Synthesis of Silicone oil and Application on Knit and Woven Cotton Dyed Fabrics

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Abstract: Silicone oil have wide spread applications in textile dyeing and finishing. In this paper four different samples of Silicone oil softeners were synthesized on the basis of a new recipe with different chemical ratio and each of them used on dyed and white cotton fabrics of knit and woven. Several tests have been done on Silicone oil like ionic surfactant test, solid content test, stability test etc. Although, several tests were done on the Silicone oil treated dyed and white knit and woven fabrics. From the test result of fastness test, color strength test, tensile strength test, Whiteness test etc. it has come out that it increases the fabric softness, flexibility, absorbency, increase abrasion resistance, crease recovery, gives excellent fastness properties etc. It can endow the textiles with soft, fluffy style. Proper stability and durability can reduce the problem of the roller sticky, floating oil. It is colorless and transparent thick liquid and easy to dilute. It has the excellent affinity to all kinds of fabrics including cotton knit fabrics, woven cotton fabrics, jute etc. The overall results come out with great prospect for silicone oil "Sample C" in textile finishing.

Key words: Synthesis, Application, Silicone Oil, Cotton, Knit Dyed Fabrics, Woven Dyed Fabrics.

1. Introduction

Silicone oil is the most important and common chemical in textile processing. Silicone softeners mostly used to get better softness properties on textile fabric. It improves the abrasion resistance of fabrics, mobility of fibres, tear strength of fabrics, soiling resistance and static protection. It also decreases yarn and fabric tensile strength by reducing fibre cohesion, reduce sewing thread breakage, pilling and flammability [1]. Softener can be classified by (a) Cationic softener (b) Anionic softener (c) Nonionic softener (d) Amphoteric softener and (e) Silicone softener.

Cationic softeners have the best soft handle properties. It is a common application in exhaust methods. Cationic softener usually used in home laundry products. They have the good inherent affinity to all fibres. It is mainly used for color textiles. They have a tendency to yellow in comparison with non-ionic. **Anionic softeners** are used less due to less handle properties than cationic and nonionic. They don't have the affinity to all fibers. They are suitable for padding application not exhaust. It improves good lubrication, strong antistatic effects, good rewetting, good foaming agents etc. because of their anionic groups. **Nonionic softeners** are less used than anionic softeners. They have good dispersing agent, high lubricity. Nonionic softeners are stable to temperature and high pH conditions. They are generally poor foamers and don't yellow. **Amphoteric softeners** have high antistatic properties. They are very sensitive to skin and they have some ecological problem.

Silicone softener improve the sample to give a silk soft hand, very good lubricity, crease recovery, tear strength, abrasion resistance etc. Silicone softeners are more expensive than fatty softeners. It shows excellent durability and temperature stability [2], [3], [4]. Silicones are the most versatile polymer known. This chemical adds value to the fabric by transforming the fabric handle to match the customer perception. Silicones are the organo metallic polymers derived from the abundant raw material on earth, sand. Silicone is a generic term that refers to a class of manmade polymers based on a frame work of alternating silicon and oxygen (Siloxane Bonds) with organic substituents attached to the silicon [5]. Methyl groups are the most important organic substituents used in the commercial silicones. The Vast majority of which are Poly dimethyl Siloxanes. The following table shows the silicone modification

with different chemical compound and their derived properties (Table 1).

Table 1: Silicone Modifications

SILICONE MODIFICATIONS	PROPERTIES DERIVED
Amino Group	Highly exhaustible and durable softness
Hydrophilic Group	Water adsorptive
Methyl Group	Water repellence and antistatic finish
Hydrogen Group	Water repellence and soil resistance
Other Organo modifications	Drapery and wrinkle recovery property.

Their distinctive chemistry imparts a range of characteristics [6]. A variety of silicone technologies have application in the textile industry. They include, Polydimethylsiloxanes, Amido, Amino Functional Silicones, Methyl Hydrogen Silicones, and Epoxy functional Silicones, Hydroxy functional Silicones, Silicone Polyethers and Epoxy Polyether Silicones [7], [8].

Silicone softener has great influence on increasing the soft hand feel of cotton fabrics, silicone softener finishing in general imparts water repellent property to the textiles. Such water repellency is provided by methyl groups which are oriented and attached to the fibre surface by silicone links [9]. Creasing of the cotton fabrics depends on the structural properties of the fibres. Crease resistance is obtained by easy care finishing which stiffens the cotton fibres by covalent cross-linking and there for resistance to bending, creasing and recovery from deforming can be promoted [10], [11]. The process mainly consists of crosslinking of the cotton fibre hydroxyl groups with a crosslinking agent; the possibility of displacement of fibre chain molecules decreases. It has been demonstrated that the degree of crosslinking has a profound effect on the crease recovery angle and dimensional stability of cotton fabric [12], [13].

Silicone oils play a very significant role in textile finishing. Many important requirements like increasing the softness, hydrophobicity, whiteness, fastness and many other requirements coming from buyer could be served by applying the appropriate silicone softener. For aiming to achieve different properties on fabric different softener has been synthesized. The development of silicone softener is still going on. In the textile industries, silicones are used in all stages of the process, on the fiber during production, on the fabric and/or directly on the finished goods. Silicones are applied from different delivery systems to provide various benefits like lubrication, softening, foam control or hydrophobic coatings [14], [15].

2. Experimental section

2.1 Materials

2.1.1 Chemicals

DMC (Dimethyl Cyclo Siloxane)

KH-6O2 (N-(2-Aminoethyl) (3-aminopropyl) methyldimethoxysilane)

C8H18OSi2 (1, 3-Dimethyl-1, 1, 3, 3-tetra methyl disiloxane)

KOH (Potassium Hydroxide)

2.1.2 Softener Treated Fabrics

Knit Dyed Fabric: Single jersey (Dye Color- Violet)

Woven Dyed Fabric: Plain fabric (Dye Color- Yellow)

2.2 Methods

2.2.1 Process of Manufacturing of Silicon oil

Table 2: Recipe for synthesis of silicone softener

	Sample A	Sample B	Sample C	Sample D

Chemical Ratio	DMC: KH-6O2: C8H18OSi2: KOH = 500: (10-50): (2-10): 0.05	DMC: KH-6O2: C8H18OSi2: KOH = 500: (10-50): (2-10): 0.05	DMC: KH-6O2: C8H18OSi2: KOH = 500: (10-50): (2-10): 0.05	DMC: KH-6O2: C8H18OSi2: KOH = 500: (10-50): (2-10): 0.05
Actual Amount	DMC = 500 ml, KH-6O2 = 10 ml, C8H18OSi2 = 2 ml, KOH = 0.05 gm.	DMC = 500 ml, KH-6O2 = 25 ml, C8H18OSi2 = 4 ml, KOH = 0.05 gm.	DMC = 500 ml, KH-6O2 = 50 ml, C8H18OSi2 = 10 ml, KOH = 0.05 gm.	DMC = 500 ml, KH-6O2 = 30 ml, C8H18OSi2 = 6 ml, KOH = 0.05 gm.

2.2.2 Procedure

At first, materials were putted (DMC + KH-6O2) into a three necked flask. Then it was stirred and heated up to 90 OC -95OC and kept the temperature fixed for 1 hour. After 1 hour C8H18OSi2 is added into the flask and continued for 10 min. After that the temperature raised to 110 ºC and catalyst KOH is added. This temperature was maintained for 3-4 hours. Then the temperature reduced to 60 OC -70 OC. Then the micro molecular under the vacuum meter condition was cleaned before the temperature raised to 120 OC.

2.2.3 Emulsification

Silicone oil and emulsifier were mixed at 500 rpm for 5 minutes. Then added little amount of water (3-5 ml) into it and mixed for 10 minutes. Then added more water (8-10 ml). After that 1 gm. of acetic acid was added and stirred for 20 min. Again more water was added (8-10 ml) and stirred for 3 min. At last the rest of the water and acid were added.

2.3 Testing Procedures

2.3.1 Ionic Surfactant Test of Silicone Oil

During the test procedure the Surfactants and Water ratio was maintained as 10gm: 90gm

1. Anion or Nonionic Test:

10ml Methylene blue, 10ml Chloroform ($CHCl_3$) & 5ml test solution have been taken into the test tube, covered and shook hard. Let the tube standing & layering, observed the color of layer. If the $CHCl_3$ layer turns blue it referred to the test sample is anionic or if the water becomes turbid, milky it referred to the sample is nonionic.

2. Cationic Test:

I) Bromophenol 1ml, test solution 5ml taken and mixed properly. If it turns blue, sky blue or violet or between these, it was referred to Cationic surfactants.

II) 10ml Methylene blue, 10ml Chloroform ($CHCl_3$) was taken and added known drops of anionic surfactants, when the Chloroform ($CHCl_3$) layer turns blue then added 1% density test solution and shook and dropped; if the Chloroform ($CHCl_3$) layer turns to colorless or weak to the test solution then it was referred as Cationic surfactants.

2.3.2 Solid Content Test of Silicone Softener

Table 3: Solid content calculation of prepared each samples of Silicone Softener

Sample A	Sample B	Sample C
Paper weight = 1.45gm Weight of Silicone Softener with paper = 6.78gm After 3 hrs. drying weight = 2.56gm	Paper weight = 1.45gm Weight of Silicone Softener with paper = 7.97gm After 3 hrs. drying weight = 2.82gm	Paper weight = 1.45gm Weight of Silicone Softener with paper = 6.36gm After 3 hrs. drying weight = 2.53gm

$= \frac{2.58-1.45}{6.78-1.45} \times 100\% = 20.82\%$

$= \frac{2.82-1.45}{7.97-1.45} \times 100\% = 21.01\%$

$= \frac{2.52-1.45}{6.38-1.45} \times 100\% = 22\%$

Table 3 shows that, Silicone Softener C (Sample C), gives the heist solid contents and Silicone Softener (Sample A) gives the lowest percentage of solid content.

2.3.3 Color Fastness to Rubbing for Knit and Woven Fabrics

ISO–105×12 method has been used for measuring the rubbing fastness. In ISO-105-X12 the wet pickup percentage of the rubbing cloth is 100%. According to ISO–105×12 method, the number of rotation was set as 10 cycles in 10 seconds, for both wet and dry rubbing fastness. In wet rubbing, the rubbing cloth was wetted according to the test method and rating was given by comparing the Staining with the gray scale and similar procedure was adapted for dry rubbing.

2.3.4 Abrasion Resistance of Knit and Woven Dyed Fabrics

Abrasion is the physical destruction of fibres, yarns, and fabrics, resulting from the rubbing of a textile surface over another surface (Abdullah et al., 2006). Textile materials can be unserviceable because of several different factors and one of the most important causes is abrasion. Abrasion occurs during wearing, using, cleaning or washing process and this may distort the fabric, cause fibres or yarns to be pulled out or remove fibre ends from the surface (Hu, 2008; Kadolph, 2007). Abrasion ultimately results in the loss of performance characteristics, such as strength, but it also affects the appearance of the fabric (Collier & Epps, 1999)6. Abrasion resistance was measured in terms of the number of abrasion cycles required for failure with a 1 lb. load on a MARTINDALE abrasion tester (ASTM D 4966) [16], [17], [18].

2.3.5 Color Strength (K/S) of Knit and Woven Dyed Fabrics

Application of silicone softener on dyed fabric imparts significant changes in fabric properties. Color strength (K/S) is one of the important properties. In this experiment the Color strength (K/S) value has been taken before applying silicone softener and after the application of silicone softener for both knit and woven dyed fabrics, by using Spectrophotometer. After that the difference between two kinds of fabrics was calculated. The testing procedure for colorfastness to rubbing (both for wet rubbing and dry rubbing) was conducted with ISO–105×12 method [19], [20].

2.3.6 Tensile (breaking) Strength Test of Woven Dyed Fabrics

Fabric samples are clamped in the jaws of a tensile tester and pulled apart until they break. Three samples were tested across the warp and the average breaking strength established is expressed in Newtons. BS EN ISO 13934-2 test standard has been followed for measure the tensile properties of fabrics (grab test method).

3. Results and Discussion

3.1 Ionic Surfactant Test of Silicone Oil

Table 4: Ionic Surfactant Test of Silicone Oil

Samples	Silicone oil A	Silicone oil B	Silicone oil C	Silicone oil D
Particles	Weakly Cationic	Weakly Cationic	Weakly Cationic	Weakly Cationic

From (Table: 4) it can be seen that after anionic, nonionic and cationic test Silicone oil A, Silicone oil B, Silicone oil C and Silicone oil D all are weak

cationic because it's Chloroform ($CHCl_3$) layer turns to colorless.

3.2 Comparison among Amino Silicone Oil:

Procedure:

20% solution of every sample were made carefully [Liq. = (60%×1g)/20%=3-1=2g]= 1 : 2 (Sample : Water)

Dipped into 10g/l and padded, then dried on 150^0C for 1 min., at last Cooling down for 30 minutes.

Then the hand feel was checked (Table: 5).

Table 5: Combined Application

Combined product name & dosing	Silicone Oil C & Silicone Oil B [10g/l]	Silicone Oil B & Silicone Oil D	Silicone Oil C & Silicone Oil A [10g/l]	Silicone Oil A Silicone Oil D [10g/l]
Recipe	E-001A-50%+50 %	E-002A-50%+50 %	E-003A-50%+50 %	E-004A-50%+50 %
	E-001B-60%+40 %	E-002B-60%+40 %	E-003B-60%+40 %	E-004B-60%+40 %
	E-001C-80%+20 %	E-002C-80%+20 %	E-003C-80%+20 %	E-004C-80%+20 %
	E-001D-40%+60 %	E-002D-40%+60 %	E-003D-40%+60 %	E-004D-40%+60 %
Hand feeling	E-001A-Smooth & Soft	E-002A-Soft & fluffy	E-003A-soft	E-004A-Soft
	E-001B-Smooth	E-002B-Soft	E-003B-Soft	E-004B-Soft & smooth
	E-001C-Soft & fluffy	E-002C-soft	E-003C-Soft & Smooth	E-004C-Smooth & soft
	E-001D-Soft	E-002D-soft & smooth	E-003D-Smooth	E-004D-Soft & fluffy

Comparing hand feel among Silicone Oil A, Silicone Oil B, Silicone Oil C and Silicone Oil D it has been found that among all of them Silicone Oil C is the best. Individual products can be tested (with Silicone Oil C) and get the best result of amino silicone oil.

3.3 Fastness Test of Knit and Woven Dyed Fabrics

Table 6: Rubbing Fastness Test of Knit and Woven Fabrics

Samples	Dry fastness of knit fabric (grey scale grade)	Dry fastness of woven fabric (grey scale grade)	Wet fastness of knit fabric (grade)	Wet fastness of woven fabric (grey scale grade)
Silicone oil A	4-5	4	4-5	3
Silicone oil B	4	3-4	4	3-4
Silicone oil C	5	5	5	4-5
Silicone oil D	4-5	4	3-4	4

Here, in case of dry rubbing fastness value, Silicone oil C gave the best result (grey scale grading 5) for both knit and woven dyed fabrics (Table: 6) and in case of wet rubbing fastness value, Silicone softener C gave the best result (grey scale result 5) for knit dyed fabric and for woven dyed fabric it is 4-5 (Table: 6). These maximum values show sample treated with Silicone oil C gives the best result for both dry and wet rubbing fastness for both knit and woven fabrics.

3.4 Abrasion Resistance of Knit and Woven Dyed Fabrics

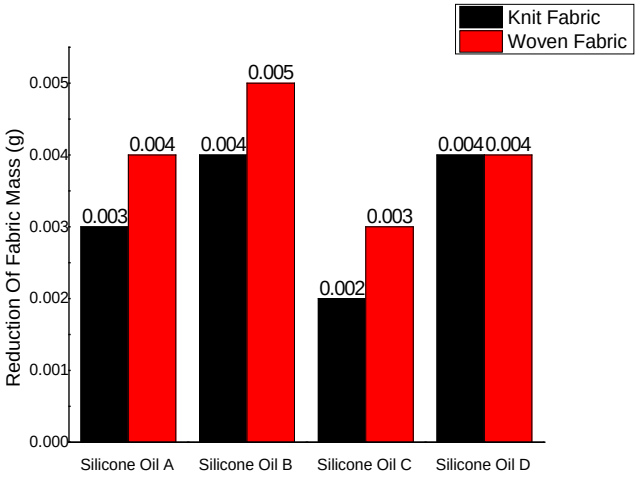

Fig 1: Abrasion Resistance for softener treated dyed fabrics (Knit & Woven).

Figure 1 gives, the test result for abrasion resistance value of the treated woven and knit fabrics with different silicone softener samples (sample A, sample B, sample C, sample D) and comparing among the samples it has come out that, Silicon Oil C treated dyed fabrics (both Knit & Woven) got the less reduction of fabric mass, which lead to the best resistant property among other.

3.5 Color Strength (K/S) of Knit and Woven Dyed Fabrics

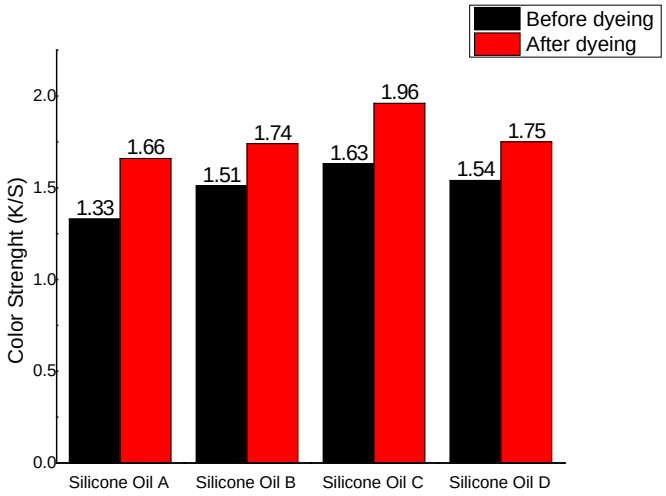

Fig 2: Change in color strength (K/S) for knit fabric.

Color strength is very important and sensitive for fabrics. Figure 2 gives, the color strength (K/S) value of the dyed fabric samples which have been treated with different samples (sample A, sample B, sample D) after dyeing. Comparing with other silicone softener treated samples, "Silicon Oil C" treated sample gives highest value in K/S and which indicates good depth in shade comparing to other treated samples.

3.6 Tensile Strength/ Elongation Test of Woven Dyed Fabrics

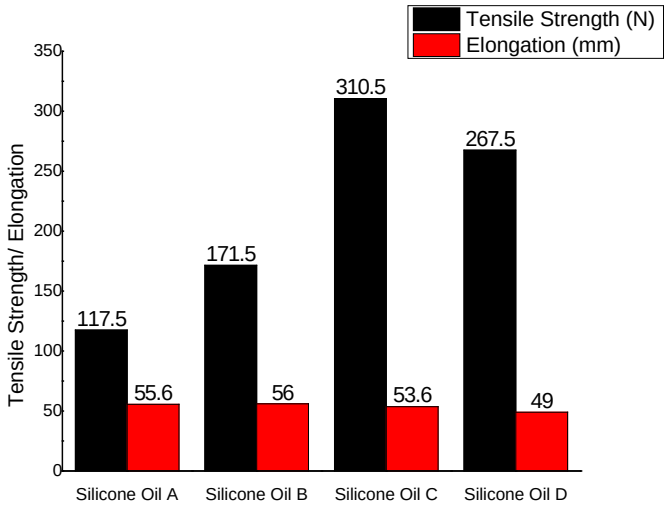

Fig 3: Tensile Strength/ Elongation Test of Woven Dyed Fabrics.

To improve the fabric quality must need to improve tensile strength of fabrics. Figure 3 gives the tensile strength of Woven dyed fabrics, treated with different samples of silicone softener (sample A, sample B, sample D) and comparing with other samples, Silicone oil C treated woven fabric gives highest tensile strength (310.5N/53.6mm) than other samples. These results show that Silicone oil C is best for woven fabrics.

3.7 Water Absorption Test of Knit and Woven Dyed fabrics

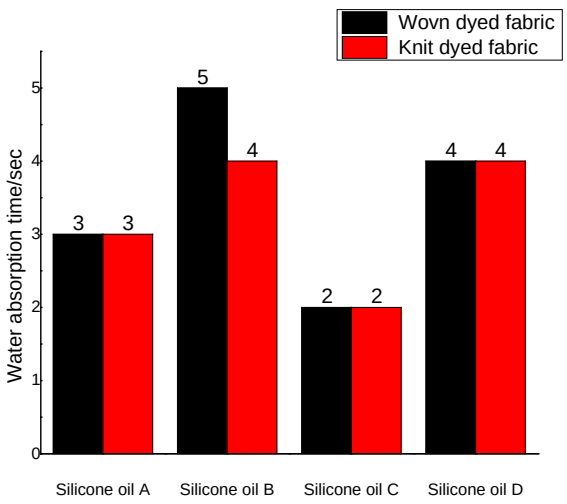

Fig 4: Water Absorption Test of Knit and Woven Dyed Fabrics

Take silicone oil 50g/l in a beaker. And then take about 5g knit fabric and 5gm woven fabric then immerse in the solution. Then pass the fabric through the padding mangle with a wet pickup of 70-80%. And then dry the fabric at 105^{o}C for 1min and cooling for 30 min. Finally, check absorption using water drop put on the treated fabrics and record the absorption time. From the figure easily understand that water absorption time is less at silicone oil C for both knit and woven fabrics.

4. Conclusions

In this paper four different samples of silicone softeners have been synthesized. To evaluate the property and to find out the best silicon oil many tests were done both on silicone oil and silicone oil treated dye fabric samples. Though there were slight differences among the test results for different samples but in most of the test shows that Silicone Oil C sample coming out with the expected results comparing with other samples. In case of color strength (K/S) test, the test result for Silicon Oil C shows greater difference in K/S value comparing with other samples. Since, Silicone Oil C came out with the best results from the test results, it is the best achievement in this paper.

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