ISSN 2229-5518

Synthesis and Application of Amino-Modified Silicone Oil on Cotton Fabric

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Abstract— The synthesis of amino-modified silicone oil carried out with dimethyl cyclosiloxane (DMC) and N-(2-aminoethyl)-3-aminopropyl methyldimethoxysilane (KH-602) as raw materials and potassium hydroxide (KOH) as a catalyst and 1,3-Diethenyl-1,1,3,3-tetramethyl disiloxane as a cured agent. The amino-modified silicone oil has been synthesized by bulk polymerization. The chemical structure has been characterized by IR spectra. The amino value measured by chemical titration and the value was 0.71mmol/g. The viscosity measured by NDJ-8S viscosity meter and the viscosity was 445 mpa.s. Factor impacting on technical indicators during preparation which were polymerization temperature, polymerization time, the amount of N-(2-aminoethyl)-3-aminopropyl methyldimethoxy silane, the amount of catalyst and the cured agent investigated then came to a full process recipe. The amino-modified silicone oil emulsified and the tested results has been studied. The results showed that the ionic surfactant of amino-modified silicone emulsion was nonionic, the thermal stability was good at 90°C, the dielectric stability was good at 90°C, the alkali stability was good at 80°C, the solid content was 25%, the viscosity was 60 mpa.s, and the p^H was 6-7. Then the amino-modifies silicone oil emulsion applied on the cotton fabrics and the performance studied accordingly. The results showed that the rate of hand feel properties (softness & smoothness) of the finished cotton knit fabric was 5 and cotton woven was 4.5 on the scale of 1-8, the hydrophilic property of finished cotton fabric was good, on the whiteness property of finished cotton fabrics had little effect but had no effect on the shade of dyed finished fabric, had little effect on the strength of finished fabric, and the feel retention rate was 60% after several times wash, which indicated that the amino-modified silicone oil had good wash fastness property.

Index Terms— Synthesis, Amino-Modified Silicone Oil, Emulsification, Functional Silicon, Wash Fastness, Cotton Fabric, Application,

1 Introduction

Silicone oil is a manufactured chemical which has been used in many industries. It is liquid polymerized siloxane with organic side chains. The most vital member is polydimethylsiloxane. As a result of their lubricating properties and high thermal stability these are very important materials in textile commercial sector [1-5]. The polymer backbone consists of alternating silicon-oxygen atoms (...Si-O-Si-O-Si…) – i.e. siloxane – rather than carbon atoms (...C-C-C-C...). And other species attach with the tetravalent silicon atoms, not with the divalent oxygen atoms which are fully responsible to forming the siloxane chain. A typical example is hexamethyldisiloxane, where three methyl groups attach with each silicon atom to form $Si(CH_3)_3OSi(CH_3)_3$ [6-13]. The carbon analogue would be an alkyl alkane, that is (2, 2, 4, 4-tetramethylpentane) $C(CH_3)_3CH_2C(CH_3)_3$. Silicone oil was first discovered in the 1930^s, then after ten years later it earned the name of silicone and began to use in many commercial applications. It is a valuable product because it deals with some high quality properties in textile industry.

Silicone oil is generally non-volatile, non-toxic, odorless, colorless or light yellow liquid. Silicone oil does not dissolve in water, methanol, methyl ethyl ketone, glycol, dimethyl ether ethoxyethanol, kerosene, with benzene or carbon tetrachloride, but slightly soluble in ethanol, acetone, butanol and dioxane [14-20]. It has a small vapor pressure, high flash point and ignition point, a low freezing point. As the number of different segments, the viscosity increases as the molecular weight increases, this solid silicone oil may have a variety of viscosities, from 0.65 centistokes until million centistokes [7-9]. To obtain a low vis-

ISSN 2229-5518

cosity silicone oil is prepared, acid clay can be used as a catalyst, and telomerization at a temperature of 180℃, or sulfuric acid as a catalyst, at low temperatures telomerization, high viscosity silicone oil or a viscous material with an alkaline catalyst [11-19]. Silicone oil has outstanding weather resistance, heat resistance, hydrophobicity, physiologically inert, electrical insulation and smaller surface tension, also has a low viscosity-temperature coefficient, high compression resistance, some varieties also have resistance to radiation performance.

2 Synthesis Of Amino-Modified Silicone Oil

2.1 Raw Materials

Dimethyl cyclosiloxane (DMC, 98%) and N-(2-aminoethyl)-3-aminopropyl methyldimethoxy silane (KH-602, 98%) were used as reactants, those were purchased from Zhejiang Runhe Organo silicone New Material Co., Ltd (Deqing, Zhejiang, China). Potassium hydroxide (KOH) was used as a catalyst, which was purchased from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China), and 1, 3-Diethenyl-1, 1, 3, 3-tetramethyl disiloxane was used as a cured agent, which was purchased from Zhejiang Runhe Organosilicone New Material Co., Ltd (Deqing, Zhejiang, China).

2.2 Instruments

Three necked flask, Thermometer, Stirring machine, High temperature oil bath heater more than 150^oC, Vacuum device, p^H meter, NDJ-8S viscosity meter for viscosity test, Dryer for solid content test.

2.3 Preparation of Amino-Modified Silicone Oil

At first setup the three necked flask with a mechanical stirrer, a high temperature oil bath heater, a thermometer and a vacuum device. The preparation of amino-modified silicone oil was carried out as follows- the mixture of the N-(2-aminoethyl)-3-aminopropyl methyldimethoxy silane (0.24mol of 50g), distilled water (0.61mol of 10.98g), and dimethylcyclosiloxane (1.68mol of 500g) were heated in the three-necked flask at 90˚C-95°C for 60min, and then added cured agent $C_8H_{18}OSi_2$ (1, 3-diethenyl-1, 1, 3, 3-tetramethyldisiloxane) into the flask and stirred & heated at 90˚C-95°C for 10min. After that, the temperature was increased to 110^oC, then the catalyst (KOH) was put into the flask, then the mixture was stirred & heated for 3-4 hours and the flask was kept airtight. Finally, the remained transparent and glutinous fluid of N-(2-aminoethyl)-3-aminopropyl methyldimethoxy silane and low boiling residues that means the micro molecules from the reaction was eliminated by the vacuum distillation at 120^oC and kept it for 2 hours. The amino value which was estimated by chemical titration, solid content and viscosity of prepared oil, and the p^H of prepared amino-modified silicone oil were tested.

2.4 Emulsification of Amino-Modified Silicone Oil

Lutensol OP-10 is a nonionic surfactant and soluble in water which is made up of 10-mole ethylene oxide adduct of octylphenol. In this research Lutensol OP-10 was used as emulsifier, dispersant, wetting agent, detergent in formulating cleaning products and synthetic latex stabilizers. It is also usually used as main emulsifiers for vinyl and acrylic emulsion polymerization and for asphalt emulsion methods. **Table 1**, shows the required quantity used to emulsify the silicon oil. Lutensol OP (octylphenol ethoxylates) are chemically effective and can stable over a wide range of p^H and in electrolyte solutions. The HLB value of Op-10 is 14.

Materials	Quantity
Amino-modified silicone oil	50 gm
Nonionic surfactant Op-10	25 gm
Acetic acid	1.875 gm
Water	173.125 gm

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Table 1: 30% Emulsification of Amino-Modified Silicone Oil

Required amount of amino-modified silicone oil and emulsifier was taken in a plastic beaker and mixed together for 5min at 500 rpm. After 5min added little water (3-5) g in the solution and mixed for 10min at 1000 rpm. Then added much water (8-10) g and mixed for 10min at 1000 rpm. After 10 min added acetic acid 1g and mixed for 20min at 2000 rpm and after 20min again added more water (10-15) g and mixed for 10 min at 2000 rpm. Then again added (10-15) g water and mixed for 5min at 2000 rpm. After 5min added rest of the acetic acid in the solution and mixed for 10min at 2000 rpm. Finally, added rest of water accordingly and mixed at 2000 rpm till to make fully homogeneous solution.

3 Sample Preparation and Characterization

3.1 Determination of Amonia Value

2-3 gm of amino-modified silicone oil was taken into a conical flask, then added 20mL toluene and 30mL isopropyl alcohol into that flask and mixed together, then added 2-3 drops bromophenol blue indicator into the solution and mixed to make blue color solution for titration. Finally, added 0.1 mol/L of concentrated hydrochloric acid drop by drop till to change the blue color solution to a light yellow color solution.

Amino calculated value AV = (V_2-V_1) c/m. Where,

A_V— amonia value, m— amount of the sample, V_2— amount of consumption of HCl acid standard solution, V_1— amount of blank standard solution of HCl acid consumption, C— concentration of the standard HCl acid solution.

3.2 Solid Content

Little amount of sample was taken into a small beaker. Then dried the sample in a dryer at 105°C for 3 hours. Finally, took the weight of the dried sample and calculated.

3.3 Viscosity Test

The viscosity of the obtainted dispersion was measured by NDJ-8S viscosity meter.

3.4 Various Stability of Silicone Oil Emulsion

Thermal Stability- 50g/l Amino-Modified Silicone oil emulsion was taken into a beaker and placed the beaker in the heat bath and heated, then raised the temperature each and after 5min. Finally, tested three time like that and recorded the changes of the solution appearance at different temperature.

Alkali Stability- 50 g/l Amino-Modified Silicone oil emulsion and 0.8g of 30% NaOH for solution p^H= (±1)12 was taken into a beaker, and placed the beakers in the heat bath and heated, then raised the temperature each and after 20min. Finally, tested three time like that and recorded the changes of solution appearance at different temperature.

Dielectric Stability- 50 g/l Amino-Modified Silicone oil emulsion and 5 g/l Na_2SO_4 anhydrous was taken into a beaker and placed the beakers in the heat bath and heating, then raised the temperature each and after 20min. Finally, tested three time like that and recorded the changes of solution appearance at different temperature.

3.5 Ionic Surfactant Testing of Oil Emulsion

Anionic or Nonionic Test- 10ml methylene blue, 10ml chloroform ($CHCl_3$) & 5ml amino-modified silicone oil emulsion was take into the test tube, then mixed together. Then after few minute observed the color of layer. If the $CHCl_3$ layer has been turned blue it referred to the test sample was anionic or if the water has been became turbid, milky it referred to the sample was nonionic.

Cationic Test- 1ml bromophenol blue and 5ml test sample was taken into the test tube and mixed together and observed the color of solution. The solution color was blue that means the ionic surfactant of tested sample was cationic.

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3.6 Particle Size and Their distribution

The particle size and its distribution of the obtained dispersion were measured by laser particle size analyzer.

4 Results and Discussion

4.1 The Structure and Reaction of Silicon Oil Polymer

The synthesis process of N-(2-aminoethyl)-3 aminopropyl methyldimethoxysilane has shown in the **Fig.1**, this figure illustrates the hydrolysis reaction of N-(2-aminoethyl)-3-aminopropyl methyldimetho ysilane and the ring-opening polymerization of dimethylcyclosiloxane. During the reaction, Si-OH condensation polymerization was produced by the polymer (A). Polymer (A) contains the units of chain of $(C_3H_6NHC_2H_4NH_2)$ (CH_3) Si-O and $(CH_3)_2$Si-O or OH group from dimethylcyclosiloxane and Si-OH on the other end of chain. **Fig.2,** shows the addition reaction of the $C_8H_{18}OSi_2$ (1,3-Diethenyl-1,1,3,3 tetramethyldisiloxane) group separate and react with the Si-OH groups of the both side of the polymers (A) and create a bond in the presence of catalyst potassium hydroxide at the high temperature.

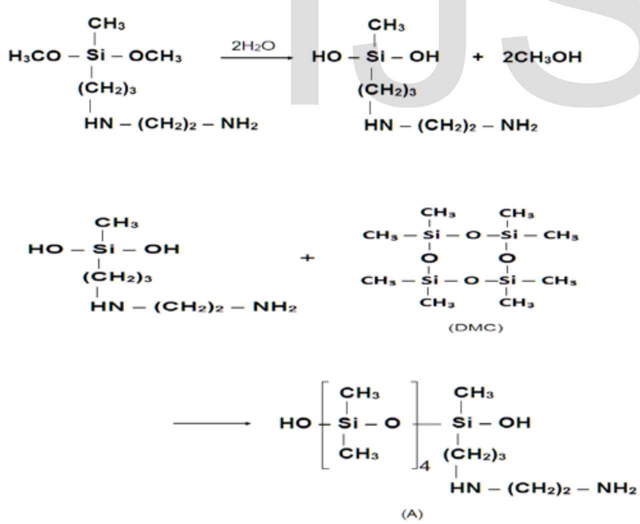

Fig.1 Synthesis of N-(2-aminoethyl)-3-aminopropyl polydimethylsiloxane.

Due to this polymerization reaction the viscosity of the polymer was increased. The final product was amino-modified silicone oil polymer (B). Due to the polymerization reaction some low boiling micro molecules like CH_3OH and H_2O was remained in the reaction. After that these micro molecule was eliminated from the reaction by the vacuum distillation at 120^oC.

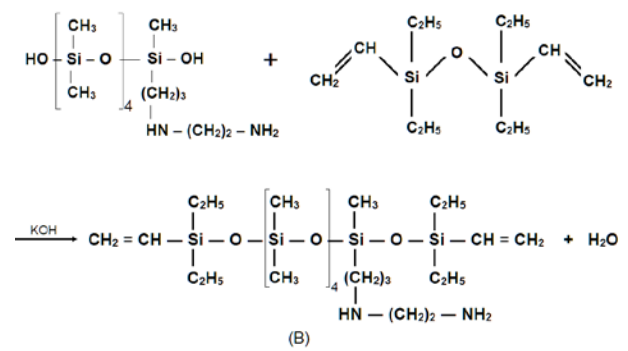

Fig.2 Addition reaction of 1, 3-Diethenyl-1, 1, 3, 3-tetramethyldisiloxane to N-(2-aminoethyl)-3-aminopropyl polydimethylsiloxane.

4.2 Effects of Temperature on Amonia Value

From the **Fig.3**, it can be seen that the polymerization temperature has slight effect on amino value. Due to the unstable pressure, the amino value was not stable (0.71-0.75) mmol/g, which may be caused by the removing of the low boiling micro molecule CH_3OH and H_2O from the reaction by the vacuum device.

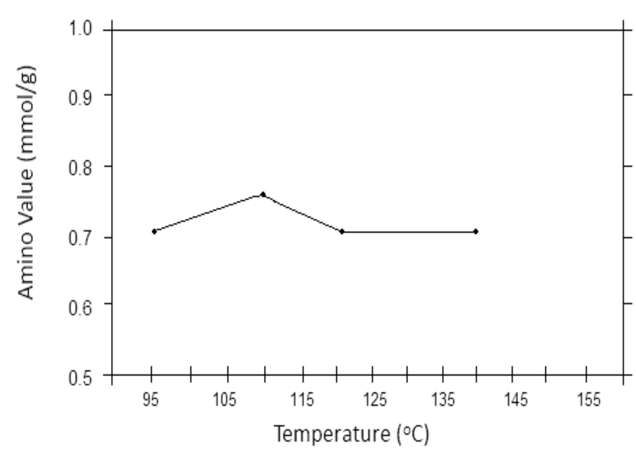

Fig.3 Effect of temp.on the value of amino

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4.3 Dynamic Viscosity Analysis on Different Temp.

The dynamic viscosity of the liquid for the silicon oil was measured by NDJ-8S viscosity meter. **Fig.4**, shows when the polymerization temperature was 110˚C, the polymer viscosity increased as the temperature increased, after charging KOH in the reaction. The viscosity remain steady while the temperature was over 120˚C. The high viscosity, clear and transparent product was obtained after removing the micro molecule under the vacuum device at 120˚C. The ideal polymerization temperature was 110˚C.

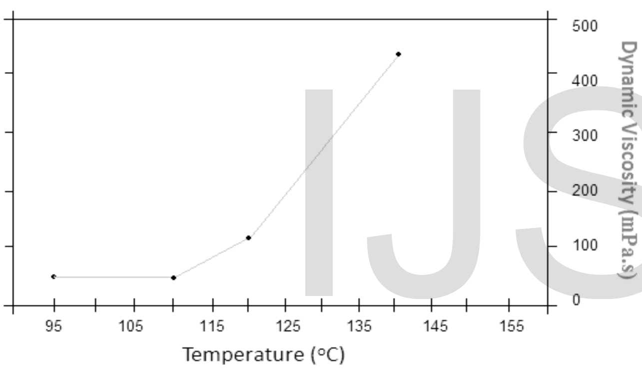

Fig.4 Effects on the dynamic viscosity of polymerization for different temperature

4.4 Effects of Polymerization Time on Amonia Value

Fig.5, shows that the effects on the amino value of polymerization time (60 min, 70 min, 90 min, 270 min, and 390 min). From that, it can be seen that the polymerization time has no effect on the amino value. After increased polymerization time, the amino value of the polymer retains relatively the same. The value was (0.71-0.75) mmol/g.

4.5 Effects of Time on Dynamic Viscosity

Fig.6, clearly shows that the effects on the dynamic viscosity of the polymers for polymerization time (60min, 70min, 90min, 270min, and 390min). From the **Fig.6**, it can be seen that the viscosity of the polymers increased as the extending polymerization time increased. It can be clarified that when the catalyst remained in the reaction the viscosity increased with the extending polymerization time, and when the catalyst reaction was completed the polymer retained same viscosity with the increasing of polymerization time.

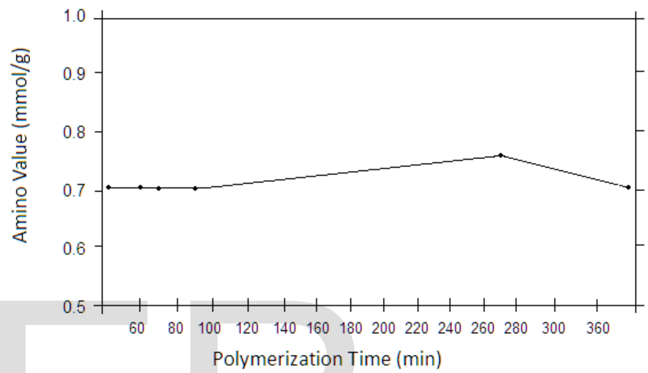

Fig.5 Effects of amino value on different time

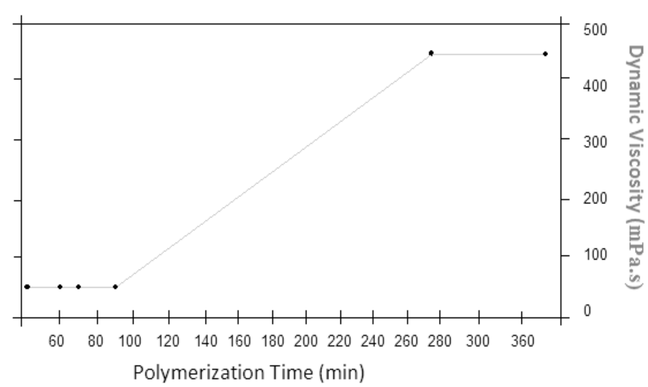

Fig.6 Effects of time on dynamic viscosity

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4.6 The Structure of Silicon Oil Polymer

The chain structure of silicon oil polymer was confirmed by FT-IR analysis. **Fig.7**, shows the FTIR of the polymer. In this figure, the absorption peak of 2962.90 cm^{-1} is attributed to a C–H expanding vibration; the absorption peak of 1019.64~1095.81 cm^{-1} is attributed to the Si–O–Si stretching vibration; 1412.50 cm^{-1} and 1260.90 cm^{-1} consists of a main absorption peak $–Si(CH_3)_2–$ groups $Si–CH_3$ and the inner surface and the outer surface of the bending vibration caused by the absorption peak at 801.47 cm^{-1} stretching vibration attributed to the Si–C, and the inner surface of the rocking $-CH_3$, absorption peak at 702.21 cm^{-1} attributed to $–Si(CH_3)_3$ groups $Si–CH_3$ stretching vibration. Among 1019.64~1095.81 cm^{-1} absorption peak is the most characteristic strong absorption band, when the molecular chain length is too long, divided into two intensity peaks nearby. This indicates that the product is methyl mesh polysiloxane compound.

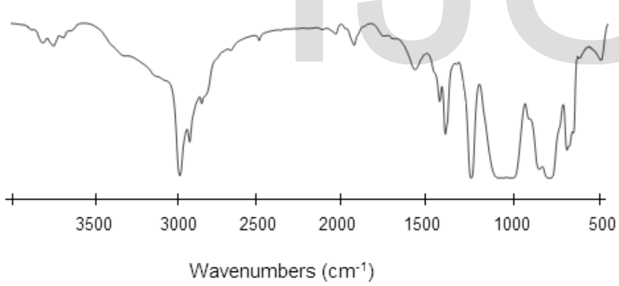

Fig.7 FT-IR spectra of amino silicone oil

4.7 Solid Content and Amonia Value

In this research study the solid content of oil and emulsion was calculated and the value of solid content was as respectively 91% and 25%. And it confirmed, the value was standard comparing with other products. Amonia value of silicon oil calculated and the value was 0.71nmol/g.

4.8 Stability Test Observation of Emulsion

From **Table 2**, we observed that the thermal stability, alkali stability and dielectric stability of the silicon oil emulsion are good in condition in different temperature and there is no any types of huge changes on precipitation and transpiration upto temperature 90˚C.

Stability	50˚c / 60˚c	70˚c / 80˚c	90^{0}c
Thermal	No change	No change	No change
Alkali	No change	Semi trans.	Sediment
Dielectric	No change	Semi trans.	Sediment

Table 2: Different Types of Stability

5 Applications of Emulsion on Fabric

5.1 Hand Feeling of the Fabric

In this study, two methods were used to determine the hand feel properties of amino silicon oil emulsion on cotton fabric. One method was padding and another one was exhaust method. And the softness test based on a scale of 1 to 8, where 8 is the excellent softest and 1 is the harshest. **Padding method**- 50g/l Amino-modified silicone oil emulsion was taken into a beaker and co trolled the p^H (5-6) of the solution by using acetic acid. Then took about 5g of 100% cotton knit and woven fabrics and immersed the both fabrics in the upper solution. Then passed the both fabrics one by one through the padding mangle with a wet pickup of 70%-80%. And then dried the fabrics at 150^{o}C for 5 min and then cooled for 30 min. Finally, checked the hand feeling of both fabrics and the result has shown in the **Table 3**, from that table it can be seen that the treated fabrics softness has increased, that means the amino modified silicone oil emulsion has good softness and smoothness properties.

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Table 3: The result of softness rating (padding method)

Fabrics	Softness Rating
Treated knit fabric	5
Untreated knit fabric	1.5
Treated woven fabric	4.5
Untreated woven fabric	1

Exhaust method- required amount of emulsion, acid, water and fabrics was taken into a conical flask. And set the flask in the dye bath. Then started the machine for 30 min at 50˚C. After 30min dried the fabrics at 150˚C for 5 min and then cooled for 30 min. Finally, checked the hand feeling of treated fabrics and result has shown in the **Table 4**. From the table it can be seen that the treated fabrics softness has increased, that means the amino-modified silicone oil emulsion has good softness and smoothness properties. Because the amino group of amino-modified silicone micro emulsion contain cationic charges (-NH_3+) which have strong attraction for the negatively charged materials. On the other hand cotton based fabrics which contain anionic charges on their surface. So, during treating cotton fabric with amino silicone emulsion it creates bond with each other into fiber and make the fabric more soft and smooth.

Fabrics	Softness Rating
Treated knit fabric	4.5
Untreated knit fabric	1.5
Treated woven fabric	4
Untreated woven fabric	1

Table 4: The result of softness rating (exhaust method)

5.2 Hydrophilicity of the Fabric

50 g/l Amino-modified silicone oil emulsion was taken in a beaker. And then took about 5g of knit fabric and then immersed the fabric in the solution. Then passed the fabric through the padding mangle with a wet pickup of 70-80%. And then dried the fabric at 150^{o}C for 1min and then cooled for 30 min. Finally, checked absorption of treated fabrics using water drop put on the treated fabrics and recorded the absorption time (**Fig-8**).

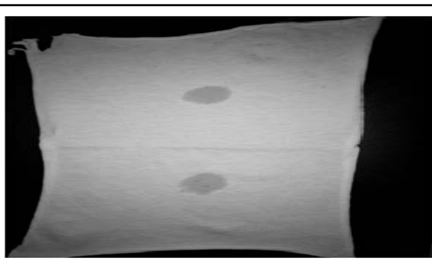

Fig.8 Treated fabric absorbency

The treated fabrics absorbed the water drop within 10 sec that means the treated fabric has good absorption property that means the amino-modified silicone oil has good hydrophilic property. Because during emulsification of silicone oil with high levels of surfactants it creates macro emulsion. So, during application the macro emulsion creates a layer on the fabrics surface as a result the treated fabric cannot be absorb water quickly. On the other hand the emulsification of amino-modified silicone oil creates micro emulsion and during the application with cotton fabric the micro emulsion goes into the fiber and creates bond with the fiber because of their alternative ionic charge as a result the treated fabrics can be absorb water quickly.

5.3 Whiteness of the Fabric

50 g/l Amino-modified silicone oil emulsion was taken in a beaker. And then took about 5g of woven fabric and merged the fabric in the solution. Then passed the fabric through the padding mangle with a wet pickup of 70-80%. Then dried the fabric at 105^{o}C for 5min and then cooled 30 min. And then tested the treated fabric whiteness by using data color machine and the result was recorded. Then again dried the treated fabric at 140^{o}C for 1min, at 160^{o}C for 1 min, and at 180^{o}C for 1min then checked the white-

ness again and the result was recorded. Finally, tested the whiteness of the treated fabric three times like that and the recorded results has shown in the **Table 5**, from that table, it can be seen that the oil emulsion has little effect on the whiteness properties of treated fabric at high temperature.

Test	Whiteness (Raw)	Treated Fabric Whiteness at Different Drying Temperature			
		105^0C 5min.	140^0C 1min	160^0C 1min	180^0C 1min
1ST	70.94%	69.9%	66.2%	63.5%	60.74%
2ND	70.9%	69.5%	67.1%	64.2%	61.5%
3RD	70.92%	69.6%	66.9%	63.9%	61%

Table 5: Treated fabric whiteness % at different temp.

5.4 Dyed Fabric Shade Change Analysis

In this work at first, dyed the cotton woven fabric with reactive red dyes. Required amount of dye, salt, soda, water and fabrics was taken into a conical flask. Set the conical flask in the dye bath. Then started the dye bath for 60min at 60°C. After 60min washed the dyed fabric by using 2 g/l soaping agent for 10min at 90°C. Then after 10min washed the dyed fabric with cold water and dried the dyed fabric in a dryer at 105°C for 5 min. Finally, checked the K/S value of dyed fabric by using data color machine and the result has shown in the **Table 6**. Then in the second stage, treated the dyed cotton woven fabric with amino-modified silicone oil emulsion. 50 g/l amino-modified silicone oil emulsion was taken in a beaker. Then took about 5g of dyed fabric and then immersed the fabric in the solution. And then passed the fabric through the padding mangle with a wet pickup of 70-80%.Then dried the treated fabric at 105°C for 5min and then cooled for 30min. Finally, checked the treated fabric shade by using data color machine and the result has shown in the **Table 6**. From the **Table 6**, it can be seen that the amino modified silicone oil emulsion has slight effect on the shade of the treated dyed fabrics.

Fabrics	K/S Value
Untreated dyed fabric	20.318
Treated dyed fabric	20.434

Table 6: K/S value of untreated and treated fabrics

5.5 Fabric Strength Observation

50 g/l oil emulsion was taken in a beaker. Then took about 5g of woven fabric and immersed the fabric in the upper solution. And then passed the fabric through the padding mangle with a wet pickup of 70-80%. Then dried the fabric at 150˚C for 1min and then cooled 30min. Finally, checked the strength of treated and untreated fabrics by using YG (b) 026E electronic fabric strength tester and the results has shown in the **Table 7**. Here it can be seen that, the amino-modified silicone oil emulsion has little effect on treated fabric strength properties compare to the untreated fabric strength properties.

Fabrics	Breaking Strength	Elongation
Untreated	152 n	10.28 %
Treated	121 n	9.06 %

Table 7: Breaking strength and elongation %

5.6 Durability Test of the Fabric

Treated the Cotton knit and woven fabrics with the amino-modified silicone oil emulsion. From the **Table 8**, it can be seen that after three times wash the fabrics has loosed only 30% of softness property compare to before wash that means the amino-modified silicone oil has good wash fastness property.

Treated Fabrics	Softness Rating			
	Before wash	1st wash	2nd wash	3rd wash
Knit Fabric	5	4.5	4	3.5
Woven Fabric	4.5	4	3.5	3

Table 8: The results of softness rate after three time wash

6 Conclusions

The hydrolyte of N-(2-aminoethyl)-3-amino propylmethyldimethoxylsilane yields with the reaction with dimethylcyclo siloxane (DMC), the polymer containing N-(2-amino ethyl)-3-amino propyl side chain attached with the hydroxyl terminated groups and polysiloxane chain. The amino value of amino modified silicone oil depends on the ratio of N-(2-aminoethyl)-3-amino propylmethyldimethoxylsilane and dimethylcyclo siloxane and the ratio was 10% {N-(2-aminoethyl)-3-amino propylmethyldimethoxylsilane dimethylcyclo siloxane} and the amino value was 0.71 mmol/g. The viscosity of the amino-modified silicone oil depends on the reactive conditions and the viscosity increased with the increasing of polymerization temperature or polymerization time and the amount of catalyst. The amount of catalyst was 0.01% of the weight of dimethylcyclosiloxane and the viscosity was 445 mpa.s. And the polymerization temperature was 120℃ and the total time was 6 hours.

The prepared amino-modified silicone oil was emulsified with emulsifier and the performance was studied. The ionic surfactant of amino-modified silicone oil emulsion depends on the emulsifier and the ionic surfactant of amino-modified silicone oil emulsion was cationic. The stability of amino-modified silicone oil emulsion was good at high temperature in alkali or in dielectric.

Then the prepared amino-modified silicone oil emulsion was applied on the cotton knit and woven fabrics and also on the dyed fabrics using different test method. The rate of softness for knit fabric was 5 and for the woven fabric was 4.5 on the scale of (1-8) it indicates that the amino silicone emulsion has good hand feel properties. And the water absorption time of treated knit fabric was 10sec it indicates that the amino silicone emulsion has good hydrophilic properties. After three times wash the durability rate was 60% it indicates that the amino silicone emulsion has good wash fastness properties. The amino silicone emulsion had little effect on whiteness property of treated fabrics after drying at 180^{o}C but had no effect on treated dyed fabric shade.

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