperature ranges at which polyethylene will not decompose) that it is particularly wasteful to use the dipping method in producing polyethylene-covered articles. Surprisingly, if reticulated polyurethane is immersed in molten polyethylene the whole foam structure becomes

clogged and filled with the polyethylene.

Other methods used for coating articles with polyethylene have been based on the relative heat transfer and content of a fluidized bed of polyethylene powder and a heated article immersed in the fluidized bed. As can easily be envisioned, metal articles are most useful in this process. However, in the case of thin metal sections (or sections incapable of maintaining a high heat content), the process presents many problems in obtaining a satisfactory coating. Obviously, because of the poor heat transmissibility and heat characteristics, foamed organic polymeric materials have not been reported to have been coated in the polyethylene powder fluidized bed.

Other methods such as an electrostatic coating system are not applicable to polyurethane foams because the foam is not conductive. According to this method, only relatively flat metal sheets can be coated, resulting in very thin coatings.

It has now been discovered that novel structures having the essential characteristics of a reticulated polyolefin foam can be produced having the desirable pore size features of polyurethane polymers and the chemical resistance of polyolefins.

In general, reticulated polyurethane structures and the correspondingly produced composites, consist of strands joined to nexae at spaced-apart points, forming approximately dodecahedrons, the faces of which are, in general, of pentagonal shape substantially free of any membranous material. This configuration of foam structures is somewhat idealized and, as obtained, the dodecahedral building blocks will vary a little.

Even though the cells are randomly oriented and thus anisotropic to each other, the whole structure behaves something like an isotropic body. This property of the foam is very desirable as the pressure-drop characteristics are superior to some of the prior-art filtering pads. Thus, structures made of the novel composites are especially desirable for acid- and alkali-filtering applications because of the uniform pore size and more isotropic nature when compared to the anisotropic filtering and contacting means such as polyethylene mats.

Moreover, other characteristics of these novel composites show improved properties in the applied stress-strain relationship when compared to regular polyurethane foams. For example, the energy required to compress the novel composites is significantly higher for polyethylene-coated reticulated polyurethane foam than uncoated polyurethane foam. This property finds ready application in self-supporting filters.

Equally surprising results are obtained when the novel composites are heat-formed into various shapes desired. By heating the polyethylene-polyurethane reticulated structure and then holding this structure in the desired form

and cooling it, a permanently set structure is obtained which will not spring back to the previous shape as polyurethane alone will do. This property of the novel structure enables production of various shape-retaining forms of the structure in hard-to-conform contours.

Further, bonding of the novel structure to various other materials is very easy because melted polyethylene exhibits good adhesion. For example, composite filters of varying pore sizes can be prepared by melting the polyethylene on the surface of one novel structure, and joining it to another of the present structures having less pores per lineal inch. Needless to say, various composites may be prepared in this manner. Similarly, the novel structure may be bonded to any roughened surface possessing sufficient affinity for molten polyethylene.

If normal polyurethane foams are heat-shaped, the original structure is densified to a considerable degree. The pressure-drop characteristics of the structure suffer as a result. Elaborate steps have been taken to avoid this densification, and although some very cumbersome methods have offered a partial solution to the shaping problem, the methods and the results have been less than desirable. For example, casting sheets of polyurethane, and then shaping and curing these before a critical time, has been one less-than-satisfactory approach. All these problems can now be avoided by heat-shaping the novel polyethylene polyurethane composites.

Novel composites of the present invention are useful in applications such as heating plant filters, humidifier and evaporative cooler pads (in this connection surface modifiers such as glass, asbestos, feldspar and pumice may be used). For example, using the coated reticulated polyurethane foam as a substrate and an adhesive to deposit on it different particulate materials or powders, such as those mentioned above, and others such as alumina and carborundum, novel surface-modified structures may be obtained.

It has now been found that many filtering, shaping and other problems encountered with polyurethanes have also been solved by essentially-reticulated polyolefin structures resembling the reticulated polyurethane structure, produced by enveloping reticulated polymeric polyurethane foam structures with a polyolefin envelope. Other products resulting from the novel structures are densified materials having good porosity and permeability, yet possessing great structural strength. The structures can be densified by building up the polyolefine coating in such a way that the thickness of the strands and nexae of the foam is increased, the void spaces of the foam thereby being decreased. Densification of from 2 to 15 times the original volume has been achieved.

The invention is accomplished by wetting, preferably with water, a reticulated poly-

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urethane foam structure, distributing and depositing on the wetted structure a polyolefin powder such as polyethylene or polypropylene, or powders of copolymers thereof, and thereafter melting the polyolefin powder onto the polyurethane structure by subjecting the water-wetted and polyolefin powder-covered polyurethane structure to a heating step such as insertion into an oven or a heating zone using infrared, radiant or convection heating, and repeating the wetting, dusting and heating steps if needed. By repeating the moistening step and the depositing step additional amounts of polyolefin powder may be deposited, depending on the pore size of the polyurethane structure and the mesh size of the polyolefin powder (hereinafter "mesh size" is U.S. Standard Mesh size unless otherwise stated).

As is well known, polyurethane foams can withstand considerably higher temperatures before decomposition (about 450°F.) than the polyolefins, although higher temperatures may be tolerated for brief periods. If the process is repeated the amount of polyolefin deposit on the foam can reach 700% and above depending on the pore size of the polyurethane foam and the mesh size of the polyolefin powder. This relationship will be further explained herein. The percentages stated herein with regard to the coatings are based on the weight of the uncoated form. Other percentages stated herein are on the basis of weight unless otherwise indicated.

As previously mentioned, products obtained according to the above process are generally of the same structural form as the reticulated polyurethane foam, and for that reason, the final product is dependent on the physical appearance of that foam. Generally, the reticulated polyurethane foam structure will have from 5 to 60 ppi (pores per lineal inch). From 5 ppi to 45 ppi represents the preferred range. Foams with 10 ppi to 25 ppi are the most preferred.

Polyurethane foams are generally made from a polymeric polyol resin and a polyisocyanate by reacting these components in the presence of water giving off blowing gases and/or a blowing agent activated by heat of reaction, while the polymer is reaching a more solid yet formable state. Most common foams are made from either a polyester or a polyether resin. Reticulated foams of the type mentioned above are available at Scott Paper Company, Foam Division, Eddystone, Pennsylvania.

Polyolefins useful in the novel process are made from precursors such as ethylene, propylene, 1-butene (and isomers thereof) or mixtures thereof as is well known in the art. If these polymers possess a melt temperature below that of the decomposition temperature of polyurethane they are acceptable for present purposes. In order to obtain certain advantageous properties such as load-bearing characteristics, the melting temperatures of the coating should not exceed about 450°F., again depending on the heat stability of the substrate. For present purposes, high temperatures (above 450°F.) are tolerable if short exposure times are used. In the present method, the high temperatures are not detrimental since short exposure times are encountered. However, preferably the temperature should be below 450°F., and more preferably below 400°F.

Polyolefin powders are available from various manufacturers, for example: polyethylene powders with mesh sizes of from 10 to 120, densities of from 0.912 to 0.950 and even higher (the densities stated herein are expressed in units of gms./c.c.) and melt indices of from 1 to 140 are available from U.S. Industrial Chemicals Co., New York, N.Y., polyethylene and ethylene copolymers are available from Dow Chemical Co., Midland, Mich., and from Union Carbide Plastics Co., New York, N.Y. From the last source various densities of polyethylene, including the highest, are available. Polypropylene powders or any other meltable powder made from polyethylene, polypropylene, their mixtures, polybutene, polypentene may be obtained by using custom-grinding facilities at Heisler Corporation, Wilmington, Delaware, and at Liquid Nitrogen Processing Corporation, Malvern, Pennsylvania. Other polyethylene powders of a mesh size of from 10 to 180 are available; similarly, high density polyethylenes which are preferred, such as those having densities of about 0.95 or preferably even higher may be obtained. In using the above listed polyolefins, it is important that their melting and flowing temperatures do not exceed the decomposition temperature of the particular polyurethane used.

The following data show the results of pressure-drop tests on an uncoated reticulated polyurethane foam having 10 pores per lineal inch and the same reticulated polyurethane foam coated with polyethylene.

These data show that the coated foam has a much higher resistance to air flow than the uncoated foam. The data are given in Table I. The pressure drop is measured in inches of water column. The flow rate is in cubic feet per minute per square foot of cross-section. The thickness of the foam is 1 inch. The temperature is 70°F. The uncoated foam has a pressure drop of 0.5 inches of water column at a flow rate of 100 cubic feet per minute per square foot. The coated foam has a pressure drop of 1.5 inches of water column at the same flow rate. This shows that the coating increases the resistance to air flow by a factor of 3.

TABLE I

## Pressure Drop Tests on Uncoated and Coated Reticulated Polyurethane Foam

| Sample                                          | Polyethylene Coating % based on Polyurethane | Pressure drop $\Delta P$ in inches of water at 350 ft./min. face velocity |
|-------------------------------------------------|----------------------------------------------|---------------------------------------------------------------------------|
| Control-uncoated polyurethane 10 ppi - 1" thick | 0                                            | 0.080                                                                     |
| Novel Structure 10 ppi - 1" thick               | 150                                          | 0.075                                                                     |

The above results indicate that the more rounded cross section of the coated strands offers less resistance to the flow of air than the concave triangular strands of the control sample. Generally, pressure drop increases very

little with the amount of polyolefin; structures having up to 300% polyethylene based on polyurethane will have only a slightly higher pressure drop.

TABLE II

## Pressure Drop Characteristics of Densified Polyethylene-Polyurethane Reticulated Foam, 24 ppi

Pressure drop  $\Delta P$  in inches of water at given face velocity

| Velocity Ft./Min. | 1/2" Thick Polyethylene Polyurethane Foam | Compressed Foam 1/2" Densified to 1/6" |
|-------------------|-------------------------------------------|----------------------------------------|
| 150               | .012                                      | .04                                    |
| 200               | .02                                       | .06                                    |
| 250               | .03                                       | .08                                    |
| 300               | .05                                       | .12                                    |
| 350               | .06                                       | .15                                    |
| 400               | .08                                       | .19                                    |
| 450               | .1                                        | .23                                    |
| 500               | .125                                      | .28                                    |
| 550               | .15                                       | .34                                    |
| 600               | .175                                      | .40                                    |

Other reticulated polyolefine-polyurethane foams densified to various degrees illustrate pressure drops of various kinds, thus illustrating an aspect of this invention—pressure drop control by means of densification of the present structure. After the polyolefin powder has been first sintered and then flowed over the polyurethane structure, the composite structure possesses as described above, a core of polyurethane and an intimately surrounding envelope of polyolefin. Strands of the novel

reticulated structures are in general elliptical in shape rather than concave as in polyurethane foam.

If it is desired to make an essentially polyolefin structure, the polyurethane core must be removed from the polyethylene. This step is carried out by simply soaking the foam in polyurethane hydrolyzing solution or by cutting the strands or nexae of the structure at appropriately spaced intervals according to the desired strength of the resulting product and

then keeping the structure in a strong hydrolyzing solution, e.g., strong acid or alkali solution, respectively having a pH less than 4 and more than 10 preferably at above ambient temperature. This action hydrolyzes and dissolves out the polyurethane structure; therefore any hydrolyzing agent suitable for this purpose is acceptable. Acids such as sulfuric acid, hydrochloric acid or phosphoric acid are preferred. Weaker acids require an unduly long time for treatment although they may be used to effect the same desired result. Strong alkali such as sodium hydroxide, potassium hydroxide and lithium hydroxide may be used instead of the acids. Similarly, the weak acid salts of the above alkalis may be used for the same purpose. The same applies for the weak alkali salts of strong acids. In all instances, the hydrolysis reaction is at the basis of the polyurethane removal, although in the case of strong sulfuric acid, other unknown factors, such as oxidation, enter into consideration.

If the polyurethane foam is improperly cured, or if it is of low index (less than 100, i.e., deficient in the stoichiometric amount of polyisocyanate) the resulting foam will be subject to the hydrolyzing action in a more efficient manner. After hydrolyzing and dissolving out the polyurethane, the pore structure of the final polyolefine product is about the same as that of the original reticulated polyurethane.

Similarly, if low density foams are used, these represent less mass subject to attack by strong hydrolyzing agents.

Generally, the polyester polyurethanes are the preferred starting materials in this process. However, the other polyol materials such as the polyethers are also acceptable if longer treating times can be tolerated.

Instead of cutting the polyurethane-polyolefin structure for easy introduction of the hydrolyzing agent, polyolefin powder may be brought up to a temperature making it flow and so as to leave exposed a portion of the strands or nexae of the polyurethane structure. By using this technique some of the surfaces of the polyurethane will be exposed to the hydrolytic action when appropriately treated. Although this structure does not represent another embodiment, it is not as strong due to the tendency of the polyethylene to split into individual cells; however, this structure has considerably greater surface area and, for that reason, is desirable in special applications.

A cut polyurethane structure will have ex-

posed edges (if not subsequently treated to cover up the polyurethane). If this structure is treated for a sufficient time in an extremely strong hydrolyzing agent such as a sulfuric acid, and if the duration of treatment is not important, then the same removal of polyurethane may be achieved. This approach is generally applicable to smaller pieces. If contamination of the filtered stream is unimportant, the whole structure may be used and in time the polyolefin structure will result.

If polyolefin powder is admixed with the polyurethane-forming starting material, after hydrolyzing, the final polyolefin structure will have a considerably smaller core. Generally, the products with hollow cores may be used as such. However, if the hollow core is undesirable, the novel structure may be treated by explosion-glazing techniques by subjecting the structure to an explosive flame front. Care should then be exercised to protect the structure from melting or burning.

The following examples and tables represent the novel method of making the novel structures. In these tables the various relationships of the process and product variable have been depicted appropriately illustrating the various aspects of the invention.

#### EXAMPLE 1

A sample of 10 ppi Scott Industrial Foam, 1" thick and 6" square, (a reticulated polyurethane foam) was coated with powdered polyethylene ("Microthene" [Registered Trade Mark] 710, U.S. Industrial Chemicals Company, 50 mesh, melt index 22, 0.916 density) by first dampening with water, then dusting with powdered polyethylene and finally drying and fusing the sample in an oven for 10 minutes at 180°C. The polyethylene coating amounted to 229% of the polyurethane weight. In a similar manner, coatings of 190%, 269% and 310% of polyethylene were obtained.

#### EXAMPLE 2

Polyethylene-coated 10 ppi polyester reticulated polyurethane foam (the ester used is a polyethylene glycol adipate) was tested for chemical resistance by varying the mesh size of polyethylene powder (22 melt index, density .916) from 20 to 140. For 140 mesh size, the melt index was 5 and density 0.924. The polymeric structure was formed by repeating the steps twice and the results are represented in the table below.

TABLE III

Effect of Particle Size on Chemical Resistance

| Mesh Size | Resin Applied<br>% of PU | % of Polyurethane Lost, 2 hrs. at 140°F. |                                    |          |
|-----------|--------------------------|------------------------------------------|------------------------------------|----------|
|           |                          | 21% HCl                                  | 70% H <sub>2</sub> SO <sub>4</sub> | 10% NaOH |
| 20        | 406                      | 100                                      | 100                                | 39       |
| 35        | 343                      | 42                                       | 93                                 | 21       |
| 50        | 281                      | 28                                       | 49                                 | 5.4      |
| 80        | 210                      | 2.5                                      | 83                                 | 0        |
| 140       | 180                      | 41                                       | 72                                 | 2.8      |

5 The data indicate that despite the heavier coating of the coarser resin, maximum chemical resistance with this type of polyethylenes is obtained with 50 and 80 mesh size polyethylene powders. Further tests have indicated that varying the melt index of the polyethylene has little influence on chemical resistance. Despite the above-demonstrated type of chemical resistance to acids, these structures may still be useful for other purposes such as shaping, contouring.

## EXAMPLE 3

15 Resin pickup versus chemical resistance was studied with a mixture of 25% polyethylene powder of 140 mesh and a melt index of 5 and 75% polyethylene powder of 50 mesh and a melt index of 22. The reticulated polyurethane foam coated was of the same characteristics as in Example 2. The results set out in the table below were obtained by immersing a cut specimen in the acid. Better results than shown are obtained when the cut edges are further treated to seal off the polyurethane core from the acid attack.

TABLE IV

Effect of Resin Pick-up on Chemical Resistance

| Resin Applied (Pickup) | % of Polyurethane Lost<br>21% HCl, 2 hrs. at 140°F. |
|------------------------|-----------------------------------------------------|
| 174                    | 47                                                  |
| 265                    | 19                                                  |
| 295                    | 16                                                  |
| 445                    | 2.5                                                 |

30 It has been noted that 140 mesh resin always gives a complete coating with no "dry" spots visible under microscopic examination. However, the coating is thinner than with coarser polyethylene particles. Use of a mixture of 25% of 140 mesh with 75% of a coarser mesh resin gives not only a better coverage, but also improves the resin pickup.

## EXAMPLE 4

35 Foam of the same characteristics as identified in Example 2 has been coated with polyethylene varying the mesh size of the polyethylene and water pickup of polyurethane. The polyethylene has been identified in Example 3 above.

TABLE V

Effect of Water Pick-up and Particle Size on Resin Pick-up

| Mesh Size<br>U.S. Std. | Water Pickup<br>as % of<br>Polyurethane | Resin Pickup<br>as % of<br>Polyurethane |
|------------------------|-----------------------------------------|-----------------------------------------|
| 20                     | 33                                      | 200                                     |
| 20                     | 58                                      | 265                                     |
| 20                     | 82                                      | 304                                     |
| 20                     | 107                                     | 285                                     |
| 35                     | 25                                      | 195                                     |
| 35                     | 50                                      | 254                                     |
| 35                     | 75                                      | 273                                     |
| 35                     | 100                                     | 287                                     |
| 50                     | 25                                      | 186                                     |
| 50                     | 50                                      | 223                                     |
| 50                     | 75                                      | 221                                     |
| 50                     | 100                                     | 250                                     |
| 80                     | 25                                      | 119                                     |
| 80                     | 50                                      | 136                                     |
| 80                     | 75                                      | 156                                     |
| 80                     | 100                                     | 197                                     |
| 140                    | 25                                      | 77                                      |
| 140                    | 50                                      | 105                                     |
| 140                    | 75                                      | 94                                      |
| 140                    | 100                                     | 94                                      |

The above data indicate that resin pickup increases as particle size increases (as mesh size decreases). However, it should be noted that the use of large particle sizes such as 20 mesh gives poorer chemical resistance even at high resin pickup, while the physical properties such as compressive stress-strain is considerably better. Other foams coated according to the above techniques have been of smaller pore sizes such as about 25 ppi foam, displaying a 217% polyethylene pickup. The starting material was an 80 mesh, 22 melt index, 916 density polyethylene. Similarly, a 10 ppi foam was fuse-coated with 9 melt index, 950 density polyethylene giving 150% pickup.

## EXAMPLE 5

An explosion-reticulated polyurethane foam produced by explosively decomposing a mixture of acetylene and oxygen produces a polyurethane foam surface having a glassy appearance. This reticulated polyurethane foam

surface, when coated with polyethylene, has no interaction with the polyethylene. Stretching of the coated foam results in loosening of the coating, and where the coating is light, it is easily removed.

Physical properties of the novel composites are unexpectedly different from the polyurethane material. For example, the strands of the novel reticulated network structure are elliptical in shape and for the 10 ppi structure of 267% resin structure, a representative average of major and minor axis diameters is about  $5.1 \times 10^{-2}$  cm.  $4.7 \times 10^{-2}$  cm. respectively. On the other hand, the cross sections of polyurethane strands are about equilaterally concavely formed triangles.

Stiffness of the novel structure related to the per cent resin pickup is a straight line function. A 10 ppi adipate polyester polyurethane foam coated with polyethylene (22 melt index, 50 mesh, 916 density) has the following stiffness properties.



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TABLE VI

## Stiffness Factor of Polyethylene-Polyurethane Composite

| Resin Pickup | Stiffness Factor -Grams* |
|--------------|--------------------------|
| 0%           | 20                       |
| 100%         | 90                       |
| 200%         | 140                      |
| 300%         | 200                      |
| 370%         | 240                      |

\* The stiffness factor is the weight required to bend a 1" square section of polyfoam, which projects 7.5" over the edge of a 6" high block, to the point where it forms a

53° angle with the horizontal. All the weights are placed on the upper surface of the foam and within 1" of the unsupported end.

TABLE VII

Effect of Resin Pickup in the 190% to 310% Range on the Physical Properties of the Novel Polyethylene-Polyurethane Composites at 74°F.  
(Polyethylene starting material is 22 melt index, 50 mesh, .916 density)

|                                                                                                          | Adipate Polyester Polyurethane Foam 10 ppi | Polyethylene on Reticulated 10 ppi Polyurethane |            |            |
|----------------------------------------------------------------------------------------------------------|--------------------------------------------|-------------------------------------------------|------------|------------|
|                                                                                                          |                                            | 190%                                            | 269%       | 310%       |
| Tensile strength, p.s.i.                                                                                 | 14.9 ± 30%                                 | 33.4                                            | 35.4 ± 29% | 36.2 ± 5%  |
| Youngs modulus in tension, p.s.i.                                                                        | 7.2 ± 25%                                  | 117                                             | 125 ± 24%  | 167 ± 9%   |
| Per cent elongation                                                                                      | 228 ± 28%                                  | 98                                              | 120 ± 31%  | 96 ± 12%   |
| Tear strength, lb./in.                                                                                   | 5.3 ± 28%                                  | 13.6                                            | 14.4 ± 24% | 14.3 ± 15% |
| Compression set at 158°F. per cent                                                                       | 19.2                                       | 29.2                                            | 37.3 ± 10% | 27.3 ± 3%  |
| Compressive energy absorption at 0.8"/" strain,** p.s.i. calculated from compressive stress strain curve | .17                                        | 6.7                                             | 11.7       | 17.3       |
| Shock absorbency *                                                                                       | 1                                          | 5.5                                             | 10         | 10+        |

\* The shock absorbing capacity of the foam is measured by the height required for a 4-pound ball to fall, in order that the shock transmitted through a 1" slab of foam is just sufficient to break 1/16" thick glass microscope slides placed beneath the foam. The ball is 3 1/2" O.D., the outer shell of which is a 30-gram polyethylene ball made of 3 melt index, .993 g/cc density, 50-mesh resin. The ball is then filled to a final weight of 4 pounds with Wood's alloy. The backup for the glass plates is vinyl tile cemented to reinforced concrete. The glass slides are supplied by Arthur H. Thomas Company, Philadelphia, Pennsylvania, Code No. 7030 Special Red Label Micro Slides, and are 3" x 1".

\*\* The term "0.8"/" refers to compression of the foam to 20% of its original thickness, i.e. a compression of 0.8 inch per each inch of the original thickness

TABLE VIII

Effect of Resin Pickup on the Compressive Stress-Strain Characteristics of 1" Thick 10 ppi Polyethylene-Polyurethane Composites at 74°F.  
(Polyethylene starting material is 22 melt index, 50 mesh, .916 density)

| % Resin Pickup<br>Based on Polyurethane | Applied Stress - psi |           |           |
|-----------------------------------------|----------------------|-----------|-----------|
|                                         | 25% Defl.            | 50% Defl. | 75% Defl. |
| 0                                       | .4                   | .6        | 1.4       |
| 100                                     | 2.0                  | 3.0       | 7.5       |
| 200                                     | 4.6                  | 7.0       | 16.5      |
| 300                                     | 9.0                  | 13.8      | 34.0      |

TABLE IX

Compressive Stress-Strain Characteristics of 1" Thick 10 ppi Polyethylene-Polyurethane Composite

(Polyethylene starting material is 22 melt index, 50 mesh, .916 density)

| Strain<br>in./in. | Applied Stress - psi  |                         |                         |                         |
|-------------------|-----------------------|-------------------------|-------------------------|-------------------------|
|                   | 0% Polyeth.<br>Pickup | 190% Polyeth.<br>Pickup | 267% Polyeth.<br>Pickup | 310% Polyeth.<br>Pickup |
| .1                | .4                    | 3.9                     | 6.0                     | 7.8                     |
| .2                | .4                    | 4.1                     | 6.6                     | 8.6                     |
| .3                | .42                   | 4.5                     | 7.5                     | 9.8                     |
| .4                | .45                   | 5.2                     | 9.0                     | 12.0                    |
| .5                | .5                    | 6.2                     | 11.0                    | 14.0                    |
| .6                | .6                    | 8.0                     | 15.0                    | 18.0                    |
| .7                | .8                    | 11.5                    | 21.0                    | 30.0                    |
| .75               | —                     | —                       | —                       | 44.0                    |
| .8                | 1.52                  | 20.0                    | 45.0                    | —                       |

The tensile strength of 267% polyethylene and 10 ppi polyester-polyurethane-polyethylene composites falls off approximately 2 psi for every 10-degree rise in temperature in the 74°F. to 180°F. range.

Under the load required for 25% deflection at ambient temperature, 10 ppi polyester-polyurethane polyethylene composites of 170%, 267% and 310% polyethylene were found to be serviceable up to approximately 210°F. The same composites also could withstand 30 minutes in boiling water while stacked to a height of 4 feet with less than 15% loss in

thickness of the bottom one-inch thick section. Data given below indicate that the composites possess higher slump temperatures than polyethylene. A 10 ppi reticulated polyester-polyurethane foam with 332% coating of 3 melt index and .918 density polyethylene was compared with the same foam having 70% of the polyurethane leached out. Two-inch square by one-inch thick samples of both types of structures were compression-loaded with 5 lbs./sq. in. and inserted in an oven at 200°F. for 15 minutes. The comparative results were as follows: the deflection at ambient temperature

was 25%, and 53.5% after heating for the composite structure. For the leached-out "polyethylene" structure, the deflection at ambient temperature was 52% and after heating 68%.

5 The above indicates that the composite structure is mutually reinforcing; as, at that loading level, uncoated, reticulated polyurethane foam structures would have easily collapsed. Similarly, the higher load-bearing ability at high temperatures illustrates the cooperating nature of the two materials.

All physical properties, except as otherwise indicated, were evaluated using ASTM Standards D1564—59T.

#### 15 EXAMPLE 6

A sample of 10-ppi Scott Industrial Foam 1" thick and 6" square (a reticulated polyurethane foam) was coated with powdered polyethylene ("Microthene" 710, U.S.I. Chemical Company, 50 mesh, melt index 22, .916 density) by first dampening with water, then dusting with powdered polyethylene and finally drying and fusing the sample in an oven for 10 minutes at 180°C. The coating amounted to 229% of the polyurethane weight. (The relatively short oven time of 10 minutes was used to insure that the molten resin did not completely cover the substrate, thus making possible subsequent acid attack.) The conditions governing the application of the powder may be varied to obtain the desired type of coating. In a similar manner polyethylene having a density of .950 was used in the process. Polypropylene powder is suitable in place of polyethylene.

#### EXAMPLE 7

The coated, reticulated polyurethane foam sample prepared in Example 6 was immersed in 21% hydrochloric acid for 5½ hours at 140°F followed by immersion for an additional 64½ hours at ambient temperatures after which the sample was washed and dried. As a result of this treatment, all polyurethane was converted to other products. The polyethylene coating appeared unaffected by the acid treatment. The above foam was also treated by an explosive mixture of oxygen and acetylene. A change in the properties was obtained such as melting of the strands and nexae.

#### 50 EXAMPLE 8

Polyester polyurethane foam (the ester used is a polyethylene glycol adipate) previously obtained in reticulated form from Scott Paper Co., Foam Division, Eddystone, Pa. and cut to 84 × 22 × 1 inch sizes was wetted out in a dip tank, passed through a set of squeeze rolls with the pick-up of water adjusted to the 45% level based on polyurethane. Throughout the wetted foam sheet were dispersed 22 melt index, .916 density, 50 mesh polyethylene particles and the sheet heated for 15 minutes at 375°F. The process was repeated.

In a similar manner using the same wetted

foam, 3 melt index, .918 density, 35 mesh polyethylene particles were fused on the polyurethane structure. Changing the melt index from 22 to 3 requires an additional fusing time if the powder is applied in two applications.

Appropriate test specimens were cut from the foam and tested according to ASTM Standards D1564—59T, with the data set out and discussed below.

The effect of increasing the melting point of the fused polyethylene as shown in the tables below illustrates the drastic increase in the applied stress required when compared with polyurethane, low-density polyethylene-coated reticulated polyurethane and low-density polyethylene structure.

The increase demonstrates highly desirable properties are obtained in the novel structures even after about 95% of the polyurethane has been removed.

TABLE X  
Compressive Stress-Strain Relationship of 10 ppi (Pores per Lineal Inch) Polyurethane Polyester Reticulated Foam

| Applied Stress—psi | Strain—in/in |
|--------------------|--------------|
| .4                 | .1           |
| .405               | .2           |
| .42                | .3           |
| .46                | .4           |
| .525               | .5           |
| .62                | .6           |
| .86                | .7           |
| 1.5                | .8           |

TABLE XI  
Compressive Stress-Strain Relationship of 10 ppi Reticulated Polyurethane Polyester Foam Fuse-Coated with 300% (Based on Polyurethane) 22 Melt Index .918 Density Polyethylene (No Acid Leaching)

| Applied Stress—psi | Strain—in/in |
|--------------------|--------------|
| 6.6                | .1           |
| 6.8                | .2           |
| 7.0                | .3           |
| 7.8                | .4           |
| 9.0                | .5           |
| 12.5               | .6           |
| 24.0               | .7           |
| 50.0               | .8           |

TABLE XII  
Compressive Stress-Strain Relationship of 10 ppi Reticulated Polyurethane Polyester Foam Fuse-Coated with 331% (Based on Polyurethane) 3 Melt Index .918 Density Polyethylene (No Acid Leaching)

| Applied Stress—psi | Strain—in/in |
|--------------------|--------------|
| 9.6                | .1           |
| 12.0               | .2           |
| 13.5               | .3           |
| 15.5               | .4           |
| 19.0               | .5           |
| 26.0               | .6           |
| 40.0               | .7           |
| —                  | .8           |

TABLE XIII

Compressive Stress-Strain Relationship of 10 ppi Reticulated 22 Melt Index, .916 Density Polyethylene Structure Prepared by Fusing 300% of Above Polyethylene on 10 ppi Reticulated Polyurethane Polyester Foam and Leaching Out with HCl About 95% of the Polyurethane.

|    | Applied Stress—psi | Strain—in/in |
|----|--------------------|--------------|
| 10 | 2.1                | .1           |
|    | 2.8                | .2           |
| 75 | 3.0                | .3           |
|    | 3.6                | .4           |
|    | 4.6                | .5           |
| 15 | 6.8                | .6           |
|    | 13.0               | .7           |
| 80 | 44.0               | .8           |

TABLE XIV

Compressive Stress-Strain Relationship of 20 ppi Reticulated 3 Melt Index, .918 Density Polyethylene Structure Prepared by Fusing 331% of Above Polyethylene on 10 ppi Reticulated Polyurethane Polyester Foam and Leaching Out with HCl About 95% of the Polyurethane.

|    | Applied Stress—psi | Strain—in/in |
|----|--------------------|--------------|
| 25 | 2.7                | .1           |
|    | 4.6                | .2           |
|    | 6.4                | .3           |
| 30 | 7.8                | .4           |
|    | 10.5               | .5           |
|    | 15.0               | .6           |
|    | 25.0               | .7           |
|    | 60.0               | .8           |

35 The above data indicate that the polyethylene structure (about 95% polyurethane removed) has excellent structural properties when compared (1) with the polyethylene-coated polyurethane and (2) when compared with the lower-density polyethylene structure (95% polyurethane removed). The uncoated reticulated polyurethane foam stress-strain data show considerably less rigid structure.

45 As illustrated above the polyolefin structures are self-supporting reticulated structures especially suitable for acid or alkali filtering or demisting operations.

50 The high-density polyethylenes such as densities of from about .950 and higher and polypropylenes give a more rigid and stronger structure while the lower-density polyethylene structures are weaker.

55 The stronger structures as characterized by polyethylene having a density of from .935 to .970 are preferred.

60 Polypropylene structures are also very suitable due to their rigidity which is generally higher than that of polyethylene. However, it must have a high-temperature stabilizer or anti-oxidant.

65 Additionally, a polyurethane reticulated foam structure may first be treated with a first polyolefin powder having a melting point at least

30°F above that of polyethylene, then treated to remove the polyurethane, wetted and then recoated with a second polyolefine powder having a melting the same as, or lower than, polyethylene. However, the margin between the melting of the second polyolefine and the collapse of the structure is considerably narrower than with polyolefin-polyurethane and, therefore, close temperature control is important.

#### WHAT WE CLAIM IS:—

1. A process of producing reticulated polymeric structures comprising wetting reticulated polyurethane foam with a liquid, distributing throughout said reticulated polyurethane foam polyolefin resin powder and melting in a heating zone said polyolefin powder at a temperature below the decomposition point of the polyurethane and polyolefin to conform the molten polyolefin to the outlines of said reticulated polyurethane foam, and, if desired, repeating the above steps.

2. A process according to claim 1, in which the reticulated polyurethane foam has from 5 to 60 pores per lineal inch and the polyolefin powder comprises a mixture of different-sized particles having a U.S. mesh size from 10 to 180.

3. A process according to claim 1 or 2, wherein the polyolefin is polyethylene.

4. A process according to any one of the preceding claims wherein the reticulated polyurethane foam has 10 pores per lineal inch and the olefin powder mixture consists of polyethylene having the U.S. mesh size from 20 to 140.

5. A process according to any one of claims 1 to 3, wherein the reticulated polyurethane foam is a polyester polyurethane of 10 pores per lineal inch, the polyolefin powder mixture consists of polyethylene having the U.S. mesh size from 50 to 80 and the polymeric structure is formed by repeating the steps twice.

6. A process for producing a reticulated polyolefin structure from reticulated polyurethane foam covered with melt-formed coating of polyolefin as claimed in any one of claims 1 to 5, further comprising treating the polyurethane component of the reticulated composite structure with a polyurethane hydrolyzing solution to dissolve the polyurethane component.

7. A process according to claim 6, wherein the polyolefin melt-formed coating is formed from a first polyolefin powder having a melting point at least 30°F above that of polyethylene and wherein, after the hydrolyzing solution treatment, the reticulated polyolefin structure is wetted, a second polyolefin powder having a melting point the same as polyethylene or lower than polyethylene is distributed throughout the polyolefin structure and melted over the reticulated polyolefin structure.

8. A process according to claim 7, wherein the first polyolefin is polypropylene and the



TABLE XIII

Compressive Stress-Strain Relationship of 10 ppi Reticulated 22 Melt Index, .916 Density Polyethylene Structure Prepared by Fusing 300% of Above Polyethylene on 10 ppi Reticulated Polyurethane Polyester Foam and Leaching Out with HCl About 95% of the Polyurethane.

|    | Applied Stress—psi | Strain—in/in |
|----|--------------------|--------------|
| 10 | 2.1                | .1           |
|    | 2.8                | .2           |
| 15 | 3.0                | .3           |
|    | 3.6                | .4           |
|    | 4.6                | .5           |
|    | 6.8                | .6           |
|    | 13.0               | .7           |
|    | 44.0               | .8           |

TABLE XIV

Compressive Stress-Strain Relationship of 20 10 ppi Reticulated 3 Melt Index, .918 Density Polyethylene Structure Prepared by Fusing 331% of Above Polyethylene on 10 ppi Reticulated Polyurethane Polyester Foam and Leaching Out with HCl About 95% of the Polyurethane.

|    | Applied Stress—psi | Strain—in/in |
|----|--------------------|--------------|
| 25 | 2.7                | .1           |
|    | 4.6                | .2           |
|    | 6.4                | .3           |
| 30 | 7.8                | .4           |
|    | 10.5               | .5           |
|    | 15.0               | .6           |
|    | 25.0               | .7           |
|    | 60.0               | .8           |

35 The above data indicate that the polyethylene structure (about 95% polyurethane removed) has excellent structural properties when compared (1) with the polyethylene-coated polyurethane and (2) when compared with the lower-density polyethylene structure (95% polyurethane removed). The uncoated reticulated polyurethane foam stress-strain data show considerably less rigid structure.

45 As illustrated above the polyolefin structures are self-supporting reticulated structures especially suitable for acid or alkali filtering or demisting operations.

50 The high-density polyethylenes such as densities of from about .950 and higher and polypropylenes give a more rigid and stronger structure while the lower-density polyethylene structures are weaker.

55 The stronger structures as characterized by polyethylene having a density of from .935 to .970 are preferred.

60 Polypropylene structures are also very suitable due to their rigidity which is generally higher than that of polyethylene. However, it must have a high-temperature stabilizer or anti-oxidant.

Additionally, a polyurethane reticulated foam structure may first be treated with a first polyolefin powder having a melting point at least

30°F above that of polyethylene, then treated to remove the polyurethane, wetted and then recoated with a second polyolefine powder having a melting the same as, or lower than, polyethylene. However, the margin between the melting of the second polyolefine and the collapse of the structure is considerably narrower than with polyolefin-polyurethane and, therefore, close temperature control is important.

#### WHAT WE CLAIM IS:—

1. A process of producing reticulated polymeric structures comprising wetting reticulated polyurethane foam with a liquid, distributing throughout said reticulated polyurethane foam polyolefin resin powder and melting in a heating zone said polyolefin powder at a temperature below the decomposition point of the polyurethane and polyolefin to conform the molten polyolefin to the outlines of said reticulated polyurethane foam, and, if desired, repeating the above steps.

2. A process according to claim 1, in which the reticulated polyurethane foam has from 5 to 60 pores per lineal inch and the polyolefin powder comprises a mixture of different-sized particles having a U.S. mesh size from 10 to 180.

3. A process according to claim 1 or 2, wherein the polyolefin is polyethylene.

4. A process according to any one of the preceding claims wherein the reticulated polyurethane foam has 10 pores per lineal inch and the olefin powder mixture consists of polyethylene having the U.S. mesh size from 20 to 140.

5. A process according to any one of claims 1 to 3, wherein the reticulated polyurethane foam is a polyester polyurethane of 10 pores per lineal inch, the polyolefin powder mixture consists of polyethylene having the U.S. mesh size from 50 to 80 and the polymeric structure is formed by repeating the steps twice.

6. A process for producing a reticulated polyolefin structure from reticulated polyurethane foam covered with melt-formed coating of polyolefin as claimed in any one of claims 1 to 5, further comprising treating the polyurethane component of the reticulated composite structure with a polyurethane hydrolyzing solution to dissolve the polyurethane component.

7. A process according to claim 6, wherein the polyolefin melt-formed coating is formed from a first polyolefin powder having a melting point at least 30°F above that of polyethylene and wherein, after the hydrolyzing solution treatment, the reticulated polyolefin structure is wetted, a second polyolefin powder having a melting point the same as polyethylene or lower than polyethylene is distributed throughout the polyolefin structure and melted over the reticulated polyolefin structure.

8. A process according to claim 7, wherein the first polyolefin is polypropylene and the

second polyolefin is polyethylene.

9. A process according to any one of claims 6 to 8, wherein the reticulated composite structure treated by said polyurethane hydrolyzing solution has from 5 to 60 pores per lineal inch, and preferably from 5 to 45 pores per lineal inch.

10. A polyolefin-covered reticulated polyurethane composite structure as prepared by a process in accordance with any one of the preceding claims having strands of elliptical cross sections, and nexae joined to said strands at spaced-apart points forming a continuous body, said polyolefin material being present from at least about 100% based on the weight of polyurethane foam.

11. A composite structure according to claim 10, having a pore size of from 5 pores per lineal inch to 60 pores per lineal inch, and preferably from 5 pores per lineal inch to 45 pores per lineal inch.

12. A composite structure according to claim 10 or 11, wherein the polyolefin is polyethylene or polypropylene.

13. A composite structure according to any one of claims 10 to 12, wherein the polyurethane is polyester polyurethane.

14. A reticulated, substantially membraneless polyolefin structure having of from 5 to 60 pores per lineal inch, and preferably from 5 to 45 pores per lineal inch.

15. A polyolefin structure according to claim 14, wherein the polyolefin is a copolymer of polypropylene and polyethylene.

16. A process of producing reticulated polymeric structures as claimed in any one of claims 1 to 9 substantially as hereinbefore described.

17. Reticulated polymeric structures whenever produced by the process of any one of claims 1 to 9 and 16.

18. Reticulated polymeric structures as claimed in any one of claims 10-15 and 17 substantially as hereinbefore described.

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