THE EVALUATION OF HYDROGEN PEROXIDE BLEACHING OF *GONOMETA POSTICA SILK*

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OPSOMMING

Degradasie van *Gonometa postica*-sy as gevolg van bleiking is nie alleen 'n belangrike probleem vir verbruikers nie, maar ook vir die vervaardiger. Die doel met die navorsing was om die effek van waterstofperoksiedbleiking op *G. postica*-sy te bepaal. Die spesifieke doelstelling was om die invloed van temperatuur, pH en tydsduur op waterstofperoksiedvrystelling, kleurverskil, breekkrag en styfheid te meet.

'n Literatuurstudie is onderneem om waterstofperoksiedbleiking op degradasie van sy te ondersoek ten einde faktore wat hierdie effek beïnvloed te identifiseer. 'n Eksperimentele studie is uitgevoer om die waterstofperoksiedvrystelling by 50 ˚C, 60 ˚C, 70 ˚C en 90 ˚C by pH 8 en pH 10 te bepaal. Die *G. postica*-sy is in 'n 5 liter waterbad met waterstofperoksied gebleik en daarna gedroog. Die monsters is onder standaard atmosferiese toestande, 20 ± 1 ˚C en 65 ± 2% relatiewe humiditeit gehou voor en met toetsing. Die kleurverskil, breekkrag en styfheid van *G. postica*-sy is na bleik behandeling met waterstofperoksied bepaal. Waterstofperoksiedvrystelling is deur middel van 'n kaliumpermanganaat-titrasie tydens die bleik aksie bepaal, waarna die kleur verskil (Delta E) van die monsters met 'n kolorimeter gemeet is. 'n 'Instron tensile tester' is gebruik om breekkrag van gebleikte en ongebleikte monsters te meet. Die breekkrag is gemeet in Newton. Die buigstyfheid is met 'n "shirley stiffness tester" gemeet. Die bevindinge dui daarop dat temperatuur, pH en tydsduur van blootstelling 'n rol speel in die bleiking van *G. postica*-sy. 'n Toename in kleurverskil (bleiking) met styging in temperatuur is waargeneem. Die beste bleiking is waargeneem na 60 minute blootstelling aan waterstofperoksied by 90 ˚C en pH 8, 180 minute blootstelling by 70 ˚C en pH 8 het ook goeie bleiking tot gevolg gehad. Hoë temperature het die breekkrag verlaag en die styfheid van die tekstielstof verhoog met toename in die temperatuur van blootstelling. Waterstofperoksiedvrystelling vind vinnig plaas by die hoër temperature. Die pH-waarde het die grootste invloed op waterstofperoksiedvrystelling en kleur verskil (bleiking). Die gevolgtrekking word gemaak dat temperatuur en pH-waarde die faktore is met die grootste invloed op kleurverskil (bleiking), breekkrag en styfheid van *G. postica*-sy.

Alhoewel aanvaarbare bleiking van *G. postica*-sy resultate verkry word met 60 minute blootstelling aan waterstofperoksied by 90℃ en pH 8 of 180 minute by 70℃ en pH 8, kan die bleiking van die tekstielstof nie aanbeveel word nie omdat die bleikproses veroorsaak dat die tekstielstof te styf raak en die breekkrag van die tekstielstof verswak.

Dit word voorgestel dat *G. postica*-sy in sy natuurlike goudbruin kleur gebruik word, of gekleur word na 'n donkerder kleur totdat 'n aanvaarbare bleikproses ontwikkel word wat die materiaal nie te hard of styf laat en die tekstielstof verswak nie.

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INTRODUCTION

Wild silk from the *Gonometa postica* exhibits a rich tan colour, a natural asset, but consumers want more variety in colour. The natural colour is the result of the carotene pigment (Mohanty, 2003:150) in the silk induced by the *Acacia erioloba* (camel thorn tree) leaves that the *G. postica* larvae feed on (Olivier, 2002). The natural colour of the *G. postica* makes it impossible to dye in colours lighter than the natural tan colour of the fibre without bleaching. Silk, like other protein fibres is sensitive to bleaches (Sharma *et al*, 1999). Wild silk is seldom dyed, and when it is dyed, it is dyed in darker solid colours (Nakamura, 1980:58).

The tenacity of wild silk is reported to vary between 2,2 and 5,2 gram/Denier (Lee, 1999). This tenacity allows it to be useful for household textiles and clothing. The fibres can easily be damaged by physical and chemical factors in the processing of the fibre to fabric and in the finishing of the fabric (Freddi, *et al*, 2003).

Wild silk possesses a natural elasticity (Joseph, 1986:60), an asset in textiles used for household purposes and clothing. The elasticity and rigidity of wild silk can also be negatively affected by processes and finishes (Corbman, 1975:329). The moisture regain of silk is 11% (Ishida, 1991:5), which adds to the excellent dyeing properties of silk.

High temperature can cause deterioration of silk fibre but authors do not agree on the temperature that would cause damage. Gohl and Vilensky (1983:87) indicate that 100 ºC might cause damage while Joseph (1986:60) indicates that temperatures up to 135ºC would not damage the silk. Other authors report yellowing as a sign of damage at 110ºC□111ºC (Lee, 1999) and disintegration takes place at 170ºC (Lee, 1999).

Silk is damaged by strong mineral acids like sulphuric acid and nitric acid, but organic acids like acetic acid and formic acid can be used in the finishing of silk (Joseph, 1986:60). Concentrated alkali dissolves silk as a result of peptide bond hydrolysis (Gohl & Vilensky, 1983:83) however; soap, borax and ammonia can be used on silk without damage (Joseph, 1986:60, 61).

Hydrogen peroxide is recommended for bleaching protein fibres like silk (Sharma *et al*, 1999; Joseph, 1986:286). Greenwood and Earnshaw (1998:633) report that hydrogen bleaching of silk has been used since 1878. There is no agreement on the exact mechanism of hydrogen peroxide bleaching and different theories are proposed (Dannacher & Schlenker, 1996; Karl & Freyberg, 2000). The more recent proposal is that free radicals are formed and the colour is oxidised by electron removal and breaking of the hydrogen bonds (Calvert, 2003; Parent, 2004).

Bleaching causes silk fibres to lose up to 10% of its strength (Amin, *et al*, 1998; Wang & Ramaswamy, 2003; Kasimir, 1990). Some authors maintain that silk can be bleached without damage if the temperature, time of exposure, alkalinity and concentration of the hydrogen peroxide is controlled (Gulrajani, *et al*, 1992; James & Mackirdy, 1990; Wynne, 1997:235).

The most important factor in bleaching seems to be the pH level with the best bleaching in an alkaline solution, although an acidic medium may also be used (Greenwood & Earnshaw 1998:635). Authors agree that an alkaline medium would be the best, but differ on the specific pH level. Some recommend pH 8 (Wynne, 1997:243) others pH 10 (Dannacher & Schlenker, 1996; James & Mackirdy, 1990). The quality of the bleached silk is dependent on the hydrogen peroxide concentration (Westbrook, *et al*, 1999). Concentrations of 35% and 50% are most often used in industry (Kroschwitz, 2003).

The purpose of this study was to determine the effect of hydrogen peroxide bleaching on *G. postica* silk fabric. The influence of temperature, pH and time duration on hydrogen peroxide release, as well as colour change, breaking load required and stiffness of *G. postica* silk fabric was determined.

MATERIALS AND METHODS

Fabric woven with *Gonometa postica* silk from Namibia was used in its natural tan colour.

Determination of the hydrogen peroxide release

Hydrogen peroxide (H_2O_2) was used as bleach (Merck chemicals 50% concentration). Hydrogen peroxide (50ml),10g tetrasodium pyro phosphate and 5g EDTA (Trotman1975:261) was added to the 5ℓ distilled water in the bath and the bath was heated to 50 ˚C, 60 ˚C, 70 ˚C and 90 ˚C respectively at pH 8 and pH10. A sodium hydroxide solution was used to set the pH. At 30 minute intervals, 25 ml of the bleach solution was removed from the water bath and the hydrogen peroxide concentration was determined (Vogel, 1961:296). A Piccolo 2 HI 1290 pH meter was used to determine the pH.

The concentration of hydrogen peroxide was determined by the following equation: 1ml.N-KMn_4O=0,01701g.H_20_2

Determination of the influence of temperature, pH and time duration on colour change

Samples of 60 mm ´ 60 mm were used and bleached in the water bath in which bleach concentration, temperature and pH could be controlled.

The specific duration for sufficient bleaching was determined at each of the following temperatures: 50 ˚C, 60 ˚C, 70 ˚C and 90 ˚C. To determine the duration of exposure required for bleaching at different temperatures and at pH 8 and pH10, the following procedure was followed:

The hydrogen peroxide bath at pH 8 was heated to 50 ˚C. Ten samples were put in the bath and one sample removed every 30 minutes for 5 hours (no further bleaching was expected on the ground of preliminary tests).The experiment was repeated three times. Another set of eight samples was exposed for up to 240 minutes to the hydrogen peroxide bath at 60 ˚C, a set of six samples was exposed for up to 180 minutes to the bath at 70 ˚C, and the final set of two samples was exposed for up to 60 minutes at 90 ˚C. Samples were removed at 30 minute intervals. The same procedure was repeated at pH10 level.

The process as illustrated below was repeated three times and 156 samples were bleached:

Duration	50 ˚C for 5 hours	60 ˚C for 4 hours	70 ˚C for 3 hours	90 ˚C for 1 hour
30 minutes	ß	ß	ß	ß
1 hour	ß	ß	ß	ß
1 hour, 30 minutes	ß	ß	ß	
2 hours	ß	ß	ß	
2 hours, 30 minutes	ß	ß	ß	
3 hours	ß	ß	ß	
3 hours, 30 minutes	ß	ß		
4 hours	ß	ß		
4 hours, 30 minutes	ß			
5 hours	ß			

ß Samples

The samples were dried in a dark room and kept under standard conditions (20 ± 1 ˚C and 65 ± 2% Relative humidity)

A colorimeter (Minolta 1998) was used to determine whiteness of the samples after bleaching. Colour differences were determined according to published methods (Greenblau 1978:25; Minolta 1998:18).

Determination of the stiffness and the load required to break the *G. postica* silk fabric before and after bleaching

A set of 24 samples (400 mm ´ 400 mm), four per temperature (60˚C; 70˚C; and 90˚C) and two per pH level (pH8 and pH 10), was bleached and dried using the same processes as described above and were cut in the required sample size for the tests used to determine the stiffness and breaking load. The samples were dried in a dark room and kept under standard conditions (20 ± 1 ˚C and 65 ± 2% Relative humidity) before the stiffness tests and the load required to break a sample were determined.

A set of 12 (two samples per temperature and for each pH level) 400mm x 400mm samples that was bleached and dried as well as two unbleached samples were cut into 250mm x 25mm smaller samples for the determination of the stiffness (bending length) using a “Shirley stiffness tester”.

Using 12 of the 400mm x 400mm samples (two per temperature and at each pH level) 72 smaller test samples were cut for determining the breaking load. Each 400mm x 400mm sample was cut into six smaller samples of 300 mm (tearing direction) ´ 60 mm and frayed in the tearing direction until a width of 50 mm was obtained. The load required to break the fabric was determined with an Instron tensile tester using SABS ISO Test Method 1999: 13934-1 (SABS, 1999).

Statistical analysis

A factorial analysis was done on the effect of temperature, pH, and duration of exposure on the colour, breaking load and stiffness of the samples. A multiple regression was done to evaluate the hydrogen peroxide release at different temperatures, pH and duration of exposure. Per pH level, the unbleached and bleached samples’ breaking loads and stiffness were compared using a t-test.

RESULTS AND DISCUSSION

The influence of temperature, pH and time duration on hydrogen peroxide release and colour change

Hydrogen peroxide release The hydrogen peroxide release at 50℃, 60℃, 70℃ and 90℃ at pH 8 and pH 10 was measured to determine the most efficient combination of temperature and pH level for hydrogen peroxide release.

Figure 1 and 2 show the pH 10 and pH 8 hydrogen peroxide release at the different temperatures over time.

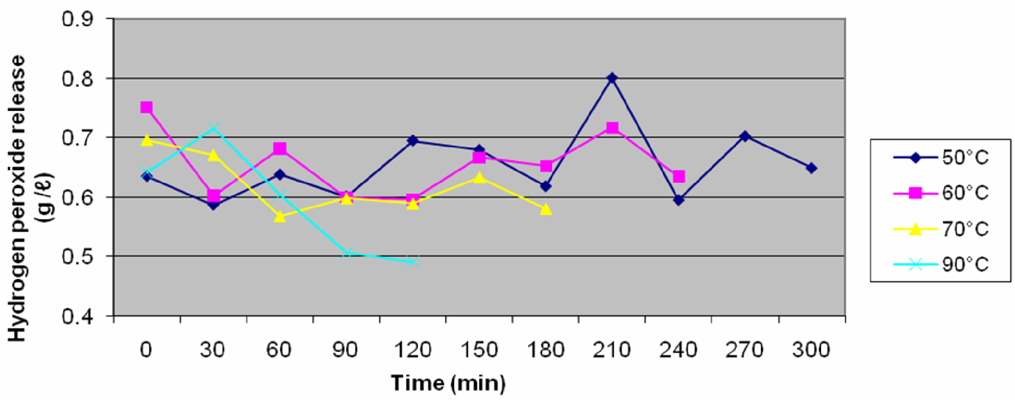

FIGURE 1: THE HYDROGEN PEROXIDE RELEASE AT 50°C, 60°C, 70°C AND 90°C AT pH8

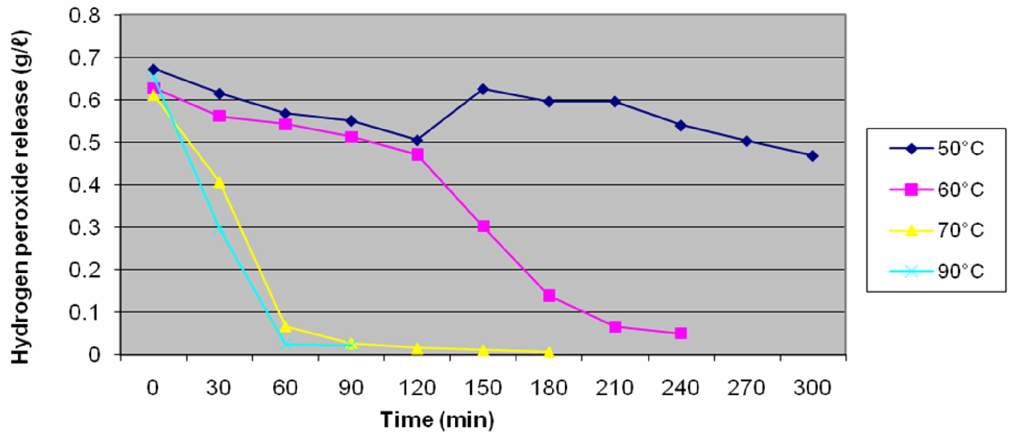

FIGURE 2: THE HYDROGEN PEROXIDE RELEASE AT 50°C, 60°C, 70°C AND 90°C AT pH10

TABLE 1: REGRESSION REPORT OF HYDROGEN PEROXIDE RELEASE

Independent variable	p-value
Temperature	.000
Time duration	.000
pH	.063

Hydrogen peroxide release lowered rapidly in the first sixty minutes of exposure at 70℃ and 90℃. At 60℃ the hydrogen peroxide release was fairly high for the first 120 minutes and at 50℃ the high release was maintained for at least 300 minutes. These results indicated that better bleaching might be expected at 50℃ or 60℃. The difference between temperatures was much less pronounced at pH8. At 50℃ and 60℃ a phased release of hydrogen peroxide was experienced over 4 to 5 hours. A lower release was experienced after the first 30 minutes at 70℃ and at 90℃ the release lowered even quicker. Time duration and the temperature of exposure are both indicated as significant factors (Table 1) in the hydrogen peroxide release but pH did not have a significant influence on the hydrogen peroxide release.

Colour change. The required duration for bleaching varied at different temperatures. The silk needed longer exposure to bleaching at 50℃ and 60℃ because bleaching takes place faster at higher temperatures in agreement with the results of other research (James and Mackirdy, 1990:642).

The colour change experienced with bleaching at 50° C, 60℃, 70˚C and 90℃ at pH 8 and pH10 is shown in figures 3, 4, 5 and 6.

The best bleaching result of 81 delta E was achieved at 90℃ (as shown in Figure 6) for 60 minutes at pH 8. The second best result of 79, 8 delta E was obtained with 180 minutes exposure to hydrogen peroxide at 70˚C also at pH8. These results are in agreement with Wynne (1997:243) who prescribed pH 8 for bleaching.

The best result (78,7 delta E) at pH 10 was for an exposure of 150 minutes at 60℃. Dannacher and Schlenker (1996) and James and Mackirdy (1990) recommended a pH10 for bleaching.

Table 2 shows the multiple regression report that indicate that temperature, duration of exposure and the pH level all have a significant influence on the colour change caused by the hydrogen peroxide. Lower bleaching values were achieved at pH10. The rapid release of peroxide at pH 10 might explain these results. The bleaching takes place for a period (30 minutes) before the oxidation process releases oxygen in the atmosphere and less of it is available for the bleaching action. Lyer (2005) and Wang and Washington (2002) reported that hydrogen peroxide is an unstable chemical compound with the tendency to release the oxygen too rapidly into the atmosphere. At 70℃ at a pH10 the bleaching that took place in the first 90 minutes seems to be reversed when the samples were exposed to the hydrogen peroxide for a

TABLE 2: MULTIPLE REGRESSION REPORT OF COLOUR DIFFERENCE AS RESULT OF BLEACHING OF *GONOMETA POSTICA* SILK

Independent variable	**p-value**
Temperature	.000
Time duration	.000
pH	.000

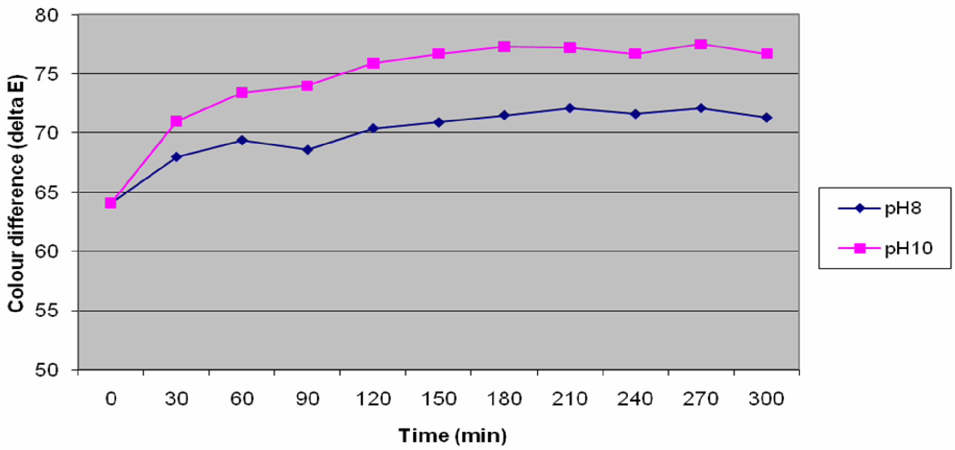

FIGURE 3 : THE COLOUR DIFFERENCE IN DELTA E OF *GONOMETA POSTICA* SILK BLEACHED WITH HYDROGEN PEROXIDE AT 50°C AT PH8 AND PH10

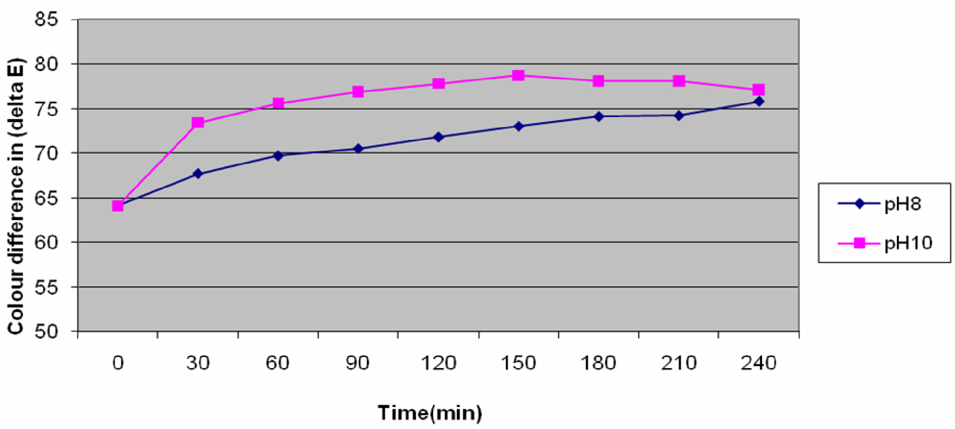

FIGURE 4 : THE COLOUR DIFFERENCE IN DELTA E OF *GONOMETA POSTICA* SILK BLEACHED WITH HYDROGEN PEROXIDE AT 60°C AT PH8 AND PH10

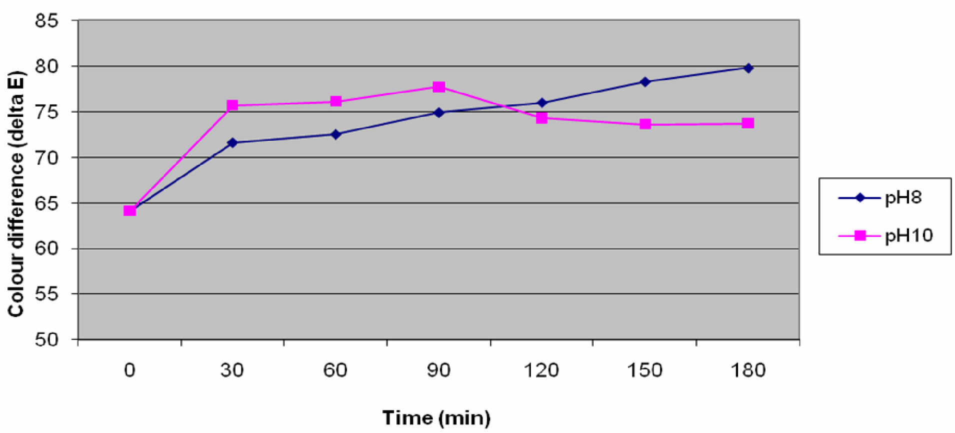

FIGURE 5 : THE COLOUR DIFFERENCE IN DELTA E OF *GONOMETA POSTICA* SILK BLEACHED WITH HYDROGEN PEROXIDE AT 70°C AT PH8 AND PH10

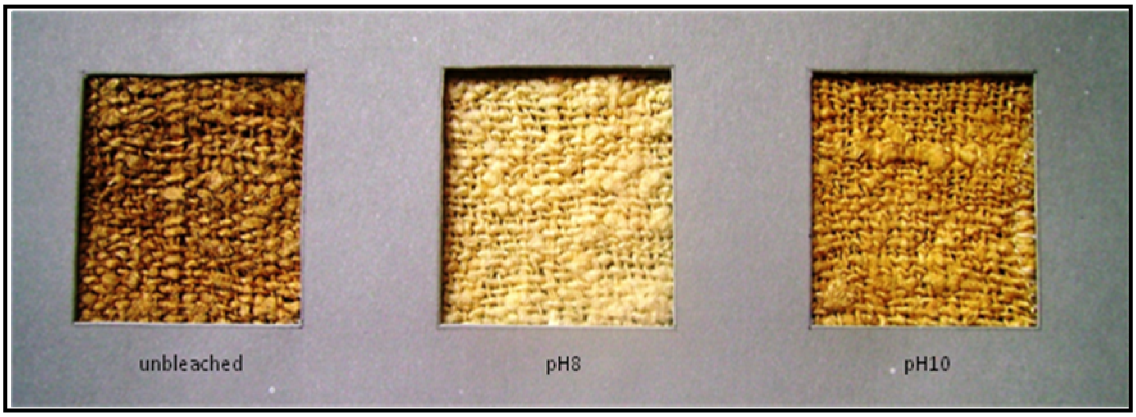

FIGURE 7: SAMPLES OF THE *GONOMETA POSTICA* SILK, UNBLEACHED, BLEACHED AT pH8 AND pH10

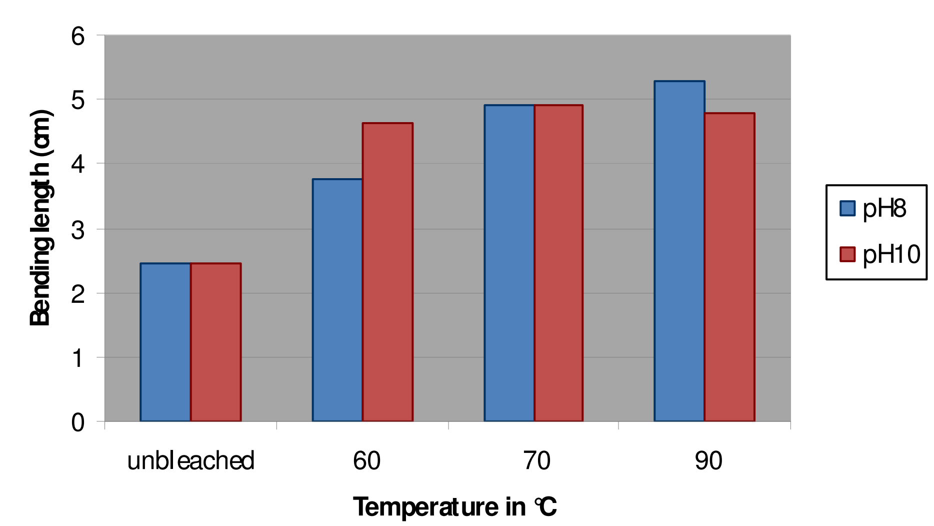

FIGURE 8: THE BENDING LENGTH OF *GONOMETA POSTICA* SILK UNBLEACHED AND BLEACHED WITH HYDROGEN PEROXIDE AT 60°C, 70°C AND 90°C AT pH 8 AND pH 10

TABLE 3: T-TEST REPORT OF THE BENDING LENGTH OF *GONOMETA POSTICA* SILK AFTER HYDROGEN PEROXIDE BLEACHING

Temperature	pH-values	p-value
60°C	8	.000
60°C	10	.000
70°C	8	.000
70°C	10	.000
90°C	8	.000
90°C	10	.000

longer period. Greenwood and Earnshaw (1998:635) claim that hydrogen peroxide can function as a oxidizing or as a reducing bleach. Bleaching with a reducing bleach tends not to be permanent. The instability of the bleach at the higher temperature and pH might have caused more oxygen in the bath which could have caused the partial return of the colour.

Stiffness and breaking load of *G. postica* silk fabric before and after bleaching

Stiffness The bending length of *G. postica* silk unbleached and bleached with hydrogen peroxide at 60°C, 70°C and 90°C at pH8 and pH10 is shown in Figure 8.

Bleached *G. postica* silk was more rigid than the unbleached fabric. The bending length of the un-

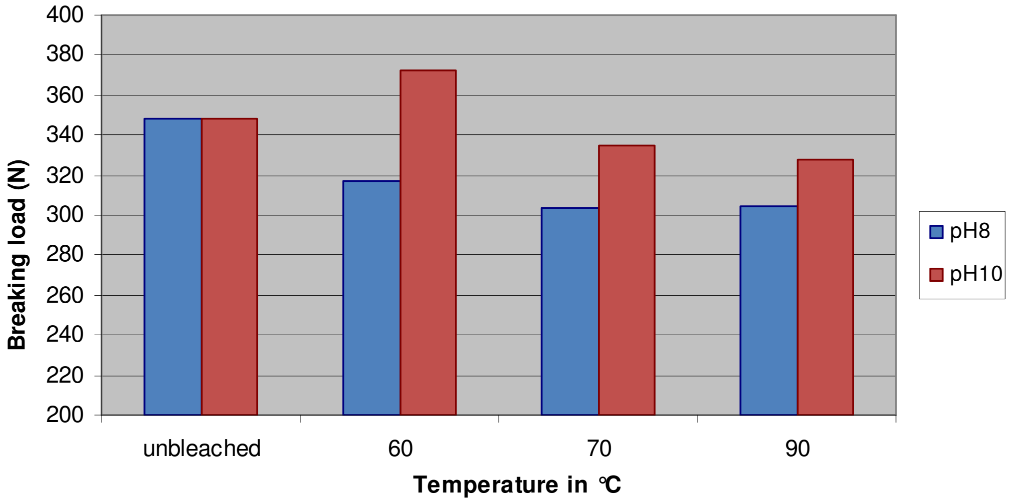

FIGURE 9: THE BREAKING LOAD OF *GONOMET POSTICA* SILK UNBLEACHED AND BLEACHED WITH HYDROGEN PEROXIDE AT 60°C, 70°C AND 90°C AT pH8 and pH10

TABLE 4: T-TEST REPORT OF THE TENACITY OF *GONOMETA POSTICA* SILK DURING BLEACHING

Temperature	pH-values	p-value of tenacity (N)
60°C	8	.000
60°C	10	.000
70°C	8	.000
70°C	10	.002
90°C	8	.000
90°C	10	.000

bleached *G. postica* silk fabric was 2.45 cm. After bleaching the bending length increased to 5.28 cm at 90°C at pH 8. Even bleaching at 60°C at pH 8 increased the bending length to 3.76 cm. The T-test report shown in Table 3 indicates that the combination of temperature and pH level significantly influenced the bending length of the fabric after hydrogen peroxide bleaching. These results were disappointing, as this rigidity causes a fabric to be too harsh for clothing and even for household textiles. This would restrict the end use of the bleached fabric to rugs, handbags and similar items.

Breaking load The load required to break *G. postica* silk fabric unbleached and bleached at 60°C, 70°C and 90°C is shown in Figure 9. Bleaching at 60°C and pH10 increased the load required to break the fabric with 7, 6%. All other bleaching temperatures caused a loss in the load required to break the fabric. This is a clear indication that the bleaching process caused damage to the strength of the fabric. The T-test report shown in Table 4 indicates that temperature and pH level were significant factors in the loss of load required to break the *G. postica* fabric. Bleaching at pH 10 caused 7% loss at 70°C and 7, 6% loss at 90°C. Bleaching at pH 8 caused a loss of 7,9% at 60°C, 12, 8% at 70°C and 12, 6% at 90°C. Amin *et al*, 1998; Wang & Ramaswamy, 2003; Kasimir, 1990, Gulrajani *et al*, 1992; James & Mackirdy 1990; Wynne, 1997:235 claim less than 10% loss in strength as a result of hydrogen bleaching in protein fibres. Bleaching at pH10 in this research would be in agreement with them.

CONCLUSIONS AND RECOMMENDATION

Gradual release of hydrogen peroxide took place at 50°C and 60°C. At 70°C and 90°C the hydrogen release took place rapidly.

The best bleaching (81 delta E) of the *G. postica* silk fabric was obtained with 60 minutes exposure to hydrogen peroxide at 90°C and pH 8. Exposure time of 180 minutes at 70°C and pH 8, however, gave comparable results (79.8 delta E), followed by 150 minutes at 60°C at pH10.

Hydrogen peroxide bleaching at pH 10 lowered the load required to break the *G. postica* silk fabric 7% at 70°C and 7,6% at 90°C, considered acceptable levels, but bleaching at pH 8 lowered the load required to break the fabric with 12,8% at 70°C and 12,6% at 90°C. The worst influence on the load required to break the fabric was thus experienced at 70°C and at 90°C at pH 8, where the best bleaching results were reached.

The rigidity of the *G. postica* silk fabric increased at

60℃, 70℃ and 90℃ at pH 8 and pH 10, the bending length was the highest (5.28 cm) after bleaching at 90℃ and pH 8, once again where the best bleaching results were reached.

Although acceptable bleaching of the *G. postica* silk fabric can be reached with 60 minute exposure to hydrogen peroxide at 90℃ and pH 8, or 180 minutes exposure at 70℃ and pH 8 bleaching of the fabric can not be recommended as bleaching causes the fabric to become too rigid for clothing or household items and 12, 6% or 12, 8% loss in the load required to break the fabric would render it too weak. Bleaching for 150 minutes at 60℃ at pH10 does less damage to the strength but still cause the *Gonometa postica* fabric to be stiff.

Gonometa postica silk fabric should be used in its natural golden brown colour or dyed in a darker colour until a bleaching practice that would not cause the fabric to be harsh and stiff is developed.

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