

[54] **CARPET WITH MICROCAPSULES
CONTAINING VOLATILE
FLAME-RETARDANT**

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[56] **References Cited**

UNITED STATES PATENTS

3,418,656 12/1968 Vassiliades..... 252/316

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[57]

ABSTRACT

A flame-resistant, tufted carpet is prepared by providing the primary backing support for the tufted yarn with microcapsules containing a volatile flame-retardant agent. The microcapsules are heat activatable so as to release the flame-retarding agent under the action of heat and form a flame-resistant atmosphere that surrounds the protruding tufts. The microcapsules may be coated onto either side of the primary backing support so as to penetrate said support and be positioned closely adjacent the yarn tufts. Additionally, the microcapsules may be incorporated into an adhesive layer which is superposed over the side of the primary backing support opposite the protruding yarn tufts to give further flame resistance to the carpet.

20 Claims, No Drawings

CARPET WITH MICROCAPSULES CONTAINING VOLATILE FLAME-RETARDANT

This invention relates to flame-resistant, tufted products. More particularly, this invention relates to flame-resistant, tufted carpet which incorporates microencapsulated flame-retarding agents.

Various proposals have been made in an attempt to improve the flame resistance of building materials and home furnishings, such as furniture and carpet. For example, it has been suggested that the flame resistance of polyester carpet can be increased by employing flame-resistant backing adhesives which are coated over the primary backing so as to penetrate the primary backing and cover the bottom of the tuft rows.

Thus, in an article entitled "Flame-Resistant Backing Adhesive for Polyester Carpets", by Barton et al. in the January, 1972 issue of "Textile Chemist and Colorist," Vol. 4, No. 1, pages 55-61, it is indicated on page 59 of the article that the flame-resistant backing adhesives should be placed so that the adhesive penetrates the primary backing fabric and the base of the yarn tuft, while filling the interstices of the fabric completely until the adhesive barely emerges onto the fabric surface. It is further indicated that overpenetration of the adhesive should be avoided because objectionable adhesive deposits will result on the upper side of the primary backing. Although such approach is useful in rendering the primary backing fabric flame resistant, the highly flammable yarn tufts are still exposed to flame conditions, and in fact, usually come into initial contact with the flame source, e.g. a dropped cigarette, match or the like.

It has now been found that a flame-resistant tufted product can be provided wherein the yarn tufts, as well as the backing are rendered flame resistant. The tufted product comprises a primary backing substrate having a first side and a second side, and yarn tufted to the first side of the primary backing substrate and projecting through the primary backing substrate to form a pile on the first side. An adhesive layer is superposed over the second side of said primary backing substrate. The primary backing substrate bears microcapsules containing a volatile or volatilizable flame-retarding agent, and the flame-retarding agent is released from the microcapsules under the action of heat to volatilize and form a flame-resistant, gaseous atmosphere surrounding each of the yarn tufts.

Thus, the flame-resistant microcapsules of the present invention provide more than an essentially localized flame resistance to the carpet backing. By virtue of the volatile nature of the encapsulated flame retardant, a flame-resistant, gaseous or vaporous atmosphere surrounds the yarn tufts, thereby providing flame resistance to the entire carpet. Such protection is particularly critical in the case of shag-style carpets, for example, where a high degree of flame resistance is necessary for the long, protruding tufts.

The microcapsules containing the flame-retarding agent may be incorporated into the carpet in any suitable manner. According to one embodiment of the present invention, the capsules are coated onto the primary backing fabric, such as jute, by spraying the microcapsular dispersion or using a coating blade. The microcapsular dispersion penetrates into the jute, and the primary backing is thereafter dried at a temperature between about 100°F. and about 200°F., preferably

bly between about 150°F. and about 200°F. The jute web may then be tufted with a suitable yarn, such as polyester, nylon or polyolefin yarn, to form the carpet. The microcapsular dispersion may be applied to either side of the primary backing fabric. Regardless of the method utilized for incorporating the microcapsules into the primary backing fabric, it is preferred to fill the interstices of the backing fabric completely with the capsules and surround the base of each yarn tuft as completely as possible. This will place the microcapsules as close to the flame sites as possible. Then, under the action of the heat of the fire to be extinguished, the flame-retarding agent will be released and completely surround each yarn tuft with a high density atmosphere of the flame-retarding vapor, thereby rendering the yarn tufts flame resistant.

The use of microencapsulated flame-retardant agents offers decided advantages over the mere incorporation of unencapsulated flame retardants into the backing adhesives, for example. By encapsulating a normally volatile and/or toxic flame-retarding agent, it is surrounded by substantially impermeable film which thereby prevents migration and volatilization of the flame-retardant material until it is needed, i.e., when the carpet is attacked by fire and the capsule walls rupture. Thus, if one were to incorporate unencapsulated tetrabromoethane into the backing adhesive, this volatile and toxic material would evaporate from the adhesive and not be available for protection of the carpet. Additionally, the use of microencapsulated volatile flame retardants, such as tetrabromoethane, has the advantage of spreading rapidly to surround the yarn tufts and thereby provide widespread protection for the carpet, whereas, the action of non-volatile flame-retardant agents is essentially local.

According to another embodiment of the present invention, an adhesive layer, such as a clay filled carboxylated SBR latex, containing flame-retardant microcapsules is superposed over the side of the primary backing support opposite the yarn tufts. The presence of microcapsules in this adhesive layer aids in rendering the overall carpet product flame resistant.

Any suitable fire retardant may be employed in the microcapsules of the present invention whether in liquid or solid form. Thus, the liquid fire-retardant material or solution of such fire-retardant material may be any one or a mixture of several classes of fire-retardant chemical compounds such as phosphate esters, halogenated phosphate esters, phosphonate esters, halogenated phosphonate esters, phosphites, halogenated paraffins, halogenated olefins, halogenated aromatics and other halogenated organic compounds generally recognized as having fire-retardant properties.

Specific examples of such compounds include tris-(2,3-dibromopropyl)phosphate, tris-(2,3-dichloropropyl) phosphate, tris-(2-chloroethyl)phosphate, bis-(2-chloroethyl)-2-chloroethyl phosphonate, mixed halogenated alkyl acid phosphates, tricresyl phosphate, cresyldiphenyl phosphate, diethyl-N,N-bis-(2-hydroxy-ethyl)aminomethyl phosphonate, triphenyl phosphite, alkylaryl phosphites, alkyl phosphites, or the like. Other halogenated, organic compounds include ethylene dibromide, tetrabromoethane, tetrabromobutane, carbon tetrabromide, hexachloroethane, hexabromocyclododecane, perchlorobicyclo [2.2.1] heptane and other chlorinated cage compounds. Halogenated olefins such as tetrachloroethylene, hexa-

chlorobutadiene, trichloroethylene, halogenated aromatics such as tribromobenzene, tribromophenol, pentabromophenol, hexabromobenzene, brominated bisphenol A and other organic compounds generally recognized as having fire-retardant properties may be suitably employed in the present invention.

Preferably, the flame-retardant material is a volatile material, such as tetrabromoethane, tetrabromobutane, dichlorodibromomethane, carbon tetrabromide, or mixtures thereof with other halogenated hydrocarbons. Obviously, the volatile flame-retarding agents are preferred, since such materials will envelop the yarn tufts and render them flame resistant, whereas less volatile flame-retarding agents will give a more localized protection.

The flame-retarding agent may be incorporated into the microcapsules by any suitable means. For example, the flame-retardant material may be incorporated into the microcapsules by forming such material into emulsion droplets by addition to an aqueous solution of a wall-forming emulsifying agent. Any suitable process for forming microcapsules may be employed including that described in U.S. Pat. No. 3,418,656 to A. E. Vasiliades wherein film-forming encapsulating agents, such as poly (vinyl alcohol), gelatin, various proteins, partially condensed formaldehyde condensation products, natural synthetic gums, polystyrene, polyvinyl chloride, and ethylcellulose are employed in the formation of the capsule walls.

Subsequent to the formation of the microcapsules, the microcapsular dispersion can be applied to the primary backing support from an aqueous slurry directly as a flame-retardant coating, or the water may be removed from the aqueous capsule slurry by filtration, centrifugation, spray-drying or by any other drying process to yield a dry powder. This results in a dry powder consisting of microcapsules having the flame-retardant core surrounded by an impermeable membrane. The dry powder can then be dispersed in either an aqueous or non-aqueous system and then applied to the primary support.

The microcapsules of the present invention may be provided in any suitable size. However, preferably the microcapsules have an average diameter between about 3 and about 30 microns, more preferably between about 3 and about 10 microns. Larger capsules are less preferred as they are more susceptible to rupture under the pressures encountered in the handling and use of the carpet, while the smaller diameter microcapsules are preferred because of their physical strength, and because of their ability to penetrate the fibrous primary backing support.

Any suitable coat weight of microcapsules may be employed for application to the primary backing support. For example, between about 0.1 and about 1.0 pounds, preferably between about 0.1 and about 0.3 pounds, of microcapsule solids per square yard of the primary backing support, such as jute, may be utilized. One-hundred grams of microcapsules generally contain between about 70 and about 80 grams of flame-retardant material.

As previously by the microencapsulated flame retardants may be incorporated into the adhesive layer, which may be a latex formed from SBR, vinyl chloride, vinyl chloride-vinylidene chloride, nitrile rubber, or the like. Such incorporation may be accomplished by adding between about 5 and about 50 parts, preferably be-

tween about 10 and about 30 parts by weight of capsules per 100 parts of latex-forming solids prior to coating of the secondary support layer over the primary support.

The invention will be more particularly illustrated by the following examples. The percentages are by weight unless otherwise specified.

EXAMPLE 1

Six grams of a 50 percent solid solution of an oil-soluble, butylated melamine-formaldehyde resin are dissolved in 22 grams (corresponding to 10 milliliters) of tris-(2,3-dibromopropyl)phosphate. This solution is slowly injected below the surface of 50 grams of a 6 percent aqueous solution of poly (vinyl alcohol) containing 0.24 grams of a 55 percent aqueous solution of melamine-formaldehyde while maintaining vigorous agitation.

The resulting emulsion is then cured at 45°C. with agitation for 2 2½ hours followed by the addition of 0.9 gram of 20 percent aqueous ammonium chloride solution.

The resulting microcapsules have an average diameter of about 6 microns.

EXAMPLE 2

A polyester shag rug consisting of a woven jute backing fabric having the polyester yarn tufted thereto is coated on the back side thereof with the capsule slurry prepared in Example 1. A coat weight of 0.067 grams of dry material per square centimeter of backing is employed. The shag rug is dried at room temperature overnight. Next, a styrene-butadiene backing is coated over the capsule coating, at a coat weight of 0.26 grams of dry latex per square centimeter. Once again, the carpet is dried overnight and finally, baked at a temperature of 170°C. for a period of 8 minutes.

The resulting carpet exhibits reduced flammability when compared with the flammability of an untreated sample.

EXAMPLE 3

A solution of 1500 grams of a chlorinated aliphatic hydrocarbon, 1500 grams of tetrabromoethane, and 120 grams of an adduct of toluene diisocyanate and trimethylolpropane (75 percent in ethyl acetate) are emulsified in a mixture of 1200 grams of a 6 percent by weight solution of a high molecular weight, 99% hydrolyzed poly(vinyl alcohol) (commercially available from CPC International as Covol 9870) and 5350 grams of an 8 percent by weight poly(vinyl alcohol) solution, said poly(vinyl alcohol) being a high molecular weight, 87 percent hydrolyzed grade (commercially available from CPC International as Covol 9740) to give particles having an average diameter of 7 microns. The resulting dispersion is cured for 2 hours at a temperature of 60°-65°C. The resulting microcapsules have an average particle diameter of about 7 microns.

EXAMPLE 4

The microcapsular product of Example 3 is diluted to 20 percent solids and is sprayed onto a moving 20 inch jute web on a Keegan Laboratory paper coater. The web is then passed through a forced draft oven at a temperature of 100°C. where it is dried. The residence time in the oven is about 3 minutes.

The jute web is then tufted with polyester yarn to form carpeting. The resulting carpeting shows a greatly reduced flammability.

EXAMPLE 5

Eighty-two grams (1.36 mols) of urea and 220 grams (2.72 mols) of aqueous formaldehyde are admixed at a pH of 8.0, adjusted with triethanolamine and are reacted at 70°-75°C. for 1 hour. The resulting prepolymer solution is cooled to room temperature and diluted with 400 milliliters of water.

The pH of the prepolymer solution is then decreased with formic acid to 5.0 and vigorously agitated while slowly adding a mixture of 30 milliliters (66 grams) of tris-(2,3-dibromopropyl)phosphate and 90 milliliters (144 grams) of tetrachloroethylene. The agitated system is adjusted to a pH of 1.0 to 1.5 with additional formic acid and is reacted for 4 hours. At the end of the first hour of reaction, 100 milliliters of water are added to reduce the viscosity. The pH is readjusted with a 6 percent monobutylacidphosphate solution and the curing reaction is continued with agitation for more than 1 hour. The product is then transferred into ice-cold water. The pH adjusted to 1.0 to 1.5 with formic acid, agitated for an additional 30 minutes, vacuumed filtered and dried at room temperature.

Microcapsules having an average particle diameter of about 15 microns in diameter are thereby provided.

EXAMPLE 6

The microcapsules of Example 5 are coated onto a moving 20 inch jute web, and the web is then passed through a forced draft oven at 100°C. and dried for a period of about 3 minutes. The jute web is then tufted with monofilament nylon to form carpeting. The resulting carpeting shows greatly reduced flammability over a sample prepared in the identical manner but without the microcapsules.

EXAMPLE 7

Twenty parts of weight of the microcapsules of Example 5 are incorporated into 100 dry parts by weight of a carboxylated SBR latex coating composition which is then coated onto the back of a jute backing that has been tufted with polyester yarn.

The resulting carpet is dried overnight and baked at a temperature of 100°C. for about 10 minutes. This sample exhibits reduced flammability over a similar untreated sample.

Although the invention has been described in considerable detail with particular reference to certain preferred embodiments thereof, variations and modifications can be effected with the spirit and scope of the invention as described hereinbefore, and as defined in the appended claims.

We claim:

1. A flame-resistant, tufted product, which comprises a primary backing substrate having a first side and a second side, yarn tufted to said first side of said primary backing substrate and projecting through said primary backing substrate to form a pile on said first side, an adhesive layer superposed over said second side of said primary backing substrate, said primary backing substrate bearing microcapsules containing a flame-retarding agent, said flame-retarding agent being releasable from said microcapsules under the action of

heat on said microcapsules to form a flame-resistant atmosphere surrounding said yarn tufts.

2. The flame-resistant tufted product of claim 1 wherein additional microcapsules containing a flame-retardant agent are incorporated into said adhesive layer.

3. The flame-resistant product of claim 1 wherein said microcapsules are present in a coating on said primary backing substrate which is between said primary backing substrate and said adhesive layer.

4. The flame-resistant product of claim 1 wherein said microcapsules are present in a coating on said first side of said primary backing substrate.

5. The flame-resistant product of claim 1 wherein said microcapsules have walls comprising a partially condensed formaldehyde condensation product.

6. The flame-resistant product of claim 5 wherein said partially condensed formaldehyde condensation product is urea-formaldehyde or melamine-formaldehyde.

7. The flame-resistant product of claim 1 wherein said primary backing substrate is jute.

8. The flame-resistant product of claim 1 wherein said secondary backing layer is a latex.

9. The flame-resistant product of claim 1 wherein said yarn is a polyester, nylon or a polyolefin.

10. The flame-resistant product of claim 1 wherein said flame-retarding agent is highly volatile.

11. The flame-resistant product of claim 10 wherein said flame-retarding agent is tetrabromoethane.

12. The flame-resistant product of claim 1 wherein said microcapsules have an average particle diameter between 10 and about 20 microns.

13. A process for the production of a flame-resistant, tufted product, which comprises providing a primary backing substrate having a first side and a second side, contacting said substrate with a dispersion comprising microcapsules containing a volatile flame-retarding agent so as to provide a uniform coating of said capsules over said substrates, said flame-retarding agent being releasable from said microcapsules under the action of heat on said microcapsules, tufting said primary backing substrate with a flammable yarn so as to provide tufts extending from said first side of said substrate, and coating said second side of said substrate with an adhesive layer.

14. The process of claim 13 wherein said primary backing substrate is a fibrous material, and said microcapsules penetrate said substrate.

15. The process of claim 14 wherein said primary backing substrate is jute.

16. The process of claim 13 wherein said adhesive layer is a latex.

17. The process of claim 13 wherein said flame-retardant agent is tetrabromoethane.

18. The process of claim 13 wherein said microcapsules have walls comprising a partially condensed formaldehyde condensation product.

19. The process of claim 18 wherein said microcapsules have walls comprising urea-formaldehyde or melamine-formaldehyde.

20. The process of claim 13 wherein said microcapsules have an average particle diameter of between about 10 and about 20 microns.

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