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Original Research Article

The effect of pH and type of silicone on cotton and polyester finished fabrics

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ABSTRACT

A Characteristic often found in textile products is softness. The compound widely used as softener is silicone compound. The aim of this research is to determine the properties that will be obtained from the type of fabric being processed by comparing the use of silicon types, namely amino-propyl-functional polydimethyl siloxane (PDMS) and blocked amino silicone to each fabric. Firstly, research was conducted by varying the pH of the finishing process. Determined optimum pH will be followed by determining concentration of 15–60 g/L for each type silicone. The experiments was carried out on pad- dry- cure method. The tests carried out include fabric stiffness, tensile strength, resistance to repeated washing and heat, yellowing effect, Fabric Touch Tester and Water Contact Angle testing. It was found that there was no significant influence of pH on the use of amino-propyl-functional polydimethyl siloxane and blocked amino silicone compounds, so the next process was carried out at pH 7. Increasing the concentration of the softener will provide a better softening effect and optimum concentration at 45 g/L for both types of fabric used. Blocked amino silicone has better resistance to repeated washing compared to amino-propyl-functional polydimethyl siloxane. Heat testing shows that differences in molecular structure have no influence on both fabrics. Fabric processed using amino-propyl-functional polydimethyl siloxane provides a yellowing effect. Amino-propyl-functional polydimethyl siloxane provides good hydrophilicity. The softness value of blocked amino silicone is better performance on cotton fabric.

Introduction

Textile finishing provides the final result of commercial textile products such as appearance, luster, handle, softness, density, usability and so on (Choudhury, 2017). One type of chemical finishing that is often carried out is grip refinement or softening. Fabric softener for domestic purposes such as washing clothes at home or in the finishing process in the textile industry uses colloids which can provide softness to fabrics (Oikonomou et al., 2017). Softeners are categorized into cationic, anionic, and nonionic softeners depending on the nature of the functional groups in them (Javadi et al., 2013). Fabric softener is a colloidal dispersion that provides softness to fabrics. Fabric softeners are made from double-chain cationic surfactants assembled into multi- or uni-lamellar vesicles (Oikonomou et al., 2017). The main components of popular softeners are long chain alkyl type cationic surfactants and silicone (Igarashi and Nakamura, 2018). Silicone based softeners are the most commonly used in the fashion industry, due to their superior smoothing effect (Hassabo and Mohamed, 2019; Zhou et al., 2020). Silicone softeners can provide good softness, higher flexibility and elasticity, good softness, increase flexibility, and provide better smoothness than other types of softeners (Jatoi et al., 2015). The molecular structure of silicone can be seen in Fig-1.

In the textile industry, a wide variety of silicone products have been developed including polydimethylsiloxane, amino functional silicone, methyl hydrogen silicone, epoxy functional silicone, hydroxy silicone, polyether silicone, and polyether epoxy silicone (Mohamed et al., 2016). Silicon combines hydrophobic properties with a low glass transition temperature, elasticity, low surface tension, film formation and chain flexibility, as well as rotational freedom of the Si-O-Si linkages and low interaction energy between methyl groups which allows the atoms to obtain a minimal energy structure at interfaces with lower surface tension than organic polymers. This type of functional silicone depends on the molecular structure of PDMS (polydimethylsiloxane) and its modification by having functional groups to bond with the surface. In addition, amino or polyalkoxy has the same function as PDMS.

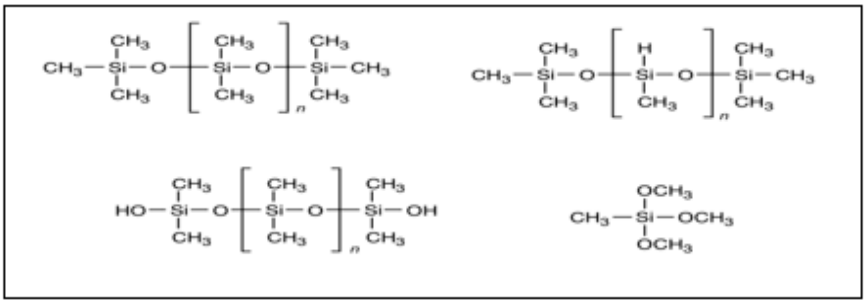


Fig-1. Structure of silicone molecules

Apart from PDMS compounds, amino silicones are widely used in textile applications to achieve permanent softness in textile fabrics. Polydimethylsiloxane compounds provide the ability to form hydrogen bonds and exhibit repulsive properties towards the outer surface of the fiber. The structure is intended to obtain a permanent final result due to the presence of silanol and/or silane groups. Silanol and silane react to form 3-dimensional cross-links that surround the fiber. These properties will provide a high water resistance effect at fairly low concentrations. However, its water repellency can decrease if the amount is excessive. This type is resistant to washing and soil repellent. This type of silicone can be applied using the exhaust or pad-dry-cure method (Hassabo and Mohamed, 2019). The softness of aminosilicon occurs at pH 5–5.5, because in the pH range softeners containing amino groups are maximally ionized (Choudhury, 2017). Cationic softeners, due to their ionic attraction, preferentially come out onto the fiber surface, forming a single layer and providing increased softness and anionic softeners do the opposite. So the hydrophilicity, softening effect is much lower than that of cationic softeners (Schindler and Hauser, 2004; Teli, 2015). The softening properties display increased adsorption and durability due to its cationic charge (Purohit and Somasundaran, 2014). There is no direct bond between the substrate and silicone, but the polymer network can cause strong adhesion with the substrate. –OH groups of cellulose and silicon atoms or amino groups, between quaternary ions and ionic dyes, and/or, due to Van der Waals forces, between silicon chains and cellulose chains (Teli, 2015). The presence of vesicles ensures the stability of the formulation, acting also as a means to mobilize surfactant bilayers and other water-insoluble additives such as essential oils (Saha et al., 2017). Recent studies have shown that vesicles have been shown to adsorb onto cellulose fibers (Kumar et al., 2017; Oikonomou et al., 2018; Mousseau et al., 2019). The presence of electrostatic forces will form a vesicular layer in water (Reimhult et al., 2003). Once dry, the vesicles may be converted into a bilayer of fibers that provides lubrication and softness properties (Igarashi et al., 2015; Igarashi et al., 2016).

Amino-functional groups linked to polydimethylsiloxanes enable an improved orientation and substantivity of the silicone on the substrate. This leads to an optimally soft handle and is often described by the term “super soft”. Amino silicones are by far the most extensively used functional silicones for textile finishing applications. These emulsions exhibit a positive surface charge and thus get attracted by the negatively charged fabric surface, leading to a strong sorption. Such favorable charge distribution facilitates superior molecular distribution and higher sorption at lower solution concentrations, leading to favorable process economics. Actual surface modification is caused by specific properties of the silicone molecules and the functional groups attached (Mahmood Zia et al., 2011).

In this study, we aim to further explore the application of Amino Propyl Polydimethyl Siloxane (PDMS) and blocked amino silicone on cotton and polyester fabrics. Our objective is to assess the effectiveness of these compounds in providing superior softness and other desired textile properties, contributing to advancements in the textile finishing industry.

Methods

Materials

The equipment used includes: Padder Machine, Infra Red Stenter Machine, Minolta CM-3600D Spectophotometer, Goniometer (Yakusa), Fabric Touch Tester (SDL Atlas) and glass equipment (Pyrex), which are commonly used in laboratories. Meanwhile, the materials used include: Cotton and Polyester fabric that has been previously washed, Na_2CO_3, teepol, CH_3COOH, Amino Propyl Polydimethyl Siloxane Compound (PT. Surya Tirta Kencana) and Blocked Amino Silicone Compound (PT. Jintex).

General Procedure

In this experiment, several stages of work were carried out, that are determine the optimum pH (varied at 4, 7 and 9) for each type of silicone softener. After the optimum pH was obtained, then continued by determining the optimum concentration with varied at 15 – 60 g/L. The experiment was carried out using two types of silicone, namely amino propyl polydimethyl siloxane and blocked amino silicone which will be applied to cotton and polyester fabrics. Each experimental variable will be applied using the pad-dry-cure method. For further evaluation of stiffness, tensile strength, repeated washing, TGA, FTT and WCA.

Determination of Optimum pH Conditions

Each silicone solution is prepared with a concentration of 30 g/l with pH variations of 3, 5, 7 and 9. The process continues with soaking the cotton and polyester fabrics for 5 minutes then the padding process is carried out, the drying process at a temperature of 100°C for 2 minutes and curing process at 160°C for 2 minutes. Then the tensile strength and softening were evaluated to get pH optimum.

Determination of Optimum Concentration

Each silicon finishing solution was prepared with varying concentrations of 15 g/l, 30 g/l, 45 g/l and 60 g/l at optimum pH. The process continues with soaking the cotton and polyester fabrics for 5 minutes then the padding process is carried out, the drying process at a temperature of 100°C for 2 minutes and the curing process at a temperature of 160°C for 2 minutes. Then the tensile strength and softening were tested to get optimum concentration.

Evaluation

Evaluations carried out in this experiment are softening and tensile strength test at optimum pH and concentration. Additional testing were carried out at optimum concentration are yellowing effect, Fabric Touch Tester, Water Contact Angle and resistance to washing at laboratory in the Polytechnic STTT Bandung and Thermal Gravimetry Analysis testing at the ITB Chemistry Laboratory.

Results and Discussion

Effect of pH

Fabric Stiffness

Fabric stiffness testing is a test carried out to see the results of the softening that occurs on the physical properties of the finished product on cotton and polyester fabrics using amino-propyl-functional polydimethyl siloxane and a modified silicone compound, namely blocked amino silicone. The smaller stiffness test value indicates that the fabric is softer. The results of tests on stiffness with pH variations on cotton and polyester fabrics can be seen in Fig-2.

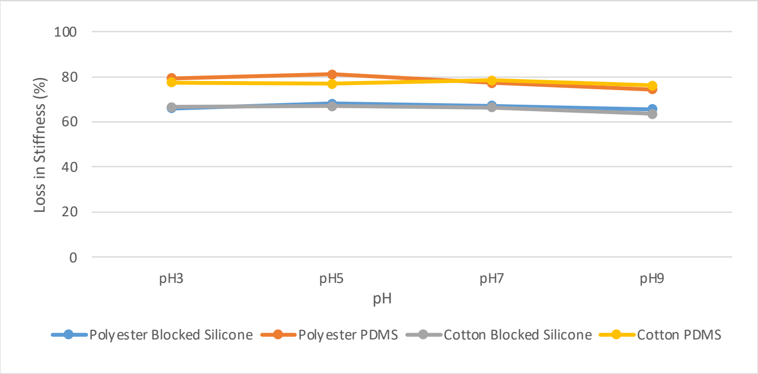
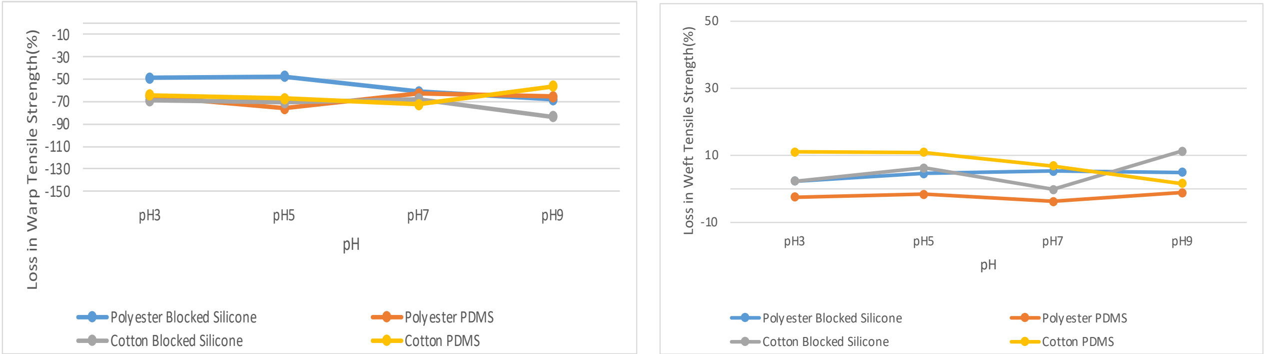


Fig-2. Effect of pH process on reduction in stiffness of polyester and cotton fabrics softened finishing process

Fig-2 shows that fabrics processed using amino-propyl-functional polydimethyl siloxane and blocked amino silicone are not affected by pH variations. Softeners at acidic pH and when dissolved in water, will ionize into a hydrophilic head with a negative charge and a hydrophobic tail carrying a positive charge (Teli, 2015). Therefore, cellulose fibers carrying a negative zeta potential attract positively charged softening species. In this study, pH conditions did not have a significant influence, this is possibly due to the resistance properties of the softener used being in the pH range 4-12.

Tensile Strength Test

Tensile strength testing was carried out to see the effect of using silicone compounds at various pH conditions on the tensile strength of the fabric. The results of tensile strength tests carried out on cotton and polyester fabrics can be seen in Fig-3. Based on the data, tensile strength is also not influenced by the use of pH process. This is proven by the data obtained which gives results that are not very significant. It is possible that the process pH used is still within the recommended process pH range. Cotton fiber shows that at acidic pH when using amino-propyl-functional polydimethyl siloxane there is a slight decrease in tensile strength. This is related to the nature of cotton fiber which is not resistant to acid.



(a) (b)

Fig-3. Effect of pH process on the percentage decrease in tensile strength of polyester and cotton fabrics softened finishing process. (a) Warp direction; (b) Weft direction

Determination of Optimum pH

The optimum pH is determined from stiffness and tensile strength values at various pH conditions, namely pH 3, 5, 7 and 9. Determination of the optimum pH is needed to be used in subsequent experiments. After testing with pH variations, the selected pH can be determined for the process that can be carried out. Because the stiffness and tensile strength results are not too affected by the pH of the process, for the next process a pH of 7 is used.

Effect of Silicon Compound Concentration

Stiffness Test

The results of stiffness testing with variations in concentration at optimum pH on cotton and polyester fabrics can be seen in Fig-4. Using amino-propyl-functional polydimethyl siloxane in cotton fibers, the more concentration used, the more likely the amine groups will bind to the reactive groups of the cotton fiber and the more siloxane groups there will be. The softening properties are obtained from the repetition of the siloxane compound, the number of repetitions of which cannot be ascertained. Likewise in polyester fibers, the higher the concentration, the more hydrophobic groups from the softener will interact with the hydrophobic fibers. This condition will provide coating properties in the gaps between fibers and threads so that the stiffness value of the fabric decreases. As research was condusted by Chattopadhay silicone nano emulsion have unique penetrability inside the fabric and fabric structure which result in the improvement in bending length and crease recovery (Chattopadhyay and Vyas, 2010). Based on the experimental results, the optimum concentration was obtained at a concentration of 45 g/l for polyester and cotton fibers.

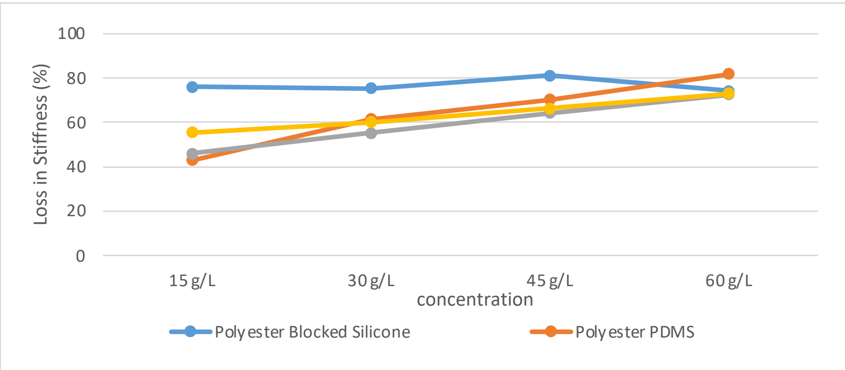
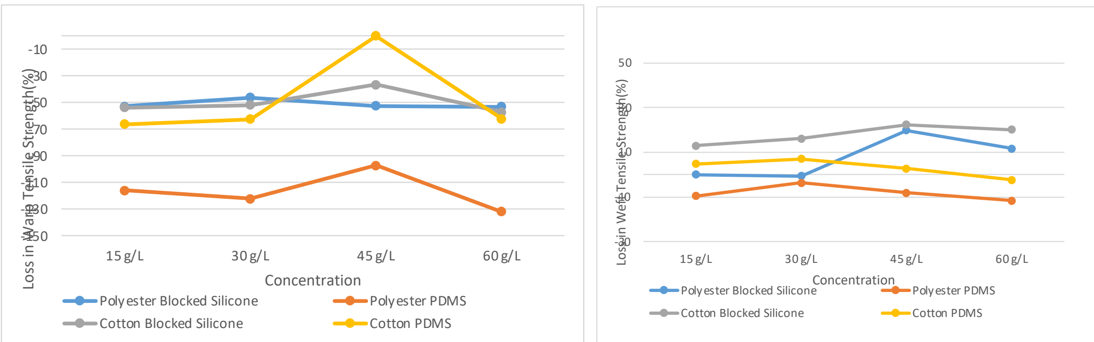


Fig-4. Effect of compound concentration on the percentage reduction in stiffness of polyester and cotton softened finishing process

Fabric Tensile Strength

Apart from stiffness parameters, tensile strength testing was also carried out to see to what extent the tensile strength would be influenced by the concentration of silicon compounds. Tensile strength test results data can be seen in Fig-5. The tensile strength test results show that it is not greatly influenced by the concentration of silicon compounds used in the process. The application of a softening emulsion can result in a loss of breaking load with an increase in elongation at break. Silicone emulsion will reduce friction between fibers in the yarn and between threads in the fabric resulting in more slippage, which can ultimately lead to a decrease in tensile strength with increased elongation. The more concentration used, the greater the tendency for slip to occur and cause a decrease in the tensile strength of the fabric same as previous research conducted by Chattopadhay (Chattopadhyay and Vyas, 2010).



(a) (b)

Fig-5. Effect of compound concentration on percentage of reduction in tensile strength of polyester and cotton fabrics softened finishing process. (a) Warp direction; (b) Weft direction

Influence of Silicone Type

The amino-propyl-functional compound polydimethyl siloxane is a compound that has hydrophobic properties with a low glass transition temperature, film formation and chain flexibility that comes from the rotational freedom of the Si-O-Si linkages and low interaction energy between the methyl groups, thus allowing the atoms to obtain minimal structural energy between surface and surface tension (Fig-6). This amino-propyl-functional polydimethyl siloxane compound can provide excellent softening properties to woven and knitted fabrics. It is feared that the presence of primary amine groups could have a yellowing effect when used when an oxidation reaction occurs. Blocked amino silicone is an organic silicon compound consisting of a silicon (Si) chain connected to an amino group (NH2) and a second group (-NH) with a low oxidation state during heating. This group functions to prevent reactions between the amino groups in the compound and other compounds during the process, thereby avoiding blockade in the fiber. The following is the structure of blocked amino silicone (Fig-7).

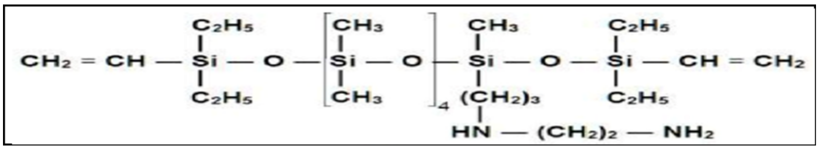


Fig-6. Chemical structure of amino-propyl-functional polydimethyl siloxane (Lahiri, 2018)

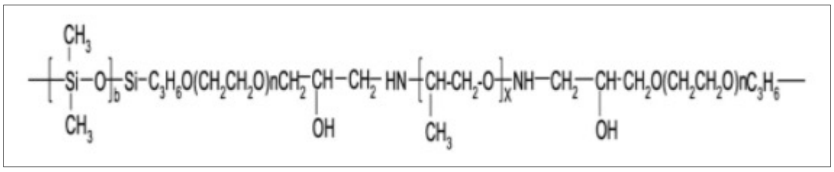


Fig-7. Chemical structure of blocked amino silicone (Jintex, 2022)

Fabric Stiffness

The research compared two types of silicone, namely the amino-propyl-functional polydimethyl siloxane (PDMS) compound and the blocked amino silicone compound by looking at the softening effect produced by each compound on cotton and polyester fabrics. The stiffness test results can be seen in Fig-8.

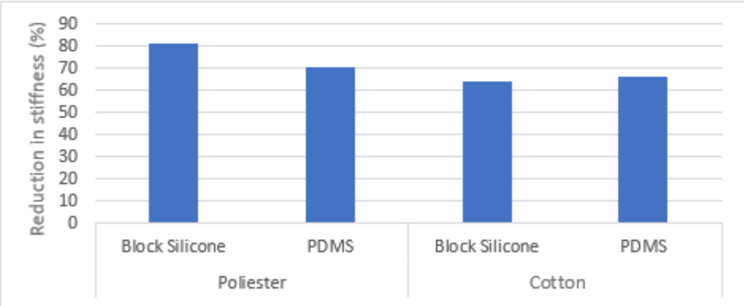


Fig-8. Influence of silicone type on the percentage reduction in stiffness of polyester and cotton fabrics softened finishing process

Fig-8 shows that amino-propyl-functional polydimethyl siloxane provides better softening properties compared to the blocked amino silicone compound. This is possible because the molecular mass is quite large compared to blocked amino silicone. However, it has not been clearly confirmed how many repetitions of siloxane monomers in amino-propyl-functional polydimethyl or blocked amino silicone provide softening. The reduced stiffness of cotton fabric occurs due to the bonds formed between the primary amine groups and the active part of the cotton fiber. In polyester fibers, the use of these two types of substances shows that the stiffness values obtained are not much different. In polyester fibers, the interactions that occur are determined by the hydrophobic part of the amino-propyl-functional polydimethyl which will bond with the hydrophobic part of the fiber.

The softening effect of using blocked amino silicone is optimum in polyester fiber compared to cotton fiber. This is possible because there are many hydrophobic parts that interact with hydrophobic polyester fibers. Cotton fiber will provide optimum softening properties when using amino-propyl-functional polydimethyl siloxane. It is possible that there are many amine groups that interact with the cotton fiber and many siloxane groups that provide softening properties.

Repeated Washing

The washing test was conducted by 5 wash cycle. This test was carried out to see the resistance of washing to the softening effect. The data can be seen in Table 1. In cotton and polyester fibers, the blocked amino silicone softener showed better resistance compared to amino-propyl-functional polydimethyl siloxane. This is caused by the molecular mass of blocked amino silicone being greater than amino-propyl-functional polydimethyl siloxane, so its resistance is better.

Table 1. Results of repeated washing tests on polyester and cotton fabrics processed using amino-propyl-functional polydimethyl siloxane compounds and blocked amino silicone

Fabric Item	Silicone type	Before Washing	After Washing	Loss of Stiffness (%)
Polyester	*Blocked Amino Silicone*	2.83	5.30	16.63
	Amino-Propyl-Functional Polydimethyl Siloxane	4.41	7.59	21.37
Cotton	*Blocked Amino Silicone*	5.71	7.64	12.07
	Amino-Propyl-Functional Polydimethyl Siloxane)	5.39	7.88	15.60

Note: Bending modulus of Blank cotton fabric ending modulus is 15.97 kg/cm3 and polyester is 14.90 kg/cm3

Yellowing Effect

The characteristic that is expected from this softening process, apart from the softening effect itself, is that there will be no yellowing effect after the drying process. Phenolic yellowing is a complex phenomenon and time consuming physicochemical process. This chemical reaction is a conjugate process of oxidation and nitration. The positive core of the amino group of the cationic softener has a negative impact on the white color of the index due to yellowing. Phenolic yellowing in textiles is caused by phenolic compounds present in the surrounding atmosphere. Butyl-hydroxytoluene (BHT) is the main phenolic compound that can react with nitrogen oxides (NOx) in the air and form nitrobenzene or quinone, which is a yellow chemical substance in slightly alkaline media. Neither nitrous oxide nor BHT alone causes a yellow color whereas when conjugated the product form will turn yellow (Sk et al., 2021). The phenolic reaction can be seen in Fig-9.

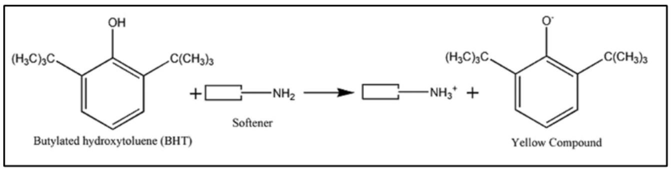


Fig-9. Phenolic yellowing mechanism (Sk et al., 2021)

Excessive drying temperatures can also contribute to the yellowing effect. Cotton fabric is dried and cured at a temperature of 120 - 140°C and polyester at a temperature of 170 -190°C. Higher temperatures or longer contact times can cause a yellowing effect. In the case of amino compounds the yellowing results mainly due to the oxidation of amino radicals in the presence of air and heat or light energy. More specifically the result of this oxidation is the formation of azo and azoxy compounds which ultimately impart yellowing to the fabric as reported by Mahmood Zia et al. (2011). The yellowing effect test was carried out using the spectrophotometric method and the test results can be seen in Table 2.

Table 2. Yellowing effects on polyester and cotton fabrics processed using amino-propyl-functional polydimethyl siloxane compounds and blocked amino silicone

Fabric Item	Type	YI D1925 (Yellow Index)
Poliester	*Blocked Amino Silicone*	-7.26
	Amino-Propyl-Functional Polydimethyl Siloxane	-9.65
Kapas	*Blocked Amino Silicone*	-7.80
	Amino-Propyl-Functional Polydimethyl Siloxane	-3.35

The data in Table 2 shows that fabric processed using amino-propyl-functional polydimethyl siloxane has a yellowing effect. This can be caused by the presence of a primary amine group in the amino-propyl-functional polydimethyl siloxane compound which has a higher oxidation level during the heating process. In the blocked amino silicone (Si) compound it is connected to an amino group (NH_2) and a second group (-NH) with a low oxidation level during heating. This group functions to prevent reactions between the amino groups in the compound and other compounds during the process so that resistance to yellowing is higher when compared to the amino-propyl-functional polydimethyl siloxane compound.

Thermo Gravimetry Analysis

Polyester fibers that have been processed with silicon compounds are characterized using TGA at a heating rate of 10°C/minute from a temperature of 25 – 800°C (Portella et al., 2016). Cotton fibers that have been processed with silicon compounds are characterized using TGA at a heating rate of 100C/minute from a temperature of 37 – 600°C (Abidi et al., 2010). The results can be seen in figure 3.6. Cotton and polyester will thermally decompose to produce gas and solid charcoal. Based on research conducted by Neumeyer et al. (1976), it was reported that in nitrogen, cotton fabric experienced a weight loss of between 270 - 370 x with the maximum weight loss, O-15 mg/minute-mg occurring at 346°C. Thermal decomposition of 100% polyester occurs in the range of 335-470°C, with a peak rate of weight loss, 0.1 mg/min-mg measured at 416°C In atmospheric air, both volatile gases and solid char as products of pyrolysis during the combustion process. The maximum weight loss rate for cotton increased to 0.25 mg/min-mg and occurred at 317°C. The maximum polyester decomposition rate remains the same in both air and nitrogen, but the temperature drops to 405°C (Neumeyer et al., 1976). The TGA test results in Fig-10 show that the softening enhancement treatment with various types of silicone does not have a significant effect on the thermal properties of the fabric.

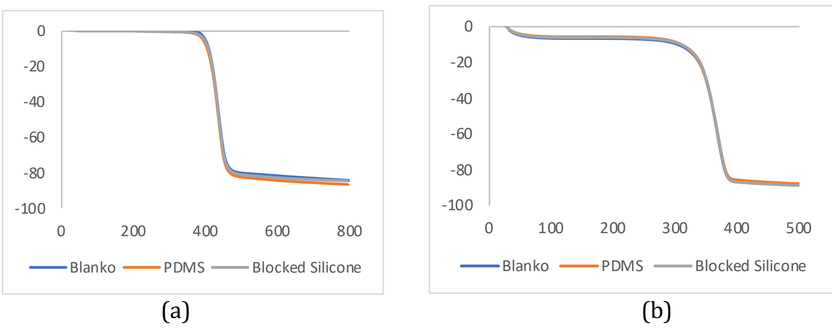


Fig-10. Effect of weight reduction due to the heating process on polyester fabric that has been processed with silicone compounds. (a) Cotton and (b) Polyester

Hidrophilicity

The wettability of a surface depends on two factors: (1) its chemical composition and (2) its structure (roughness). Organofunctional polysiloxanes with hydroxyl, epoxy or amino groups in their structure have been observed to be able to react with functional groups present on the fiber surface or allow cross-linking to occur (Przybylak et al., 2016). The results of the contact angle test can be seen in Fig-11.

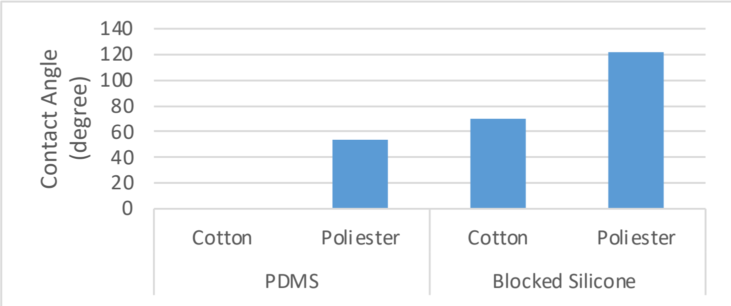


Fig-11. Contact angle values for cotton and polyester fabrics finished with various types of silicone

Cotton and polyester provide more hydrophobic properties when reacted with amino blocked silicone compounds. This is due to the polymerization of the siloxane compound with more types of silicon, thereby increasing its water resistance properties.

Fabric Handling

Fabric grip testing is one way to assess the physical features of fabric, namely its smoothness and softness. The higher the index value, the smoother and softer the fabric will be. Based on the results of the fabric handle testing that has been carried out, the results obtained can be seen in Fig-12.

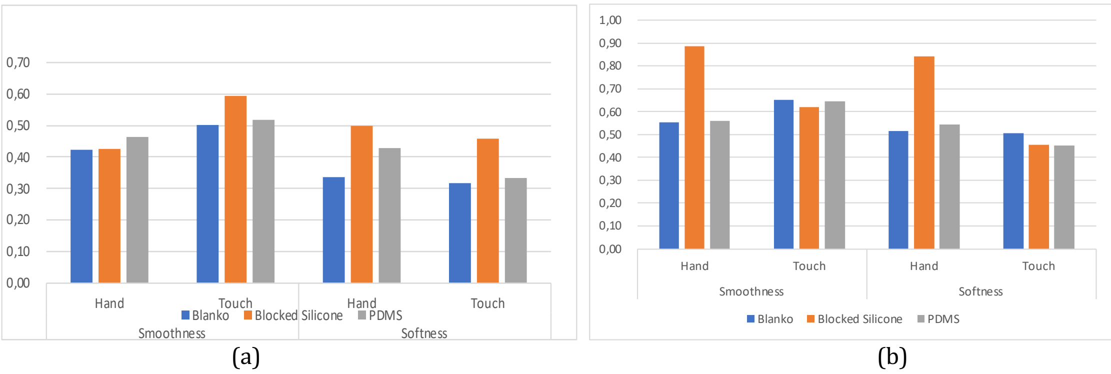


Fig-12. graph of smoothness and softness values for cotton (a) and polyester (b) fabrics that have been finished with various types of silicone

Based on the data in Fig-12, it can be seen that the process of using blocked amino silicone compounds on polyester fabrics provides the best smoothness and softness results compared to other silicone compounds. however, cotton fabric is no different from other compounds. this is caused by a fairly strong hydrophobic bond formed between the polyester fiber and the silicon amino blocked compound or its density by polymerization of the silicone softener compound which is higher compared to other silicone compounds. As reported by Islam, et,al, that treated fabrics softness has increased, that means the amino modified silicone oil emulsion has good softness and smoothness properties (Lahiri, 2018).

Conclusion

Based on the data and analysis of the test results that have been carried out, it can be concluded that the pH parameter of the process does not have any influence on softening and tensile strength when using the two types of softener used. The optimum concentration was obtained at 45 g/L for all types of silicone compounds used. The use of blocked amino silicone compounds as a softener provides better washing resistance compared to amino-propyl-functional polydimethyl siloxane. In cotton fiber, the use of the amino-propyl-functional compound polydimethyl siloxane tends to produce a yellowing effect when heated at high temperatures. The thermal properties of the fabric are not affected by the use of these two types of silicone compounds in cotton and polyester fabrics. The blocked amino silicone compound provides higher hydrophobicity properties on cotton and polyester fabrics than the amino-propyl-functional polydimethyl siloxane compound, while the best smoothness and softness properties are obtained when used on polyester fabrics.

Conflict of Interests

The author(s) declares that there is no conflict of interest in this research and manuscript.

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