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FLEXIBLE COVERING AND METHOD OF MAKING SAME

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This invention relates to covering structure and method of and coating composition adapted to make the same. This invention relates especially to flexible sheetlike covering structures suitable for floors, walls, articles of furniture and the like and is particularly adapted to be practiced in connection with covering structures comprising a bituminized sheetlike fibrous foundation member, a sealing coat applied to the foundation member, and a decorative and wear-resistant coating applied over said coat.

It is a purpose of this invention to afford a coating composition including an aqueous emulsion which is adapted to dry to form a water-resistant and permanently adherent coating. It is also a purpose of this invention to afford a method whereby a coating having desirable properties such as water-resistance and high and permanent adherence may be obtained using an aqueous emulsion. It is a further purpose of this invention to afford a covering structure including a sealing coat which has a high degree of resistance to moisture and alkalis and affords a strong and permanent bond between the surface coating of decorative and wear-resistant material and the bituminized foundation member.

Coverings, such as floor coverings sold in roll form or in the form of preformed rugs have been extensively made and sold, which coverings include a sheet of felt impregnated with a heavy bituminous saturant adapted to augment the strength of the felt and to increase the resistance of the felt to water and which coverings include a wear-resistant and decorative surface coating that is usually applied by printing and that is composed of some suitable base such as a drying oil or cellulose derivative together with pigment and filler. Inasmuch as the bitumen in the felt base tends to bleed into the decorative surface coating with resulting discoloration and softening of the surface coating, it has been universal practice to interpose between the bituminized base and the surface coating, one or more sealing coats which are composed of materials designed to resist penetration by the bitumen in the foundation and to thereby prevent the bitumen from bleeding into the wear-resistant and decorative surface coating.

Various materials have been used as the sealing coat, and, in order to prevent a solvent in the sealing coat from dissolving the bitumen in the foundation member, sealing coats have sometimes been applied in the form of an aqueous emulsion. The purpose in using an aqueous emulsion is to

help prevent the bitumen in the foundation member from bleeding into the sealing coat during the drying of the sealing coat, as would be the case if a solvent were used in the sealing coat which is likewise a solvent for the saturant in the foundation member.

A floor covering of the character above mentioned is generally cleaned by washing it with water and, especially when used in kitchens or bathrooms, water is frequently spilled by accident onto the floor covering. It is therefore important that the floor covering be resistant to water and to mixtures of water and alkali. Heretofore, however, considerable difficulty has been encountered due to the destructive action of water and of water and alkali on floor coverings of this character.

Some of the features of this invention relate to a novel coating composition in the form of an aqueous emulsion which is adapted to produce a coating having especially high resistance to water and to mixtures of water and alkali. According to this invention, certain substances mentioned hereinbelow are incorporated in an aqueous emulsion in combination with special emulsifying agents so as to afford an aqueous coating composition which, after application and drying, yields an ultimate coating having increased resistance to water and to mixtures of water and alkali.

Other features of this invention reside in coverings of the character above mentioned which have increased durability and resistance to water and to water and alkalis. Heretofore attempts have been made to improve upon coverings of the character above mentioned by increasing the resistance to the action of water and alkalis of the wear-resistant and decorative surface coating of the covering. As contrasted to such attempts to improve coverings of this character, the present invention proceeds in an entirely new way and is concerned chiefly with the resistance to water and to alkalis of the part of the interior of the covering where the sealing coat is in juxtaposition with the bitumen impregnated foundation member. While the upper surface of a floor covering is subjected to washing, the water used in washing frequently gets underneath the covering, especially adjacent the edges thereof, and, due to the fact that the foundation felt is somewhat porous even though bituminized, the water tends to penetrate from the back of the covering so as to come into contact with the sealing coat. When alkali is present in the wash water, the

alkali will likewise be carried through to the sealing coat. Moreover, if such a covering is used on a damp foundation, the moisture will penetrate through the foundation member to the sealing coat. It is liquids of this character which penetrate the covering from the back and which have been found to be the most insidious in causing premature disintegration of the floor covering. According to the present invention, a new type covering of the character above mentioned is afforded wherein the destructive liquids which enter the covering from the back are met by a new type of sealing coat directly applied to the bituminized felt foundation. In this manner, the covering is given increased resistance to what may be referred to as "internal rot" that in the absence of this invention tends to cause premature disintegration of the covering. When this internal rot occurs, the bond between the bituminized felt base and the decorative and wear-resistant surface coating is prematurely destroyed with resulting peeling off or chipping off of the surface coating regardless of the high quality and thickness of this surface coating.

In order to enable this invention to be readily understood, it will first be described in connection with a specific illustration thereof. In this illustration, reference is to parts by weight. A mixture containing 150 parts of fish oil, 50 parts of China-wood oil, and 33 parts of calcium resinate (4% lime) is heated to about 400° F. to form a homogeneous mixture. Then 2.5 parts of cobalt acetate is added with stirring and the temperature is raised to 485° F. After removing the source of heat, 11 parts of an oil soluble phenol-aldehyde resin formed by previously reacting cresylic acid with formaldehyde is added with stirring until it becomes completely dissolved.

The binder vehicle or base prepared in the foregoing manner is then made up into an emulsion paint. To this end, 28 parts of the vehicle is agitated with 2 parts of linseed oil fatty acids, 2 parts of ammonia (29° Bé.) and 40 parts of water. The ammonia and linseed oil fatty acids react to form an ammonium soap which acts as an emulsifying agent for the oil-resin vehicle or base and causes it to be dispersed in an emulsified condition in the water. A filler is then added, such as 186 parts of whiting and 2 parts of iron oxide. If desired, the resulting emulsion paint can be ground as in a colloid mill to increase the emulsion characteristics of the paint.

The resinous product of reaction between cresylic acid and formaldehyde can be prepared in any suitable way. In order that the illustration hereinabove given may be complete in this regard, the following procedure will be described. 100 parts of crude cresylic acid, 40 parts of formaldehyde (40% soluble in water) and 1 part of concentrated hydrochloric acid are placed in a container provided with a reflux condenser and an agitator. The liquid while being agitated is slowly heated to about 160° F. when a vigorous exothermic reaction takes place. The exothermic reaction carries the temperature of the reaction materials to about 210° F., at which temperature the reaction materials are maintained until the reaction is completed (about two hours). The reaction products are then heated in an open vessel to drive off water and any unreacted fraction of the cresylic acid and until a removed sample is brittle when cooled. The melting point of the resin thus produced usually lies between about 126° F. and about 142° F.

The emulsion paint prepared in the foregoing manner is ready for use. While it may be used for a variety of purposes, the use of the emulsion paint will be described for the purpose of further illustration as a sealing coat in the manufacture of a covering structure comprising a bituminized sheetlike foundation member, a sealing coat applied to the foundation member, and a decorative and wear-resistant surface coating applied over the sealing coat. In such a covering structure, the foundation member may, for example, be made by impregnating a sheet of water-laid felt with a bituminous saturant having a melting point of about 160° F. The emulsion paint can be applied directly to the bituminized felt while it is still warm or after it has cooled by spreading it with a doctor blade or roll coater or by any other suitable method of application. After the emulsion has been applied as a coating, the water in the emulsion is caused to evaporate. Moreover, since the emulsion is stabilized by an ammonium soap, the ammonium component of the emulsifying agent is also caused to be driven off or evaporated. Likewise, the component of the paint which is adapted to dry or harden by oxidation is caused to become oxidized. All of these operations can be performed simultaneously in a drying operation by disposing the coated belt base in a suitable atmosphere, preferably warmed slightly above ordinary atmospheric temperatures, into which the water together with the ammonium component of the emulsifying agent can become dissipated and in which the drying oil component of the coating can become oxidized.

After the emulsion paint has been applied and has become dried, one or more additional coats thereof can be applied and permitted to dry, depending upon the ultimate thickness of the sealing coat that is desired. Alternatively, one of the well-known types of oil-solvent compositions may be employed for such additional coatings as may be desired. It is usually desirable after the final application of the sealing coat to elevate the temperature to accelerate the completion of the oxidation and hardening of the drying oil component of the sealing coat or coats and the driving off of the ammonia.

As a wear-resisting and decorative surface coating, any suitable material may be used. Surface coatings which are extensively used for this purpose comprise a drying oil base mixed with suitable pigments and fillers and are applied by printing. Of course, other surface coatings may be used and applied in any desired manner. After the surface coating has been applied, the resulting covering can be baked, if desired, so as to hasten the drying of the coatings to their ultimate desired condition. Since the improved sealing coat renders the covering highly resistant to water and to alkalis, it is not necessary to utilize a phenol-aldehyde resin in the decorative and wear-resisting surface coat although such a resin can be used, if desired. In usual practice, the omission of a phenol-aldehyde resin in the surface coat is to be preferred, especially from the point of view of facility and cost of manufacturing the covering.

Any suitable protective coating for the back of the covering structure may be used, but an emulsion paint of the type above described will be found particularly satisfactory.

A floor covering such as that above illustrated, when subjected to tests, has been found to have

highly desirable characteristics. In the first place, the sealing coat was found to provide a very strong and permanent bond between the bituminized felt foundation and the decorative wear-resisting surface coating and to be highly resistant to chipping off of the surface coating even after moisture has been permitted to be absorbed from the back of the covering over long periods of time. Moreover, upon long standing, the sealing coat was found to prevent the bitumen in the foundation from bleeding into the surface coating. Specimens of the covering were tested after having been dried for four days by immersing the specimens in boiling water and from time to time noting the condition of the sealing coat. The sealing coat prepared in the manner above illustrated was found to have high resistance to the action of the boiling water as evidenced by the low rate of softening or other failure of the sealing coat. The covering made as hereinabove illustrated likewise was found to have increased resistance to kerosene.

Features of this invention which are exemplified in the foregoing specific illustration relate to the completed covering and to the emulsion paint used in making the sealing coat.

Improvements in the completed covering result from several factors which preferably occur in conjunction but which likewise constitute separate features of this invention. Specimens of the covering which included the cresylic acid-formaldehyde resin in the sealing coat, when tested in comparison with specimens similarly prepared but omitting the resin or using a natural resin in lieu thereof, were found to afford a very strong and permanent bond between the surface coating and the bituminized felt so as to result in a flooring having improved durability under normal conditions of use. The sealing coat was found to give the covering as a whole increased resistance to water and alkalis by preventing damage due to absorption of water and alkalis from the back of the covering. For these reasons, the employment of such a resin or an equivalent oil-soluble phenol-aldehyde resin in a sealing coat applied directly to a bituminized foundation and between such foundation and a wear-resistant and decorative surface coating is regarded as a feature and advantage of this invention.

Further advantages result from the fact that the resulting sealing coat is substantially free of an emulsifying agent. As hereinabove mentioned, the emulsifying agent used in the aqueous emulsion paint is an ammonium soap, but since the ammonium content of ammonium soap is driven off during the drying of the coating and since the residue, i. e., the linseed oil fatty acids, remaining in the coating hardens by oxidation and is ineffective as an emulsifying agent, the sealing coat in its ultimate condition is substantially free of an emulsifying agent. This absence of an emulsifying agent in the dried sealing coat contributes greatly to its water-resistance. If an emulsifying agent such, for example, as an ordinary alkali metal rosin soap were used, such soap would remain in the sealing coat after it had been dried and the presence of this soap in the sealing coat would render the sealing coat susceptible to attack by water. Much greater resistance to water is secured when a sealing coat containing a drying oil or an acid resin is substantially free of alkali metals which, if present, would tend to form emulsifying agents with any acid resin or drying oil present in the

sealing coat. Moreover, the absence in the sealing coat of any substance which is water-soluble contributes to its resistance to water.

The use of a water-insoluble metallic resinate such as calcium resinate in the sealing coat of the covering is likewise advantageous in the practice of this invention. Water-insoluble metallic resinates impart to the sealing coat a high resistance to water and to alkalis and the use thereof in the sealing coat of a covering of the character mentioned constitutes an advantageous feature of this invention. Moreover, the employment of a water-insoluble metallic resinate in combination with a phenol-aldehyde resin, as hereinabove mentioned, results in a sealing coat which is superior to a sealing coat wherein the phenol-aldehyde resin is not employed in combination with water-insoluble metallic resinate.

It is regarded as apparent from the foregoing description of some of the features and advantages of coverings embodying this invention, that in the practice of this invention covering structures are afforded which are superior to coverings of like character which have been manufactured heretofore using different types of sealing coats. The aim in covering structures of this character is to achieve maximum durability without necessitating excessive cost. According to the present invention, coverings are afforded without substantial increase in cost which, as compared with coverings heretofore manufactured, have improved resistance to what has been referred to above as internal rot.

From the foregoing description, it is apparent that an aqueous emulsion paint is afforded which by virtue of its novel composition can readily be applied and will dry to an ultimate coating that is much more permanent than coatings that can be produced using emulsion paints heretofore made. Various factors contribute to this result which are preferably employed in conjunction but which may, however, be employed separately. In this connection, the emulsion paint is adapted to produce an ultimate coating which is substantially free of water-soluble substances and of any emulsifying agent. Moreover, the ultimate coating is composed of substances which impart permanent adherency to the coating and resistance to water and alkalis. The phenol-aldehyde resin gives to the drying oil increased resistance to water and alkalis and increases the permanent character of the bond afforded by the coating. The water-insoluble metal resinate likewise results in an ultimate coating having high resistance to alkalis. Moreover, since an unstable emulsifying agent is used which decomposes after the coating has been applied, the ultimate coating does not contain an emulsifying agent as would be the case if a stable emulsifying agent such as an alkali metal resinate were used. Likewise, from another point of view, instead of using an emulsifying agent which is undesirable in the hardened coating, there is used an emulsifying agent having a volatile component and a non-volatile component, which emulsifying agent loses its volatile component during drying, leaving a component of the emulsifying agent which does not act as an emulsifying agent and which is water-insoluble. When, for example, the emulsifying agent that is used is an ammonium soap of a drying oil fatty acid, as in the foregoing illustration, the component of the emulsifying agent which remains in the coating after the evaporation of the water, is a drying oil fatty acid, namely, a substance that is adapt-

ed to become hardened to form a water-resistant mass and this is of still further advantage. An ammonium resinate will likewise decompose to leave a resin in the ultimate film, the resin being desirable in the binder vehicle for the coating, although less desirable than a drying oil acid. In this manner, an emulsion paint is afforded which is capable of drying to form an ultimate coating that is free of any water-soluble matter and is likewise free of any emulsifying agent. In this manner, certain substances which are desirable in the coating for reasons such as increased resistance to water and alkalis are combined with emulsifying agents that preserve the advantages resulting from the use of such substances. Thus according to this feature of this invention full advantage is taken of the effects of the oil-soluble phenol-aldehyde resin or water-insoluble metal resinate, which effects would to a large extent be counteracted if an emulsifying agent or water-soluble material were present in the ultimate coating.

While the emulsion paint of the invention is desirable for many purposes, it is especially desirable in a covering structure, as a sealing coat applied directly to a bituminized foundation member and between the foundation member and a decorative and wear-resisting surface coat. According to this invention, there has been afforded for the first time a paint comprising an oil-soluble phenol-aldehyde resin which can be applied in the form of an aqueous emulsion to a bituminized foundation and which in the completed covering affords a strong and permanent bond between the foundation member and a decorative and wear-resisting surface coating and which has high resistance to moisture and to mixtures of water and alkali.

Features of this invention also relate to the method of making a covering of the character herein mentioned. In this connection, a coating containing a drying oil and a resin selected from the group consisting of oil-soluble phenol-aldehyde resins and water-insoluble metal resinates is applied in the form of an aqueous emulsion to a bituminized foundation member, a decorative and wear-resistant surface coating being applied thereafter over the sealing coat after it has sufficiently dried. To provide the aqueous emulsion, an unstable emulsifying agent such as an ammonium soap is used which decomposes after application of the emulsion and during the drying of the sealing coat. According to this method, a sealing coat can be applied which is impervious to bitumen during and after application, which forms a strong and permanent bond between the bituminized foundation member and the wear-resistant and decorative surface coating, and which is highly resistant to water and alkalis.

It is to be understood that this invention is not to be limited to the illustration which has been described above. In this connection, the resinous reaction product of cresylic acid and formaldehyde is but one example of what are referred to herein as "oil-soluble phenol-aldehyde resins". These resins, as is already known, are formed by resinification between a phenolic body and a body containing a methylene group; reference herein being confined to those resins of this type which are oil-soluble. Other examples of oil-soluble phenol-aldehyde resins include the resinification reaction products of compositions such as the following, reference being to parts by weight:

	(1)	Parts by weight
Phenol.....		100
Tertiary amyl phenol.....		33
Formaldehyde (40% soluble in water).....		57
Concentrated HCl.....		1
	(2)	
Phenol.....		100
Tertiary butyl phenol.....		33
Formaldehyde (40% soluble in water).....		57
Concentrated HCl.....		1
	(3)	
Cresylic acid.....		100
Paraformaldehyde.....		16
Concentrated HCl.....		1
	(4)	
Cresylic acid.....		100
Hexamethylenetetramine.....		14
Concentrated HCl.....		1

While calcium resinate has been suggested as a water-insoluble metal resinate, the water-insoluble resinates of other metals may also be used, such as the resinates of the other alkaline earths and of zinc and aluminum. Moreover, other drying oils can be used in the binder or base for the emulsion point such as linseed, perilla, soya bean and hempseed. Such drying oils, when lightly bodied, are regarded as preferable to heavily bodied oils.

As the emulsifying agent, ammonium soaps may be used, ammonium resinates and ammonium soaps of fatty acids being very much preferred inasmuch as each of these substances results in a desired binder component for the binder vehicle. The ammonium soaps of drying oil fatty acids are most desirable because after the ammonium component of the soap has been dissipated, the drying oil fatty acid hardens by oxidation to promote the formation of a hard and permanent component of the binder vehicle of the coating. It is preferable either to form an ammonium soap prior to incorporating the same in the emulsion paint or to incorporate in the mixture some substance such as a drying oil fatty acid which readily takes up separately added ammonia to form an ammonium soap so as to avoid the presence of an excessive amount of free ammonia. However, it is possible to incorporate ammonia in sufficient quantity to react with any acid material in the drying oil or acid resin constituents of the emulsion paint and form an ammonium soap, but this is not preferred due to the fact that an undesirably large amount of ammonia has to be used. The ammonium soaps of resins and of drying oil acids are regarded as especially desirable emulsifying agents which decompose and give an ultimate coating that does not contain the emulsifying agent. Emulsifying agents which decompose after application of the coating so as not to occur in the ultimate coating after it has been dried are referred to as unstable.

Any suitable filler may be used in addition to or other than the whiting and iron oxide above mentioned such as powdered silica, clay, barium sulphate, talc, etc. Likewise any suitable drier, if desired, may be used in the emulsion paint such as cobalt or manganese compounds that are oil-soluble or react with oil to become oil-soluble.

The proportions of the ingredients in the emulsion paint (and in the hardened coating resulting therefrom) may be varied depending upon the characteristics desired in the paint or in the ultimate coating. The amount of water that is used

will depend to some extent upon the consistency desired during application and upon the thickness of the coating that is to be produced, the resulting coating being thinner upon using increased amounts of water. Likewise, the amount of filler that is used is subject to considerable variation although generally enough filler to afford a satisfactory body is desirable while avoiding such excess of filler as would result in a coating of a loosely coherent character.

The binder portion of the coating composition usually comprises a major proportion of drying oil or of equivalent substance adapted to dry by oxidation and preferably comprises about 80 to 84% of drying oil. The balance of the binder portion of the coating composition preferably comprises about 4% to 16% of an oil-soluble phenol-aldehyde resin together with some other resin which is preferably a water-insoluble metal resinate. The entire resin component may consist of an oil-soluble phenol-aldehyde resin but in practice the use of this resin in combination with another resin, preferably a water-insoluble metal resinate, is to be preferred. While the oil-soluble phenol-aldehyde resin may be omitted in the practice of some features of this invention, its omission is accompanied by the omission of the advantages that result from its use and for this reason the inclusion of an oil-soluble phenol-aldehyde resin is regarded as distinctly preferable in the practice of this invention. For example, a binder vehicle which contains about 50% to about 95% of a drying oil and about 5% to about 50% of a resin selected from the group consisting of phenol-aldehyde resins and water-insoluble metal resinates may be used in the practice of this invention although in order to achieve the special advantages which result from the employment of an oil-soluble phenol-aldehyde resin about 3% to 20% of the binder vehicle should be a synthetic resin of this type.

More specifically, the following is illustrative of a binder vehicle which may be used:

	Per cent
45 Drying oil.....	50 to 92
Oil-soluble phenol-aldehyde resin.....	3 to 20
Water-insoluble metal resinate.....	5 to 47

While the base or binder portion of the coating composition may contain a resin other than a phenol-aldehyde resin and water-insoluble metal resinate, such as rosin, ester gum and the like, it is preferable that at least $\frac{1}{2}$ of the resin component consist of an oil-soluble phenol-aldehyde resin, and that the amount of oil-soluble phenol-aldehyde resin in the binder vehicle as a whole be about 3% to 20%, drying oil component being as aforesaid at least 50%. An example of a desirable binder vehicle which may be used in the practice of this invention is one containing 75% to 85% of a drying oil and 15% to 25% of a resin, of which resin at least $\frac{1}{2}$ is an oil-soluble phenol-aldehyde resin. When a water-insoluble metal resinate is also used, at least about $\frac{2}{5}$ of such resin is desirable in the total resin.

It is to be understood that the hereinabove described examples of this invention are merely for the purpose of affording illustrations thereof and that the scope of this invention is to be limited only by language of the following claims.

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1. A method of making a flexible covering which comprises impregnating a fibrous sheet-

like foundation member with a bituminous saturant, applying directly to a surface of said foundation member an aqueous emulsion including water, a drying oil, water-insoluble and alkali-resistant resin and an ammonium soap as an emulsifying agent, subjecting the applied emulsion to treatment causing the water to evaporate, the ammonium component of the ammonium soap to be driven off and the drying oil to oxidize, to form a continuous sealing coat for the bituminized foundation member, and subsequently applying over said sealing coat a decorative and wear-resisting coating.

2. A method of making a flexible covering which comprises impregnating a fibrous sheet-like foundation member with a bituminous saturant, applying to a surface of said foundation member an aqueous emulsion including water, a drying oil, an oil-soluble phenol-aldehyde resin, and an ammonium soap as an emulsifying agent, subjecting the applied emulsion to treatment causing the water to evaporate, the ammonium component of the soap to be driven off and the drying oil to oxidize, to form a sealing coat for said bituminized foundation member, and thereafter applying over said sealing coat a decorative and wear-resisting coating.

3. A method of making a flexible covering which comprises impregnating a felted sheet with a bituminous saturant, applying to a surface of said felted sheet an aqueous emulsion including water, a mineral filler, an ammonium soap selected from the group consisting of ammonium resinates and ammonium soaps of drying oil acids, and a binder vehicle comprising a mixture of a drying oil, oil-soluble phenol-aldehyde resin and a water-insoluble metal resinate, subjecting the applied emulsion to treatment causing the water to evaporate, the ammonium component of the ammonium soap to be driven off, and the drying oil to oxidize, to form a sealing coat for the bituminized felted sheet, and thereafter applying over said sealing coat a decorative and wear-resisting coating.

4. A flexible covering which comprises a sheet-like foundation member of felted fiber impregnated with a bituminous saturant, a sealing coat applied directly to one surface of said fibrous foundation member, which sealing coat comprises a filler and a binder vehicle containing 5% to 47% of a water-insoluble metal resinate selected from the group consisting of alkaline earth metal resinates, zinc resinate and aluminum resinate, 3% to 20% of an oil-soluble phenol-aldehyde resin and 50% to 92% of a drying oil, and a decorative and wear-resistant covering applied over said sealing coat.

5. A flexible covering which comprises a sheet-like fibrous foundation member impregnated with a bituminous saturant, a sealing coat applied directly to one surface of said foundation member, which sealing coat comprises a filler and a binder vehicle containing about 75% to 85% of drying oil and about 15% to 25% of a resin of which at least about one-fifth is an oil-soluble phenol-aldehyde resin and of which at least about $\frac{2}{5}$ ths is water-insoluble metal resinate selected from the group consisting of alkaline earth metal resinates, zinc resinate and aluminum resinate, and a decorative and wear-resistant coating applied over said sealing coat.

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