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(54) **Title:** POLYURETHANE BASED COATING COMPOSITIONS

(57) **Abstract:** The present invention relates to a polyurethane based coating composition which exhibits superior properties in terms of stability, corrosion resistance and moisture permeability and which has low viscosity of the coating composition. The polyurethane based coating composition comprises a base and an activator the base component comprising a polyol, a filler, a defoamer, a catalyst and a molecular sieve and the activator comprises polyisocyanate. Preferably the polyol comprises castor polyol, 2-methyl 1, 3 dipropane diol or 1, 4 butane diol and the filler comprises mica. Mica besides being a filler also functions as a pigment extender. In some embodiments the said composition is devoid of catalyst. The said coating composition is useful in coating concrete and metal surfaces.

POLYURETHANE BASED COATING COMPOSITIONSField of the Invention

The present invention relates to novel polyurethane based coatings, particularly for selectively imparting excellent water vapour impermeability and in-can settling stability, in addition to low viscosity, for application in industrial coating. The composition of the present invention is used particularly for protection of concrete and metal surfaces.

Background of the Invention

Polyurethane (PU) coatings are widely known for their versatility of application with many advantages over the conventional coating systems. PU coatings can be divided into one pack and two pack systems, wherein the one pack system contains a dissolved fully reacted PU or blocked isocyanate, while the two pack system can contain various types of polyols, varying in functionality, chain length and backbone in one pack and isocyanate in the other pack. Both these systems can be further classified as solvent based, water based or solvent free systems. The solvent free polyurethane system offers several advantages over the solvent based system as they are environmental friendly (due to very low volatile organic compound content) and have a wide range of superior properties such as chemical resistance (due to eliminated solvent entrapment and less permeability), impact strength, and high build application in single coat apart from coating varied number of substrates. Better impermeability also helps in longer corrosion protection by making the coated film a better barrier to water vapour.

A frequently encountered problem with coating composition is that of the settling of paints over some period of storage thereby resulting in some of the pigments remaining at the bottom of the can/drum. Improper mixing affects the stoichiometry of the finished product and results in inconsistent & inferior performance on application. In case of sticky heavy settling, desired bits free consistency is not achieved even after considerable mixing during application, resulting in inferior performance. A number of publications are known that deal with solvent free polyurethane compositions for application as a protective coating.

US 2007-185241 describes cured resin matrix with a plurality of bubbles which provide improved insulative properties and improved adhesion to overlying paint coatings which in turn offers protection against corrosion and acts as heat insulator.

US 2008-229976 discloses polyurea compositions and polyurethane compositions related to stain and fouling resistant coatings that provide durable, light weight coatings which exhibit oil-repellency, water-repellency, and stain resistance.

US 6977279 relates to solventless reactive system which cure at room temperature based on blocked polyisocyanates, primer amines, compounds with oxirane groups and 2,3 dimethyl-3,4,5,6-tetrahydropyridimine.

However, most of these compositions known in the art suffer from several drawbacks, such as high cost, poor adhesion to certain substrates, high moisture permeability, tendency to settling over a period of storage and high viscosity of solvent free coating compositions which limits high filler loading required for better impact strength & cost optimization. High viscosities of the solvent-free coating compositions also need heating of the base part to reduce viscosity during application for ease of application.

Therefore there is a need to develop a coating composition which materially alleviates the aforesaid problems faced by the state of the art technologies.

Summary of the Invention

The objective of the present invention is to provide a cost effective coating which exhibits superior properties in terms of corrosion resistance, moisture impermeability, in-can settling stability and having low viscosity.

Accordingly one embodiment of the present invention is to provide a polyurethane based coating composition having a base component and an activator as another component wherein the base component comprises polyols and ingredients such as catalyst, defoamer, molecular sieve and fillers; while the activator consists essentially of polyisocyanate. The base component of the composition may comprise other ingredients such as pigments, additional polyols, amines and chain extenders.

The components of the coating composition interact synergistically to confer a unique combination of the physical and chemical properties to the resultant composition which make it superior to the state of the art compositions.

In a further aspect, the instant coating composition comprises the individual components in the following concentration:

- components of the base
 - polyol in an amount of 35 to 75 %
 - filler in an amount of 20 to 60%
 - catalyst in an amount of 0 to 5%
 - defoamer in an amount of 0.5 to 1.5 % and
 - molecular sieve in an amount of 2 to 10 %
- polyisocyanate as activator

The base and an activator is in 3:1 (v/v) proportion.

The polyol could be a diol, a triol, a higher polyol, or a mixture of one or more of the said polyols. Some other additives such as pigments, chain extenders etc also may be used to control and modify the reaction process and performance characteristics of the polymer.

This invention also relates to a process for preparing the polyurethane based coating composition wherein the process comprises initially of preparing the base component and mixing the base with the activator in a suitable proportion to provide the coating composition for use in corrosion protection of metal and concrete structures. This invention also relates to articles and surfaces coated with the instant coating composition.

The present invention possesses the following advantages:

- Excellent moisture impermeability
- In-can settling stability
- Low viscosity
- Excellent corrosion resistance and chemical resistance (towards a number of chemicals)

Other features and advantages of the present invention will become apparent as the following detailed description proceeds or may be learned by practice of the invention. The advantages of the invention will be realized and attained by means of the elements and combinations particularly pointed out in the description. It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory only and are not restrictive of the invention.

Brief description of the drawings

The present invention is illustrated in the accompanying drawings:

Figure 1 shows a graph plotting the water vapour permeability of the standard coating composition of the invention.

Figure 2 shows a graph plotting the water vapour permeability of a coating composition without mica.

Detailed Description of the Invention

For the purposes of the following detailed description, it is to be understood that the invention may assume various alternative variations and step sequences, except where expressly specified to the contrary. Moreover, other than in any operating examples, or where otherwise indicated, all numbers expressing, for example, quantities of ingredients used in the specification are to be understood as being modified in all instances by the term "about". It is noted that, unless otherwise stated, all percentages given in this specification and appended claims refer to percentages by weight of the total composition.

Thus, before describing the present invention in detail, it is to be understood that this invention is not limited to particularly exemplified systems or process parameters that may of course,

vary. It is also to be understood that the terminology used herein is for the purpose of describing particular embodiments of the invention only, and is not intended to limit the scope of the invention in any manner.

It must be noted that, as used in this specification and the appended claims, the singular forms "a," "an" and "the" include plural referents unless the content clearly dictates otherwise. Thus, for example, reference to a "polyol" may include two or more such polyols. Unless defined otherwise, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art to which the invention pertains.

The coating composition of the present invention is a solvent free urethane based composition comprising a base and an activator, wherein the base comprises a number of additives like polyols, defoamer, molecular sieve, filler, catalyst and other optional additives; while the activator consists essentially of polyisocyanate.

The polyols form an essential ingredient of the base component of the present invention. A polyol is a polymeric alcohol containing multiple hydroxyl groups. In polymer chemistry, polyols are defined as compounds with multiple hydroxyl functional groups available for organic reactions. A molecule with two hydroxyl groups is a diol, one with three is a triol, one with four is a tetrol and so on. Monomeric polyols such as glycerin, pentaerythritol, ethylene glycol and sucrose often serve as the starting point for polymeric polyols. These materials are often referred to as the "initiators" and reacted with propylene oxide or ethylene oxide to produce polymeric polyols. Polymeric polyols could be polyether polyols, polyester polyols or OH terminated polyacrylics or combination of them. The characteristics of polyols depends on are their molecular backbone, initiator, molecular weight, percentage primary hydroxyl groups, functionality, viscosity and so on. Many polyols are polydispersive materials, being blends of two or more polyols each of specific molecular weights, to give intermediate molecular weight materials.

Natural oil polyols or saturated aliphatic polyester polyols with low viscosities at room temperatures are used in the production of polyurethanes. Natural oil polyols, also known as NOPs or biopolyols, are polyols derived from vegetable oils by several different techniques. The primary use for these materials is in the production of polyurethanes. The most promising natural oils for the industrial development of biobased polyols are soybean oil, castor oil, palm oil, and canola oil. Castor oil, which is obtained from castor seed, is a triglyceride of fatty acid wherein 90% of the fatty acid chains are ricinoleic acid, has presence of OH functional group on 12th carbon atom, which makes it polar and reactable, which is not the case with other seed oils. Moreover, castor polyols derived from castor oil possess long C chains with a functional unbranched "OH" group which provides flexibility on reaction with isocyanate to form the polyurethane bond. Of particular importance is the castor oil of BSS Grade or commercial grade castor oil. More particularly, the castor polyol is Jagropol 115. Also, tri-functional oil-derived polyol having hydroxyl number ranges

from 320 to 350 mg KOH/g containing both primary and secondary hydroxyl groups, which are completely miscible with a wide variety of natural oil polyols, demonstrate ability to rapidly build polyurethane cross-linked networks. Preferably the trifunctional polyol is polypropylene ether polyol (Desmophen 1380BT).

5 Di primary diols also can be used as an ingredient of the base composition of the present invention. Diols - alcohols that contain two reactable hydroxyl groups, can be classified as linear/branched or aliphatic/aromatic diols. Of particular interest is 2-methyl 1,3 dipropane diol due to its unique branched structure (i.e) asymmetrical nature, conferred upon it due the presence of the odd number of carbon atoms between the two hydroxyl groups thus inhibiting close packaging
10 thereby exhibiting greater transparency, weatherability, longer shelf life, aiding in suspension of heavy fillers/solids, decreased viscosity, resist settling over the resin when stored for long periods of the end use applications. Preferably the 2-methyl 1,3 dipropane diol is MPDiol. 1,4 butane diol is also a preferred diol.

Fillers in this invention serve multiple functions based on the type of filler used. Fillers may
15 thicken the composition, support its structure, prevent sagging, increase volume and also enhance the technical properties of the composition.

Among the fillers used, of particular importance in the dried film is mica. Mica acts as filler providing a smooth consistency, improving the workability of the compound, and providing resistance to cracking. Mica's value is based on its unique lamellar shape, which forms compact
20 packing in the dried film and hence reduces film impermeability.

Also in this invention, mica functions as a pigment extender that facilitates suspension, reduces chalking, prevents shrinking and shearing of the paint film, increases resistance of the paint film to water penetration and weathering, and brightens the tone of colored pigments. In addition mica coated with titanium dioxide (TiO_2) results in a pigment that produces a reflective color
25 depending on the thickness of the coating. Also, the TiO_2 acts as a white pigment providing opacity to the dried film. Of preference is TiO_2 Anatyze or Rutile. In addition to TiO_2 , carbon black such as 318M Black serves as a black pigment which in combination with the white pigment provides a grey colour to the composition. Various pigments can be added for different color of the final composition. Other fillers such as barium sulphate not only might serve as a white pigment but also
30 modify the consistency and help in providing impact resistance to the application. Of preference is Barytes 2080. One other filler used in these polyurethane compositions is Silica. Silica not only leads to cost reductions by increasing the bulk of the composition but also prevents sagging of the resin.

Catalysts are used to catalyse the polymerization of the polyol with the polyisocyanate.
35 Amine compounds and organo metallic complexes are used as catalysts. Particularly, amines such as tertiary amines, difunctional primary amines etc are used. Of particular preference is an aromatic

diamine such as diethyl toluenediamine which is commercially available under the name Ethacure 100. The catalyst could be used in an amount of 0 to 5% depending on the mode of application by spray or brush. Thus in an embodiment of the invention the composition is devoid of catalyst. The said composition without catalyst is suitable for application by brush also by increasing the usable
5 life.

Defoamers are used to modify the characteristics of polyurethane composition by eliminating process defects like entrapped bubbles and surface defects such as pin holes and orange peelmarks, acting as air release and anti-foaming agents. Of particular preference are silicone free defoamers such as BYK 054 or BYK 535 or BYK 1794 or Foamex N.

Molecular sieves or moisture scavengers which contain tiny pores of a precise and uniform size are used as an adsorbent for gases and liquids. Of preference is highly effective moisture scavenger for coating systems that adsorbs practically only water. Furthermore, the presence of moisture will consume isocyanate, which is highly reactive to water. Of particular preference are micronized, highly porous, crystalline aluminosilicate with pore openings of approximately 3 Å such
10 as SYLOSIV A3 or Purmol 3ST or A4 with 4 Å pore openings. Paste forms of the molecular sieves P3 and P4 can also be used.

Chain extenders are low molecular weight hydroxyl and amine terminated compounds that play an important role in the polymer morphology of polyurethane chains. Preferably, the chain extender is an amine. More preferably the amine is polyetheramines such as Jeffamine D 400.

Pigments which could be used in the invention comprises synthetic iron oxides. Preferred examples of pigments are Bayferrox Black 318M along with Titanium dioxide to create Gray color of the composition.

In a preferred embodiment of the present invention, the base of the polyurethane based coating composition comprises 35 to 75 % of polyol mix which includes 0.5 to 5 % of trifunctional polyol and 1-10% of 2-methyl 1,3 dipropane diol, 3 to 25% of mica, 0.5 to 1.5 % of defoamer, 2 to
25 10 % of molecular sieve, and 0 to 5% of catalyst.

Further, the composition may optionally contain 0 to 5% of chain extender, 5 to 40% of barium sulphate, 5 to 40% of silica, 2 to 10% of titanium dioxide and 0.15 to 5% of color pigments.

Another major embodiment of the present invention is related to a process for preparing the polyurethane based coating composition wherein the process comprises of initially preparing the
30 base component. The base component is nothing but mixture of OH functional polyol components with molecular sieve, fillers, pigments, catalysts and additives. The base component is prepared by dispersing the solid materials viz., pigments, fillers & other additives in polyols in high speed disperser (HSD) or twin shaft disperser and stabilizing the same with rest of the resin and additives.
35 The solid matters can be ground to finer to medium grind depending on the application requirement.

The base component is mixed with the activator in a suitable proportion to provide the coating composition for use in corrosion protection of metal and concrete structures.

The solvent free urethane coating of the invention is applied by plural feed spray equipment. Most of the known solvent-free urethane compositions are high in viscosity and hence the base component is always heated in the range of 40 to 55°C to reduce the viscosity (viscosity reduces with increase in material temperature) to facilitate the ease of application. Since the viscosity of the instant composition is low, it can be easily applied without heating the base part at temperature above 30°C. The same product can be made applicable by brush also by increasing the usable life. This needs removal of the catalyst from the composition.

The following examples merely illustrate certain aspect of the invention and is to enable those skilled in the art to more clearly understand and to practice the present invention. They should not be construed as limiting the scope of the invention, but merely as being illustrative and representative thereof.

EXAMPLE 1:

In a preferred embodiment of the present invention, the standard urethane coating composition of the invention has the following composition. The composition comprises A) Base component and B) Activator.

A) Base component has the composition as shown in Table 1

Table 1

Components	Weight percentage range	Exact weight percentage
Jagropol 115 Polyol	20-50	22.76
Castor Oil	20-50	20.81
M P Diol	1 - 20	2.88
Desmophen 1380BT	0.5-5	1.28
Mica	3-25	5
Baryte 2080	5-40	10.38
Silica 1240	5-40	25.97
Etahcure 100	0-5	0.64
Sylosiv A3	2-10	4.21
Jeffamine D400	0-5	0.96
Defoamer	0.5-1.5	0.96
TiO ₂	2-10	4
Color pigment	0.15-5	0.15

B) Activator is polyisocyanate

The base component was prepared by dispersing the solid materials viz., pigments, fillers and other additives in polyols in high speed disperser (HSD) or twin shaft disperser and stabilizing the same with rest of the resin and additives.

The polyols, especially polyester polyol and some portion of castor oil were taken in the vessel followed by addition of defoamer under stirring. The mix was stirred at slow speed and subsequently the fillers like barium sulfate, silica, mica, molecular sieve, titanium dioxide and pigments were added in sequence under stirring. The speed of the HSD blade was adjusted such a way to ensure proper vortex formation. The solids were dispersed at a tip speed of about 3 to 8 meter per seconds till the finish was achieved. Once the finish was achieved, the composition was stabilized with remaining resin, polyols, amines and additives. Defoamer is used to take care of process generated air-entrapments. During processing, the composition was maintained below 60°C by circulating cooling water through the jacket of vessel. The composition was then filtered through filter and packed.

The second part of the composition, viz., the activator, was prepared by packing the solvent free polyisocyanate under nitrogen blanketing environment.

The polyurethane based coating composition was obtained by mixing 3 parts of the base with 1 part of the activator, by volume.

EXAMPLE 2:

The product as obtained by Example 1 was subjected to tests for various performance parameters such as specific gravity, flowability, tensile strength, elongation, flexibility, abrasion resistance etc., at the Corrosion Science and Engineering Laboratory of Indian Institute of Technology, Mumbai. Tables 2 and 3 show the details and results of the tests for various performance parameters respectively.

Table 2

S.No	Test and Test method	Description	Results and Significance
1	Direct Pull off Adhesion (D-4541)	Done to test the adhesion of the cured film on surface on which it is to be applied.	Excellent Adhesion. One of the critical parameter to ensure long term corrosion protection.
2	Tensile Strength (D-638)	Done to test strength of the cured film in case of expansion of the surface on which it is to be applied.	Excellent Tensile strength. Ensures the coating does not crack in case of surface expansion
3	Recoverable Elongations % (D-638)	Done to test flexibility of the cured film	Excellent. Ensures coating does not crack in case of surface expansion or contraction.
4	Surface Hardness, Shore Hardness (D-2240)	Done to test hardness of the cured film	Very Good. Ensures coating is hard enough once cured.
5	Water Vapour Permeability, 24 hrs (E-96/F-1249-90)	Done to test moisture permeability of the cured film	Excellent. One of the critical parameter to ensure long term corrosion & chemical protection
6	Resistance to Temperature 80°C Dry (30 Days) (Internal test Method)	The panels coated with cured coating film are kept in hot air oven at 80°C for 30 days. After 30 days, the film is observed for defects like cracking, delamination, flaking or corrosion	Very Good. Ensures the coating can withstand service temperature of 80°C.
7	Resistance to Temperature 80 °C Wet (30 Days) (Internal test Method)	The panels coated with cured coating film are dipped in hot water at 80°C for 30 days. After 30 days, the film is observed for defects like cracking, delamination, flaking or corrosion	Very Good. Ensures the coating can withstand service temperature of 80°C.
8	Water Absorption 30 Days (D-570)	Done to test water absorption by the cured Film in case of immersed application	Very Good. Lesser the absorption, better.
9	Abrasion Resistance H-10 Wheel, 1 kg load, 1000 Cycles (D-4060)	Done to test abrasion resistance of the cured film.	Excellent. Ensures less erosion in applications where abrasive conditions prevail.
10	Sea Water Resistance (30Days) (D-2240 and D-638)	Done to test resistance towards the Sea water	Excellent. Ensures excellent corrosion performance in corrosive environment.
11	Impact Resistance (BS-3900)	Done to test impact resistance of the cured film.	Excellent. Very critical for application where impact force is involved.
12	Flexibility 180° Bend (D-1737)	Done to test flexibility of the cured film, similar to elongation test	Excellent. Ensures coating does not crack in case of surface expansion.

Table 3

S.No	Test	ASTM	Results
1	Direct Pull off Adhesion	D-4541	14.65 N/mm ²
2	Tensile Strength	D-638	18.57 N/mm ²
3	Recoverable Elongations %	D-638	51.0%
4	Surface Hardness, Shore Hardness	D-2240	62
5	Water Vapour Permeability, 24 hrs	E-96/F-1249-90	0.3 gms/M ² /24 hrs
6	Resistance to Temperature 80°C Dry (30 Days)	Internal test Method	Passes
7	Resistance to Temperature 80 °C Wet (30 Days)	Internal test Method	Passes
8	Water Absorption 30 Days	D-570	Passes (wt. Variation less than 1%)
9	Abrasion Resistance H-10 Wheel, 1 kg load, 1000 Cycles	D-4060	13.7 mg
10	Sea Water Resistance (30Days)		
	A. % Weight Change		Passes (wt. Variation less than 0.5%)
	B. Hardness Change	D-2240	Passes (% Hardness change less than 3%)
	C. % Tensile strength change	D 638	Passes (% Change less than 03%)
11	Impact Resistance	BS-3900	Passes (27 Joules)
12	Flexibility 180° Bend	D-1737	Passes

As evident from the above table, the polyurethane based coating composition of the present invention exhibited excellent performance parameters that it can be very effectively applied to concrete and metal surfaces.

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EXAMPLE 3:

An ambient condition stability test for the urethane coating composition of the present invention was performed in order to ensure that the product is stable under recommended storage conditions.

Test Method:

10

The material was packed in standard pack size with standard packaging material and was stored under recommended storage conditions without opening it. The observations were taken at regular intervals for degree of settling or phase separation. The material was also tested for performance parameters. Samples were stored in sealed container at ambient conditions. The details of the test are shown in Table 4.

Table 4

Name of the Sample	Approximate exposure period	Rating	Observation
Cyclone Trial 1 (70 Lts, HSD, Vendor) - 20 Lt Can	6 months*	1	Phase separation, No settling, Easily miscible.
Cyclone Trial 2 (70 Lts, HSD, Vendor) - 20 Lt Can	6 months*	1	Phase separation, No settling, Easily miscible.
Cyclone Trial (500 Kgs, HSD, Vendor) - 200 lts Drum	7.5 months*	1	Phase separation, No settling, Easily miscible.
Cyclone Mfg Batch (500 Kgs, HSD, Vendor) - 200 lts Drum	11 months**	1	Phase separation, No settling, Easily miscible.

** Based on field feedback; *Based on Internal observations

Ratings are shown in Table 5.

Table 5

Rating	Description
0	No settling, No phase separation
1	Phase separation, No settling, Easily miscible
2	Phase separation, Soft settling, Easily dispersible
3	Phase separation, Soft to sticky settling, miscible with agitation
4	Phase separation, Sticky, Miscible with vigorous agitation
5	Hard settling, Cake formation, Not miscible or dispersible

5

EXAMPLE 4:

An accelerated condition stability test to study the anti settling property of the urethane coating composition of the present invention was performed. Accelerated stability study was conducted to predict the long-term stability in short period. In this field, 8 week accelerated stability translates in to over 1 year stability in actual storage conditions.

10

Test Method:

The material was packed in 200 ml or 500 ml cans, sealed and was kept at elevated temperature of 60°C for weeks. Four packs were kept for taking observations at the interval of 1 week, 2 week, 4 week and 8 weeks. The observations were taken for degree of settling or phase separation. The material was also tested for performance parameters. The experiment the details are shown in Table 6 and the observations are shown in Table 7. Ratings are as explained in Table 5.

15

Table 6

Experiments	Details
Exp 1	Standard Urethane Coating
Exp 2	Std Urethane Coating without M P Diol
Exp 3	Std Urethane Coating without Castor oil
Exp 4	Std Urethane Coating without both M P Diol and Castor Oil
Exp 5	Std Urethane Coating with 1,4 butane diol in place of M P Diol
Exp 6	Std Urethane Coating without Mica
Exp 7	Std Urethane Coating with higher filler loading (56.5% Vs 49.5% in Standard)

Duration: 72 days; Exposure- 8 weeks in 500 ml containers @ 60°C Incubator

Table 7

Sample	Initial Viscosity, cPs, 23 °C	End Viscosity, cPs, 23 °C	Rating
Exp 1	11940	11440	1
Exp 2	11480	12860	2
Exp 3	19900	22400	2
Exp 4	24600	29300	3
Exp 5	14400	12040	1+
Exp 6	9400	8840	2
Exp 7	37800	28330	2

From the results it may be noted that for the instant composition even with the low viscosity, anti-settling is excellent. MP Diol and castor oil are contributing towards the excellent anti-settling performance as is evident from Exp 1 to 4; viz., the moment castor oil and MP diol are removed the rating goes down to 3. As explained in Exp 5 when MP diol is replaced with 1,4 butane diol, still the anti-settling is very good though slightly lesser than Exp 1 which is having MP diol. So 1,4 butane diol also a good alternative diol which is useful in the invention. At last, as shown in Exp 7, this excellent anti-settling property and low viscosity of composition allows formulator to increase filler loading further (which helps in cost reduction). Even with 56.5% filler loading, anti-settling is very good.

The above example clearly shows that the various components of the coating composition synergistically interact to give rise to a coating composition with the desired superior properties. The field trials proves the better stability and desired lower viscosity of the composition. Hence the present composition is a good energy efficient composition with low viscosity.

The field trials also highlighted the following advantages.

- The product was applied by plural feed spray equipment and, in field, product could be applied without heating at ambient conditions of 35 - 40°C temperature.

- Film thickness of 1000 microns could be built up without sagging with multi-pass wet on wet manual spray application.
- No settling observed in the field in 200 Lt drum after 11 months, phase separation though observed, it was easily miscible.

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EXAMPLE 5

Chemical Resistance Test

The instant composition was tested to determine the resistance of the coating towards various chemicals in case of chemical splash, spillage or complete immersion.

Test Method:

10

Standard panels were coated with the coating composition, edge sealed and were dipped in the chemicals at 23°C and 50% humidity conditions. The coating film was observed at regular intervals for any defects like blistering, delamination, cracking or peeling. The coating was also tested for adhesion and hardness.

15

The instant coating composition was observed to have excellent chemical resistance against chemicals such as ammonium hydroxide 25% (v/v), citric acid 5% (v/v), methanol, sodium carbonate, sodium hydroxide, glycerine, sulphuric acid 30% (v/v), NaCl, HCl, diethylene glycol, acetonitrile, iso propyl alcohol diesel, lubricating oil and urea solution.

EXAMPLE 6

Water vapor permeability Test

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The instant composition was tested for permeability towards water vapors following standard ASTM E96.

Test Method:

25

The testing was done with Water method. Distilled water was filled in standard payne permeability cup and the cured film of the coating, which was of same dimension as the cup, was put on the cup opening. The edges were sealed with suitable sealant. The assembly was then kept under controlled temperature and humidity conditions. The temperature was maintained at 23 +/- 1°C and the relative humidity was maintained at 50 +/- 2 %.

30

Loss of weight resulting from the vapor transmission through the film was calculated every 24 hrs and plotted against the time. Once the steady state was achieved, the permeability was calculated. Results of the test are shown in the below. Table 8 shows the details of the test with the standard coating composition of the invention and Figure 1 shows its graphical representation.

Table 9

Date	Hrs	Weight (gm)	Wt loss (gm)	Cumulative Wt loss (gm)
19.01.11		62.2894		
20.01.11	24	62.2256	0.0638	0.0638
21.01.11	48	62.2157	0.0099	0.0737
22.01.11	72	62.2045	0.0112	0.0849
23.01.11	96	62.186	0.0185	0.0134
24.01.11	120	62.1758	0.0102	0.1136
25.01.11	144	62.1648	0.011	0.1246
26.01.11	168	62.1575	0.0073	0.1319
27.01.11	192	62.1502	0.0073	0.1392
28.01.11	216	62.1439	0.0063	0.1455
29.01.11	240	62.0972	0.0467	0.1922
30.01.11	264	62.0917	0.0055	0.1977
31.01.11	288	62.0854	0.0063	0.2040
01.02.11	312	62.0809	0.0045	0.2085
02.02.11	336	62.0750	0.0059	0.2144
03.02.11	360	62.0694	0.0056	0.22
04.02.11	384	62.0637	0.0057	0.2257
05.02.11	408	62.0577	0.0060	0.2317
06.02.11	432	62.0524	0.0053	0.237
07.02.11	456	62.0472	0.0052	0.2422
08.02.11	480	62.0424	0.0048	0.247
09.02.11	504	62.0367	0.0057	0.2527
10.02.11	528	62.0298	0.0069	0.2596
11.02.11	552	62.0245	0.0053	0.2649
12.02.11	576	62.0189	0.0056	0.2705
13.02.11	600	62.0147	0.0042	0.2747
14.02.11	624	62.0097	0.0050	0.2797

In figure 2:

Y2 0.2797

Y1 0.2422

5 X2 624

X1 456

Water Vapour Permeability (Rate of Permeation per sq. meter area) = 5.357143 gm/24hrs/Sq meter.

10 The data above shows that the instant composition has a superior effect with respect to the impermeability improvement, which is contributed by mica.

WE CLAIM:

1. A polyurethane based coating composition comprising a base and an activator wherein the base component comprises
- 5 a polyol,
a filler,
a catalyst,
a defoamer and
a molecular sieve;
and the activator comprises polyisocyanate.
- 10 2. The polyurethane based coating composition as claimed in claim 1 wherein the base and the activator are present in a ratio of 3:1 v/v.
3. The polyurethane based coating composition as claimed in claim 1 and 2 wherein the base optionally comprises a pigment, additional polyols, amines and chain extender.
- 15 4. The polyurethane based coating composition as claimed in claim 1 to 3 wherein
the polyol is present in an amount of 35 to 75%,
the filler is present in an amount of 20 to 60%,
the catalyst is present in an amount of 0 to 5%,
the defoamer is present in an amount of 0.5 to 1.5 % and
the molecular sieve is present in an amount of 2 to 10 %.
- 20 5. The polyurethane based coating composition as claimed in claim 1 to 4 wherein
the trifunctional polyol is present in an amount of 0.5 to 5 %,
2-methyl 1,3 dipropane diol is present in an amount of 1 to 20%
mica is present in an amount of 3 to 25%
catalyst is present in an amount of 0.1 to 5%
25 the defoamer is present in an amount of 0.5 to 1.5 %, and
the molecular sieve is present in an amount of 2 to 10 %.
- 30 6. The polyurethane based coating composition as claimed in claim 1 to 5 wherein
2-methyl 1,3 dipropane diol is present in an amount of 1 to 10%,
barium sulphate is present in an amount of 5 to 40%,
silica is present in an amount of 5 to 40%,
chain extender is present in an amount of 0 to 5 % ,

titanium dioxide is present in an amount of 2 to 10% and
colour pigment is present in an amount of 0.15 to 5%.

7. The polyurethane based coating composition as claimed in claim 1 to 6 wherein the polyol comprises a diol and/or a triol.

5 8. The polyurethane based coating composition as claimed in claim 1 to 7 wherein the polyol comprises natural oil polyols or saturated aliphatic polyester polyols.

9. The polyurethane based coating composition as claimed in claim 1 to 8 wherein the natural oil polyol is derived from soybean oil, castor oil, palm oil or canola oil.

10 10. The polyurethane based coating composition as claimed in claim 1 to 9 wherein the natural oil polyol is a castor polyol.

11. The polyurethane based coating composition as claimed in claim 1 to 10 wherein the diol is a linear diol or a branched diol.

12. The polyurethane based coating composition as claimed in claim 1 to 11 wherein the diol is an aliphatic diol or an aromatic diol.

15 13. The polyurethane based coating composition as claimed in claim 1 to 12 wherein the polyol comprises 2-methyl 1,3 dipropane diol.

14. The polyurethane based coating composition as claimed in claim 1 to 13 wherein the polyol comprises 1,4 butane diol.

20 15. The polyurethane based coating composition as claimed in claim 1 to 14 wherein the polyol is polypropylene ether polyol.

16. The polyurethane based coating composition as claimed in claim 1 to 15 wherein the catalyst is diethyl toluenediamine

17. The polyurethane based coating composition as claimed in claim 1 to 15 wherein the catalyst is absent from the composition.

25 18. The polyurethane based coating composition as claimed in claim 1 to 17 wherein the defoamer is a silicon free defoamer.

19. The polyurethane based coating composition as claimed in claim 1 to 18 wherein the filler comprises one or more of substances selected from mica, barium sulphate and silica.
20. The polyurethane based coating composition as claimed in claim 1 to 19 wherein the filler comprises mica.
- 5 21. The polyurethane based coating composition as claimed in claim 1 to 20 wherein mica functions as an extender.
22. The polyurethane based coating composition as claimed in claim 1 to 21 wherein the mica is coated with titanium dioxide (TiO₂) or carbon black
- 10 23. The polyurethane based coating composition as claimed in claim 1 to 22 wherein the molecular sieve is crystalline aluminosilicate.
24. The polyurethane based coating composition as claimed in claim 1 to 23 wherein the molecular sieve has a pore openings of about 3 Å.
25. The polyurethane based coating composition as claimed in claim 1 to 24 wherein the chain extender is a polyether amine.
- 15 26. The polyurethane based coating composition as claimed in claim 1 to 25 wherein it the pigment comprises synthetic iron oxides.
27. A process for preparing the polyurethane based coating composition as claimed in claim 1 wherein the process comprises the steps of
- 20 a) preparing the base component by dispersing the solid materials like pigments, fillers and other additives in polyols in high speed disperser (HSD) or twin shaft disperser and stabilizing the same with rest of the polyols and additives;
- b) preparing the activator and
- c) mixing the base with the activator.
- 25 28. The process as claimed in claim 26 wherein it comprises the steps of mixing the polyol with the defoamer under stirring at slow speed, adding other additives like barium sulfate, silica, mica, molecular sieve, titanium dioxide and pigments in sequence under stirring, wherein the speed of the HSD blade is adjusted to tip speed of about 3 to 8 meter per seconds, stabilizing the composition with remaining resin, polyols, amines and additives, preparing the activator by packing the solvent

free polyisocyanate under nitrogen blanketing environment and by mixing 3 parts of the base with 1 part of the activator, by volume.

29. An article coated with the polyurethane based coating composition as claimed in claim 1 to
- 26.

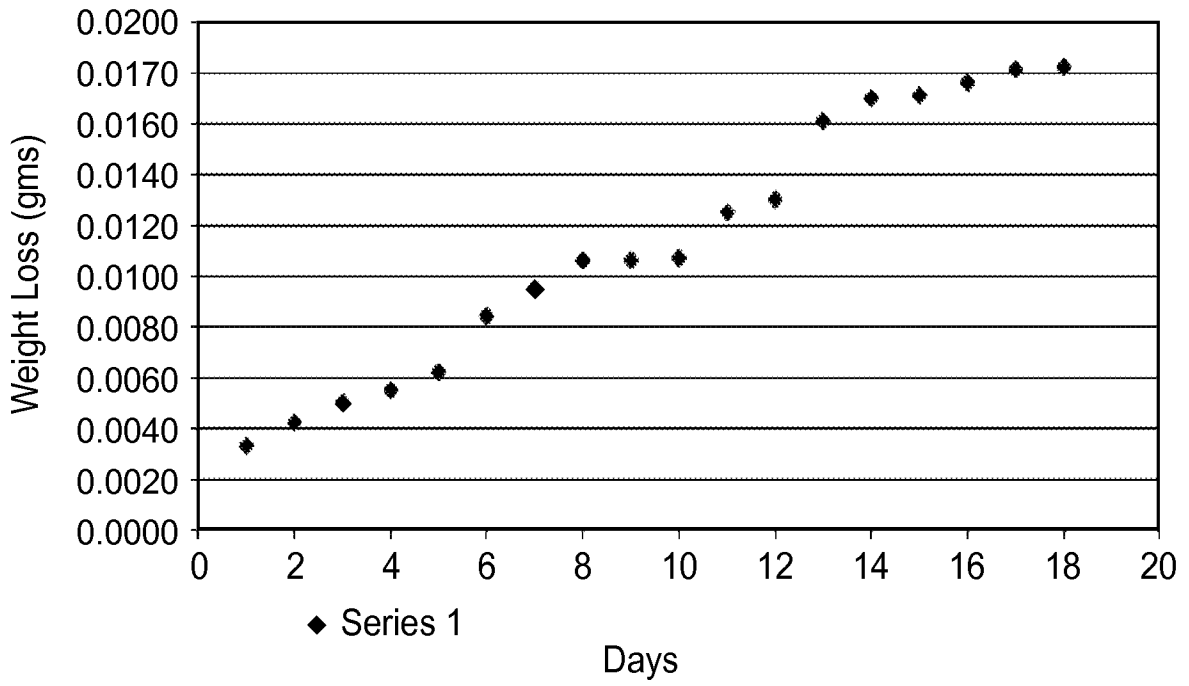


FIG. 1

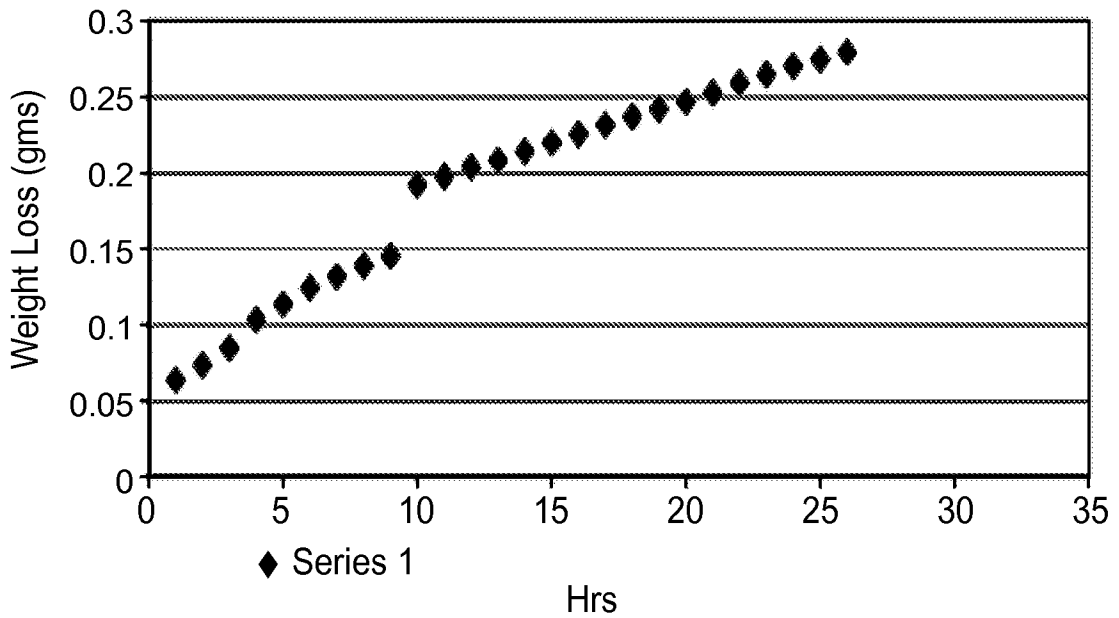


FIG. 2