A COMPARISON OF THE INFLUENCE OF CATHOLYTE VS PHOSPHATE DETERGENT ON THE MECHANICAL PROPERTIES OF POLYAMIDE 6,6 WOVEN FABRIC

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OPSOMMING

Die elektrochemiese aktivering van vloeistowwe is 'n relatiewe nuwe tegnologie. Die alkaliese gedeelte van die geaktiveerde vloeistof (*catholyte*) beskik oor goeie skoonmaakvermoë. *Catholyte* kan 'n omgewingsvriendelike alternatief wees tot tradisionele fosfaatbevattende detergente. Daar is egter baie min bekend oor die invloed van *catholyte* op tekstielstowwe soos poliamied 6,6 het. Die fokus van hierdie studie was dus om die invloed van *catholyte* op sekere eienskappe van poliamied 6,6 te evalueer. Die resultate toon dat die verlies aan treksterkte veroorsaak deur catholyte nie 'n noemenswaardige verskil toon teenoor die verlies in treksterkte wat die detergent veroorsaak het nie. Die tekstielstof se styfheid het toegeneem wanneer dit met *catholyte* gewas is, alhoewel die invloed minder was as dieselfde tekstielstof gewas met detergent. Kreukelherstel het verbeter nadat die poliamiede met *catholyte* gewas is. *Catholyte* het nie 'n noemenswaardige invloed op die dimensionele veranderinge van die poliamied 6,6 tekstielstof na was getoon nie. Uit boge-noemde kan afgelei word dat *catholyte* nie 'n beskadigende invloed op die poliamiede 6,6 tekstielstof wat in hierdie studie evalueer is gehad het in terme van treksterkte, styfheid, kreukelherstel en dimensionele verandering nie.

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INTRODUCTION

Laundry detergents are commonly used washing agents (Hollis, 2002) in almost every household in the developed world (Cameron, 2007). The chemicals in laundry detergents are, however, non-renewable (Bajpai & Tyagi, 2007) and subsequently enter the sewage system (Stalmans *et al*, 1991). Given the immense amounts of laundry detergents that are used, environmentalists are concerned about the detrimental effects of chemicals entering water systems. Water is one of the most critical elements for humans to survive and it is important that fresh water supplies are protected (Bajpai & Tyagi, 2007).

Phosphate, one of the most important ingredients in conventional laundry detergents, is also associated with environmental issues. One such issue is eutrophication, which occurs when the nutrient level in the water increases. The nutrient increase causes the formation of large algae blooms, which turns slow moving and stagnant water masses murky or even toxic (Köhler, 2006). Thus eutrophication of natural water resources can cause serious damage to water life (Hui & Chao, 2006).

In 2007 there were virtually no phosphate formulations on the U.S. detergent market, but 68% of the European and approximately 50% of the Canadian detergents contained phosphate. Furthermore, Latin America and some of the Pacific region countries are still using phosphate -based detergents (Bajpai & Tyagi, 2007). As of 30 June 2013, the European Union has subsequently limited laundry detergent phosphorus content to 0,5 g in the recommended

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quantity to be used in a standard washing machine (European Union Regulation, 2012). In contrast there is no regulation introduced in South Africa to limit the phosphorus content in detergents. However, the Water Research Commission undertook pilot studies regarding the different eutrophication-management options to determine their effectiveness and applicability for implementation (Van Ginkel, 2011). The most recent of these was to investigate the consequences of introducing zero-phosphate detergents into South Africa. They concluded that the projected reduction in phosphate concentration loading due to introduction of zero-phosphate detergents is significant, although being extremely costly and the feasibility is questioned (Quayle *et al*, 2010). It is evident that alternative detergents need to be investigated.

The fibre production in the world has increased by 30% in the last ten years. Cotton and polyester accounts for approximately 83% of the total of fibres being produced. Polyamide 6,6 is the third most produced fibre, which accounts for 3,8 million tonnes of total fibre produced (Mowbray, 2011). Over the last few years an increasing interest in polyamides can be observed. This is mainly due to the outstanding properties for example, high strength, stiffness, wear resistance and dimensional stability. These favourable properties renders polyamide 6,6 suitable for a wide variety of applications (Gaitonde *et al*, 2010). Polyamides are easy to care for but tend to discolour during the laundering process (Kadolph, 2007:128).

Van Zyl conducted a study to determine the influence of catholyte on the soil removal as well as on certain properties of cotton, polyester and a cotton/polyester blend (2008). Since such a study was yet to be done on polyamide, Van Heerden *et al* (2012) evaluated the soil removal efficacy of catholyte on polyamide 6,6 woven fabric. Catholyte proved to have effectively removed the soil from the fabric. It was the focus of this paper to evaluate the effect of catholyte on certain properties of polyamide 6,6.

REVIEW OF LITERATURE

Polyamide, commonly known as nylon, is an important textile fibre (Kumar & Gupta, 1998:10) and due to its outstanding properties mentioned earlier, is suitable for a wide variety of applications, lingerie and hosiery products in particular (Kadolph, 2007:123).

Polyamide 6,6 fibres are made up of a well-orientated polymer system (Gohl & Vilensky, 1983:104), which is synthesized by condensation polymerization of 1,6-diaminohexane and hexandioic acid (adipic acid) to form a macromolecule (Wynne, 1997:86). Polyamide 6,6 is resistant to alkalis (Kadolph, 2007:124). Alkali hydrolysis will however occur when it is frequently exposed to alkali. The fabric weakens as a result. The prolonged exposure of polyamide 6,6 to alkali causes white polyamide fabrics to yellow and coloured polyamide 6,6 fabrics to become dull (Gohl & Vilensky, 1983:110).

Fabrics made from polyamide 6,6 are machine washable (Collier & Tortora, 2001:172). Ruscher (2004) considers temperatures of 30˚C or 40˚C sufficient for laundering polyamide 6,6, compared to the previously recommended 60˚C of Cook (Cook, 1984:253). Laundering in warm water with gentle agitation helps to prevent wrinkling, although wrinkles that have been set by hot wash water may be permanent (Kadolph, 2007:128).

Electrochemically activated aqueous media have recently been developed. The catholyte part of the electrochemically activated water is used in a wide range of applications including medicine, agriculture (Lobyshev, 2007), microbiology and the food industry (Khrapenkov & Gernet, 2002). Catholyte is also used as environmentally friendly anti-microbial and aqueous washing media (Bakhir, 2005), and may provide an environmentally sensible alternative to chlorine and other solvents (Thantsha & Cloete, 2006).

Electrochemical activation is a physico-chemical process (Lobyshev, 2007). Exposure of the water to electrochemical activation results in the altering of the molecular state of the water from stable into metastable (activated) aqueous media (Khrapenkov & Gernet, 2002). In the activated state, the water is marked by unusual physical and chemical parameters (Tomilov, 2002). Through electrochemical activation it is possible to purposefully change the acid-base and oxidative-reductive properties of the water (Bakhir, 2005).

The water is passed through the electrochemical cells, anode and cathode. These cells or electrodes are specifically designed to activate the two different media, each of which has a unique set of properties and characteristics (Thantsha & Cloete, 2006). The electrodes are unipolar with a double electric layer near the surface of the electrode (Leonov, 1997). Catholyte is alkaline with reduced redox potential, which means electrons can be given up (Forostyan *et al*, 1987). The concentration of the alkalis in catholyte is proportional to the mineralisation of the water and the electricity consumption during the process when it was synthesised (Bakhir, 2005).

Catholyte will remain in a state of metastability for a few days. The anomalous properties of the media disappear after a long inactive period (Forostyan *et al*, 1987). Even in an activated state, catholyte is non-toxic to the environment. Furthermore, the electrochemically activated water is easy to handle and compatible with other water treatment chemicals (Thantsha & Cloete, 2006).

Electrochemical activation is an attractive technology because it simplifies, accelerates and reduces the price of some routine technological processes. The product quality also increases and wastewater contamination decreases (Bakhir, 2005).

The catholyte part of the activated water is a promising alternative to laundry detergents, the feasibility of which still needs to be established. Hence the focus of this paper was to evaluate the effect of catholyte on certain properties of polyamide 6,6.

EXPERIMENTAL DETAILS

The standard unsoiled, undyed polyamide 6,6 fabric was purchased from Testfabrics, Inc. The polyamide 6,6 fabric was made from 100% machine spun Du Pont type polyamide 6,6 yarns meeting the ISO 105 standard. The fabric was woven using a plain weave construction with a weight of 124 g/m².

Laundering was conducted using an Atlas Launder-Ometer (Atlas Electric Devices Co). Standard test methods for laundering (AATCC 61-2009, test IIA) and measuring the maximum load at break (tensile strength) including displacement at maximum load (ISO/SANS 13934-1:1999), wrinkle recovery (AATCC 66), bending length (BS 3356) and dimensional change were used. Dimensional change was determined by counting the threads in a determined dimension before and after laundering and calculating the recorded readings as a percentage (%) value.

The polyamide 6,6 fabric was laundered with catholyte, phosphate detergent or distilled water (treatment). Unlaundered and thus untreated polyamide 6,6 fabric was also included in the test methods. The catholyte was prepared by the electrolysis of a 5% NaCl concentration distilled water solution. Electrolysis was carried out under uniform conditions of a continuous electric current of twelve ampere and pressure of seventy-five kilo-Pascal. Electrolysis was continued until the catholyte reached a pH between 12 and 13 (Annandale *et al*, 2008). The catholyte was used within eight hours after preparation. The ECE Phosphate Reference Detergent Type B without optical brightener was purchased from James H. Heal & Co. Ltd, Halifax, England. The detergent (code 706-736) met the ISO 105 standard for testing. According to the manufacturer, the composition of the detergent is as follows: linear sodium alkyl benzene sulphonate (8%), ethoxylated tallow alcohol (2,9%), sodium soap (3,5%), sodium tripolyphosphate (43,7%), sodium silicate (7,5%), magnesium silicate (1,9%), carboxy methyl cellulose (1,2%), ethylene diamine tetra acetic acid (0,2%), sodium sulphate 21,2%%), water (9,9%). The Institute for Groundwater Studies, University of the Free State, Bloemfontein, South Africa determined the composition of the distilled water: pH (7,54), electrical conductivity (18,8 mS/m), Ca (17,5 mg/l), Mg (5,4 mg/l), Na (9,9 mg/l), K (2,46 mg/l), Cl (13,8 mg/l), PO_4 (<0,1 mg/l), SO_4 (10,7 mg/l), Al 0,008 mg/l), Cu (0,061 mg/l), Fe (0,018 mg/l), Mn (0 mg/l), Ni (< 0,01 mg/l), Zn (0,056 mg/l).

Detergent dose included per wash was 0,23 g detergent per 150 ml distilled water according to the AATCC Test Method. Fifty stainless steel balls were placed in each stainless steel cylinder as well as 150 ml of the washing solution in accordance with the IIA test in AATCC Test Method 61-2009. The balls were counted after each laundering cycle to ensure all the samples were subjected to the same degree of agitation. One sample per cylinder was allowed. Thus, 150 ml distilled water was added to samples laundered with distilled water, and 150 ml catholyte was added to samples laundered with catholyte.

Per treatment (catholyte, detergent, distilled water), the polyamide 6,6 fabric was laundered at 30°C or 40°C (temperature), for five, ten, twenty or fifty laundering cycles (number of laundering cycles) respectively, using a fully crossed 3×2×4 factorial design. Five replicate measurements (per direction: weft and warp) were taken of tensile strength, four replicate measurements of bending length, three measurements of wrinkle recovery and five measurements of dimensional change.

Data were analysed using analysis of variance (ANOVA). The dependent variables, tensile strength, bending length and wrinkle recovery were analysed separately for the polyamide 6,6, and two directions of measurement, warp and weft. The analysis of variance (ANOVA) model fitted the factors treatment (catholyte, detergent, distilled water), temperature (30°C, 40°C), and number of laundering cycles (5, 10, 20, 50), as well as all two-factor interactions and the three factor interaction between these variables. F-tests and associated P-values for all effects in the model were obtained from the ANOVA.

TABLE 1: INFLUENCE OF TREATMENT, TEMPERATURE AND LAUNDERING CYCLES ON THE TENSILE STRENGTH (MEAN MAXIMUM LOAD) OF POLYAMIDE 6,6 FABRIC

DIRECTION	MAIN EFFECT		MEAN MAXIMUM LOAD (N)	DIFFERENCE	P-VALUE	DIFFERENCE	P-VALUE
WEFT	NO TREAMENT		724,66				
WEFT	TREATMENT			(Relative to Distilled Water)		(Catholyte relative to Detergent)	
		Catholyte	674,40	53,88	0,0992	7,32	0,8217
		Detergent	667,08	46,57	0,1534		
		Distilled Water	620,52				
	TEMPERATURE			(Relative to 40ºC)			
		30ºC	654,27	0,53	0,9841		
		40ºC	653,73				
	NUMBER OF LAUN-DERING CYCLES			(Relative to 5 cycles)			
		5	646,09				
		10	658,44	12,35	0,7417		
		20	655,70	9,61	0,7977		
		50	655,76	9,67	0,7964		
WARP	NO TREATMENT		1173,18				
WARP	TREATMENT			(Relative to Distilled Water)		(Relative to Detergent)	
		Catholyte	1138,46	-40,64	0,3317	65,73	0,1178
		Detergent	1204,19	-106,36	0,0122*		
		Distilled Water	1097,83				
	TEMPERATURE			(Relative to 40ºC)			
		30ºC	1101,50	-90,66	0,0090*		
		40ºC	1192,16				
	NUMBER OF LAUN-DERING CYCLES			(Relative to 5 cycles)			
		5	1124,36				
		10	1134,62	10,25	0,8316		
		20	1168,75	44,39	0,3583		
		50	1159,57	35,20	0,4659		

*Statistically Significant

Furthermore, least squares means for all treatments, temperatures, cycles and combinations of these three factors were calculated with their standard errors. Estimates of the differences between least squares means, with associated P-values were also reported. All analyses were carried out using the GLM procedure of the SAS software package (SAS Institute Inc., 2004).

RESULTS AND DISCUSSION

Tensile Strength

The effect of laundering with catholyte, detergent and distilled water on the tensile strength in the weft and warp directions of polyamide 6,6 fabric is summarised in Table 1. The differences between the three treatments were not statistically significant. The rather anomalous large drop in tensile strength caused by laundering with distilled water cannot be explained, although Vermaas (2011:100) found similar results.

Regarding the warp direction, laundering with detergent had the highest tensile strength, followed by “catholyte” and “distilled water”. The difference between detergent and distilled water was statistically significant, and between catholyte and detergent insignificant. These results were not unexpected as it is generally accepted that polyamide 6,6 is resistant to alkalis (Collier & Tortora, 2001:172). It is expected that the warp direction can withstand a higher maximum load as stronger yarns are used for in the warp direction to withstand the stress of the weaving process (Kadolph, 2007:129). The insignificant differences (warp and weft) between catholyte and detergent are encouraging. This indicates that the effect on tensile strength of polyamide 6,6 will be similar when phosphate detergents are replaced with catholyte.

Laundering temperature (30°C versus 40°C; averaged over the factors treatment and number of laundering cycles) had no significant effect on the tensile strength in the weft direction of polyamide 6,6. In contrast, the tensile strength in the warp direction when laundered at 30°C was significantly lower than when laundered at 40°C (Table 1). These are unexpected results and an explanation as to why only the warp yarns were affected significantly still eludes the researchers. Furthermore, the behaviour of the warp direction of the fabric seems to be corresponding with literature regarding the

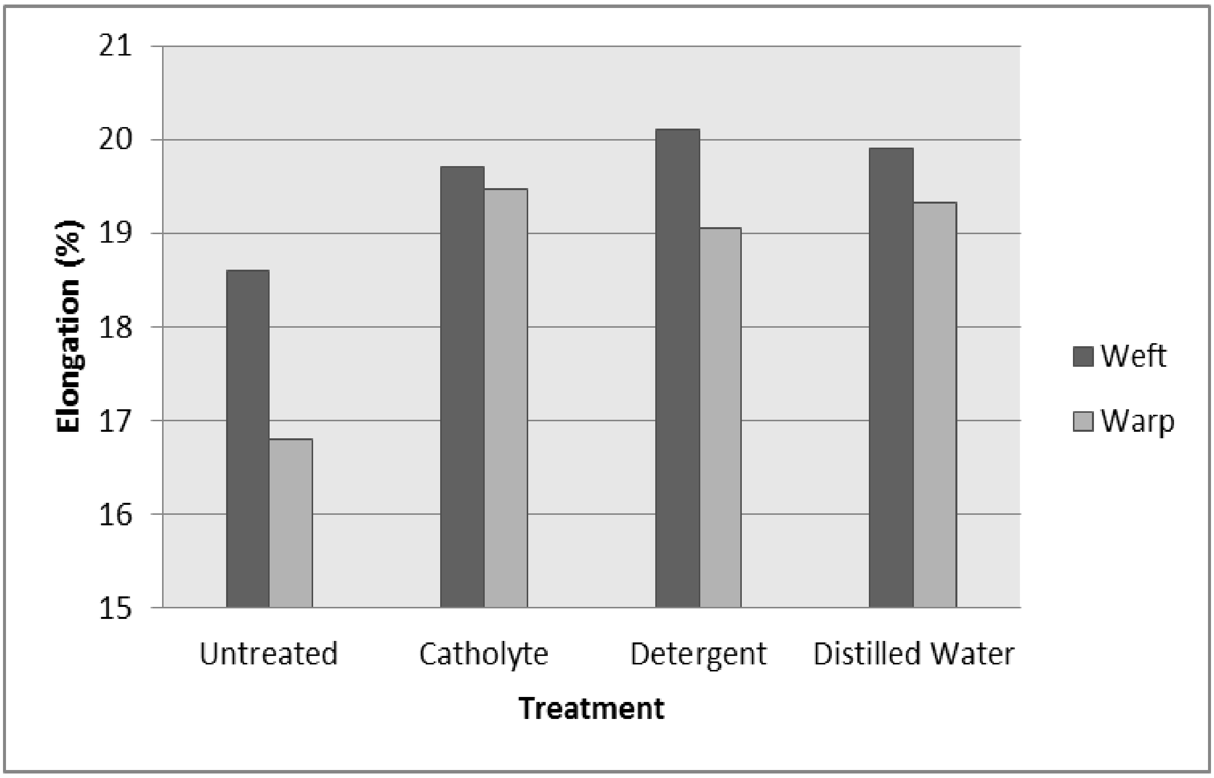

FIGURE 1: ELONGATION AT MAXIMUM LOAD FOR POLYAMIDE 6,6 FABRIC LAUNDERED WITH CATHOLYTE, DETERGENT AND DISTILLED WATER

TABLE 2: INFLUENCE OF TREATMENT AND LAUNDERING CYCLES ON THE BENDING LENGTH OF POLYAMIDE 6,6 FABRIC

DIRECTION	MAIN EFFECT		MEAN MAXIMUM LOAD (N)	DIFFERENCE	P-VALUE	DIFFERENCE	P-VALUE
WEFT	NO TREAMENT		2.35				
WEFT	TREATMENT			(Relative to Distilled Water)		(Catholyte relative to Detergent)	
		Catholyte	3,53	-0,05	0,2841	-0,30	<0,0001*
		Detergent	3,83	-0,35	<0,0001*		
		Distilled Water	3,48				
	NUMBER OF LAUNDERING CYCLES			(Relative to 5 cycles)			
		5	3,34				
		10	3,90	0,56	<0,0001*		
		20	3,66	0,32	<0,0001*		
		50	3,55	0,22	0,0002*		
WARP	NO TREATMENT		2,58				
WARP	TREATMENT			(Relative to Distilled Water)		(Catholyte relative to Detergent)	
		Catholyte	3,22	-0,02	0,4093	-0,55	<0,0001*
		Detergent	3,77	0,53	<0,0001*		
		Distilled Water	3,24				
	NUMBER OF LAUNDERING CYCLES			(Relative to 5 cycles)			
		5	3,14				
		10	3,53	0,39	<0,0001*		
		20	3,58	0,44	0,0001*		
		50	3,48	0,33	<0,0001*		

*Statistically Significant

findings of the wrinkle recovery, dimensional stability and stiffness respectively. The number of laundering cycles (averaged over the factors temperature and treatment) had no significant effect on the maximum load tensile strength in either the weft or warp direction (Table 1). These results are not unexpected as polyamide 6,6 is known for excellent strength and launderability (Johnson & Cohen, 2010:87).

Elongation displacement of yarns before breakage occurred is correlated to the breaking force or the maximum load the yarns could carry before breaking (Kadolph, 1998:161). Figure 1 illustrates the effect of catholyte, detergent and distilled water on the elongation at maximum load in the weft and warp directions of the polyamide 6,6 fabric. The elongation in weft direction of the polyamide 6,6 fabric ranged from 19,7% to 20,1% , resulting in a negligible difference of 0,4%. The elongation in the warp direction ranged from 19% to 19,5%, a negligible difference of only 0,5%.

Bending Length (Stiffness)

Bending length is a simple and accurate way in which the stiffness of a fabric is expressed. Table 2 summarises the effect of catholyte, detergent and distilled water on the bending length of polyamide 6,6. The difference between catholyte and distilled water was not significant in both the weft and warp directions. There was, however, a significant difference between catholyte and detergent, and between detergent and distilled water. Detergent caused

TABLE 3: INFLUENCE OF TREATMENT, TEMPERATURE AND LAUNDERING CYCLES ON THE WRINKLE RECOVERY OF POLYAMIDE 6,6 FABRIC

DIRECTION	MAIN EFFECT		MEAN WRINKLE RECOVERY ANGLE (°)	DIFFERENCE	P-VALUE	DIFFERENCE	P-VALUE
WEFT	NO TREAMENT		115,00				
WEFT	TREATMENT			(Relative to Distilled Water)		(Catholyte relative to Detergent)	
		Catholyte	118,38	-2,96	0,1701	2,67	0,2153
		Detergent	115,71	-5,63		0,0109*	
		Distilled Water	121,33				
	TEMPERATURE			(Relative to 40ºC)			
		30ºC	118,11	-0,72	0,6789		
		40ºC	118.83				
	NUMBER OF LAUN-DERING CYCLES			(Relative to 5 cycles)			
		5	123,22				
		10	119,89	-3,33	0,1804		
		20	115,39	-7,83	0,0025*		
		50	115,39	-7,83	0,0025*		
WARP	NO TREATMENT		116,33				
WARP	TREATMENT			(Relative to Distilled Water)		(Catholyte relative to Detergent)	
		Catholyte	117,13	-3,83	0,0439*	-1,55	0,4093
		Detergent	118,67	-2,30	0,2220		
		Distilled Water	120,96				
	TEMPERATURE			(Relative to 40ºC)			
		30ºC	121,75	5,67	0,0005*		
		40ºC	116,08				
	NUMBER OF LAUN-DERING CYCLES			(Relative to 5 cycles)			
		5	120,61				
		10	123,94	3,33	0,1257		
		20	113,67	-6,94	0,0021*		
		50	117,44	-3,17	0,1452		

*Statistically Significant

the polyamide 6,6 fabric to be the most rigid or stiff. Sodium tripolyphosphate (an anionic surfactant that is present in most of the detergents that is available today) is found to cause increased stiffness in textile fabrics. This happens as a result of sodium tripolyphosphate forming a film on the surface of the textile (Chattopadhyay & Inamdar, 2012). As sodium tripolyphosphate forms 43,7% of the total detergent formulation used in this study, this might explain why the polyamide laundered with catholyte and distilled water was not as stiff as when the fabric was laundered with detergent.

The number of laundering cycles had a significant effect on bending length for both weft and warp directions (Table 2). Laundering the polyamide 6,6 fabric for ten cycles caused an increase in the bending length in the weft direction. Laundering the polyamide 6,6 fabric for another ten cycles, decreased the bending length slightly. Laundering the polyamide 6,6 fabric for fifty cycles again caused a slight decrease in the bending length in the weft direction. The bending length in the warp direction of the polyamide 6,6 fabric laundered for ten cycles was higher than the polyamide laundered for five cycles. Laundering the polyamide 6,6 fabric for twenty cycles resulted in slightly higher bending length in the warp direction while laundering for fifty cycles, slightly decreased bending length. Thus the stiffness increased as the number of laundering cycles increased up to ten cycles, and gradually decreased thereafter.

Similar results were found by Lau *et al* (2002). They found a significant increase in the initial four laundering cycles, followed by a levelling in the subsequent washes. The increased stiffness is related to yarn-to-yarn friction.

Wrinkle Recovery

Table 3 summarises the effect of repeated laundering with catholyte, detergent and distilled water (averaged over the factors temperature and laundering cycles) on the wrinkle recovery of polyamide 6,6. The highest wrinkle recovery in the weft direction was achieved for distilled water, followed by catholyte and detergent. Fabric laundered with catholyte recovered less well from induced wrinkles than fabric laundered with distilled water, but better than fabric laundered with detergent. The difference between detergent and distilled water was significant, but the difference between detergent and catholyte was not significant.

Regarding the warp direction, laundering with distilled water resulted in the highest wrinkle recovery, followed by detergent and catholyte. The difference between detergent and catholyte was not significant, but the difference between catholyte and distilled water was significant.

Laundering temperature had no significant effect on wrinkle recovery in the weft of the fabric, but in the warp direction fabric laundered at 30°C had significantly higher wrinkle recovery than fabric laundered at 40°C.

A higher number of laundering cycles resulted in a significant decrease in wrinkle recovery, with both 20 and 50 cycles having significantly lower wrinkle recovery than 5 cycles in the weft direction. In the warp direction, wrinkle recovery for 5 and 10 laundering cycles was not statistically different, but 20 cycles significantly decreased wrinkle recovery relative to 5 cycles. Overall, wrinkle recovery seemed to decrease with increasing number of laundering cycles (Table 3).

Polyamide 6,6 is a strong fibre having high elastic recovery and elongation properties (Kadolph, 2007:127), mainly due to the regularity of the strong hydrogen bonds. These bonds operate over short distances, so it is able to exert optimum strength that prevents polymer slippage and causes the polymers to return to their original position. This means that the fibres return readily to their original shape, do not wrinkle or crease easily (Gohl & Vilensky, 1983:104) and recover well (Joseph, 1986:103).

Dimensional Change

A particularly important aspect of textile fabric properties is dimensional change. Most measurements of dimensional change were zero, so that statistical analysis using ANOVA was inappropriate. The mean dimensional change under any of the experimental conditions did not exceed 1,6% in either the weft or warp directions. These results were expected because polyamide is known for its dimensional stability (Kadolph, 2007:128).

CONCLUSION

The differences between laundering with catholyte and detergent were generally small and not significant. Laundering generally reduced fabric tensile strength, with laundering in distilled water generally causing a greater decrease than laundering in either catholyte or detergent. Laundering generally increased fabric stiffness, more so when using either catholyte or detergent, than when using distilled water. Laundering slightly increased wrinkle recovery, more so when using distilled water, than when using catholyte or detergent. Dimensional stability was not in adversely affected.

It can thus be concluded that catholyte is a suitable alternative detergent for polyamide 6,6 fabric. The influence that it has on the properties examined in this paper was not to the detriment of the fabric.

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