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Microwave Assisted Dyeing of Enzyme Treated Wool - A Comparative Study

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Abstract

The scaly structure of wool is responsible, to a great extent, for the tendency of wool to felt and shrink and its unique properties. Chlorination is a commonly used process to modify the scales of wool fibers with the purpose of providing resistance to felting and shrinkage but this process shows a number of drawbacks leading to the quest of ecologically clean alternatives. Enzyme processing is one such process. Conventional dyeing methods for wool require long dyeing periods of forty-five minutes and high energy consumption. Microwave assisted dyeing is an alternative dyeing method for wool fabrics saving on time, energy and cost. In the present study, an attempt was made to treat wool fabric with acid and alkaline protease enzyme. Enzyme treatment followed by microwave dyeing and their effects on physical properties including handle, weight, scanning electron microscope (SEM) imaging, tensile strength and color fastness (washing, rubbing, light and perspiration) properties of treated and untreated wool in comparison with conventionally dyed wool was under study. Untreated wool fiber showed fair handle, rough and sharp scales on the surface of fiber, however acid and alkaline enzyme treatment of the wool fabric showed improvement in softness, change in fabric weight, smoother surface scales. The broad conclusion of the research study is that alkaline enzyme treatment gave optimum results when compared to acid enzyme treated samples. On overall comparison, the fastness properties of both conventionally dyed and microwave-assisted dyed samples showed similar results, though it may be noted that the time for dyeing is lesser in microwave assisted dyeing. Thus, microwave assisted dyeing may be suggested as an alternative energy conserving method of dyeing but may find limited commercial viability.

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Introduction

Origin of the research problem: Environment and ecology occupy the most important place amongst the key focal issues faced by the world today. Natural fibers are eco-friendly and bio-degradable. Wool is a natural proteinic fiber that is obtained comes from the fleece of domesticated sheep. It is a natural, protein and a multi-cellular staple fiber. Wool consists of three major morphological parts: the cuticle, the cortex and medulla. The fiber form is composed of overlapping cells (scales) that surround the latter. This scaly structure is responsible for difficulty in dyeing of wool fibers and for its tendency to felt and shrink. This complex structure makes it difficult for the dye molecules to permeate into the fibers, resulting in low levels of dye exhaustion. A number of studies have featured in the literature that aims at improving the dye ability of wool by modifying the wool fiber. In recent years, modification and dyeing some of materials have been conducted under microwave assisted dyeing conditions http://www.hrpub.org

Microwave-assisted dyeing is one of the powerful techniques of non-heating contact. It is an alternative to conventional heating that is more rapid, uniform and efficient. It has been assumed that the microwave irradiation could affect dye ability of wool fabric. www.virginiafarm woolworks.com.au. Chlorination is one of the most common methods used to modify the scaly structure of wool fibers towards enhancing its texture. In today's times, chlorination process shows many drawbacks like: limited durability, poor handle, yellowing of wool most importantly its environmental impact. Thus, enzymatic treatment is gaining importance in textile technology. The bio-enzymatic process reduces the cost of processing and effluent treatment as compared to the chlorine process. www.patagonia.com/chlorine_free_wool.

Enzymes are natural protein molecules that act as highly efficient catalysts in biochemical reactions. Enzymes not only work efficiently and rapidly, but they are also biodegradable and eco-friendly. Thus, in this study the bio-protease enzymes treatment was used to modify the properties on wool fabric. Modification and enzyme treatment of wool alters the dyeability and changes the properties of wool due to the reduction in the number of scales.

Microwave assisted dyeing has shown to have enhanced the dyeability of wool reducing the dyeing and contact time of the substrate with the treating chemicals. The present study therefore aims at exploring microwave assisted dyeing of enzyme treated wool in comparison to conventional dyeing of untreated wool. The study involves examining the physical properties of wool fabric after enzyme treatment and its effect on dyeing with regards to dye-uptake and colorfastness.

Methodology

Raw Materials

Ready to Dye Wool fabric, Acid andAlkaline Protease enzymes, Acid Dye.

Enzyme Treatment

Enzyme treatment was carried out on woven woolen fabric using acid and alkaline protease enzyme. The following recipe was used for enzyme treatment of woolen fabric.

Process parameters	**Parameter Specifications (as recommended by the sponsoring industry)**	
	Acid enzyme treatment	**Alkali enzyme treatment**
Enzymes	1% (o.w.f.)	1% (o.w.f.)
M: L: R	1:20 (gms/ml)	1:20(gms/ml)
Time	60 min.	60 min.
Temp	40^0C	37^0 C
pH	4.7	8

The two-separate solution was prepared maintaining the material liquor ratio 1:20. In acid enzymes solution temperature was raised and maintained up to 40ºC. In alkaline solution temperature was raised and maintained up to 37ºC. Samples were added to two different solution and treated for 60 minutes. After that, the samples were taken out and dried at room temp.

Dyeing of wool fabric

The untreated and acid, alkaline enzyme treated wool fabric samples were dyed with acid dye using 2 % and 10 % shade in order to find out the effect of enzyme treatment. The following recipe given was used for dyeing of samples.

Conventional Dyeing

Process parameters	**Conventional Acid Dyeing Specifications 2% &10% (o.w.f.)**
Glauber's salt	15% (o.w.f.)
Sulphuric acid	4% (o.w.f.)
M: L: R	1:40 (gms/ml)
Time	45 min.
Temp	60^0C

Conventional Dyeing Procedure

The treated wool samples were dyed with acid dye (% shades -2 and 10) subsequent prior and post enzyme treatment in order to investigate the effect of enzyme treatment on dyeability of wool. Dye paste was prepared by dissolving the required amount of dye in a small amount of water, Glauber's salt and sulphuric acid. A material to liquor ratio of 1:40 was maintained. The samples were added to dye bath and dyeing was continued for 45 minutes. Temperature was raised and controlled at 60ºC with continuous stirring to ensure uniform dyeing. After dyeing the dyed samples were taken out from the dye bath, and rinsed thoroughly with water and were dried.

Microwave Dyeing

Process Parameter	**Microwave Assisted Dyeing Specification 2% & 10% (o.w.f.)**
Acid dye	2% (o.w.f.)
Glauber"s salt	15% (o.w.f.)
Sulphuric acid	4% (o.w.f.)
M: L: R	1:40 (gms/ml)
Time	20 min.
Power microwave	900W
Temp	65^0C-70^0C

Microwave Dyeing Procedure

The treated wool samples were dyed with acid dye (2 and 10% Shades) in order to find the effect on microwave assisted dyeing with enzyme treatment. Dye paste was prepared by dissolving required amount of dye in small amount of water, Glauber's salt and sulphuric acid. More water was added to make the dye solution, to keep the material to liquor ratio 1:40. The samples were dyed in a glass bowl in the microwave. The power of the microwave was maintained 900W for 20 minutes reaching a temperature upto 70°C. After dyeing the dyed samples were taken out from the microwave, rinsedthoroughly with water and dried.

Testing for physical properties

All the samples (untreated and enzyme treated) will be subjected to various tests for assessment physical properties in order to check the effect of treatment on these properties.

http://www.astm.org/Standards/textile-standards.htm

Sr. No.	Test	Purpose
1.	SpectrophotometerTest (AATCC:79-2010)	To determine K/S values for dye uptake
2.	Fabric Tensile Test (IS:1969(Part 2):2010/ISO 13934-2:1999)	To determine tensile strength of fabric
3.	Oven Dry Weight (ASTM D 2720-94 (2012))	To determine weight of fabric.
4.	SEM (Scanning Electron Microscope)	To scan longitudinal and cross-sectional view of fiber

Colour fastness testing of dyed samples

All the dyed samples were subjected to colour fastness tests which included:

S.No.	TEST	EQUIPMENT	TEST NUMBER
1	Colourfastness to washing	Launder-O-Meter	ISO -105, CO6, C1S 60°C
2	Colourfastness to rubbing	Crockmeter	AATCC-RA 38, 2005
3	Colourfastness to perspiration	Perspirometer	AATCC-RA 52, 2006

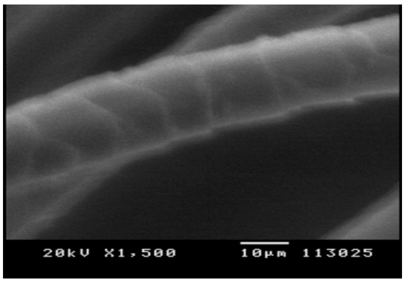
Results and Discussions

Tensile Strength

Fabric tensile strength test was carried out on "Tensile strength tester" and results are reported in the above Table. It shows that enzyme treatment increased in the tensile strength in both warp and weft direction.

Samples	Sample Parameter	Fabric strength N./mm(warp)	Fabric strength N./mm(weft)
Untreated	Undyed Sample	1.41	1.54
	2% Shade	1.41	1.54
	10% Shade	1.41	1.54
Enzyme Treated/Undyed	Acid	1.37	1.58
	Alkaline	1.48	**1.82**
Acid Enzyme Treated	Conventional 2% Shade	*1.27*	1.72
	Conventional 10% Shade	1.58	1.84
	Microwave 2% Shade	**1.68**	1.88
	Microwave 10% Shade	**1.68**	**1.92**
Alkaline Enzyme Treated	Conventional 2% Shade	1.45	1.66
	Conventional 10% Shade	**1.50**	1.72
	Microwave 2% Shade	1.43	1.76
	Microwave 10% Shade	**1.68**	**1.90**

Scanning Electron Microscope (SEM) Test



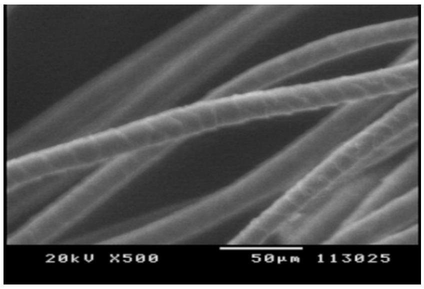


Plate 1: Untreated fibbers

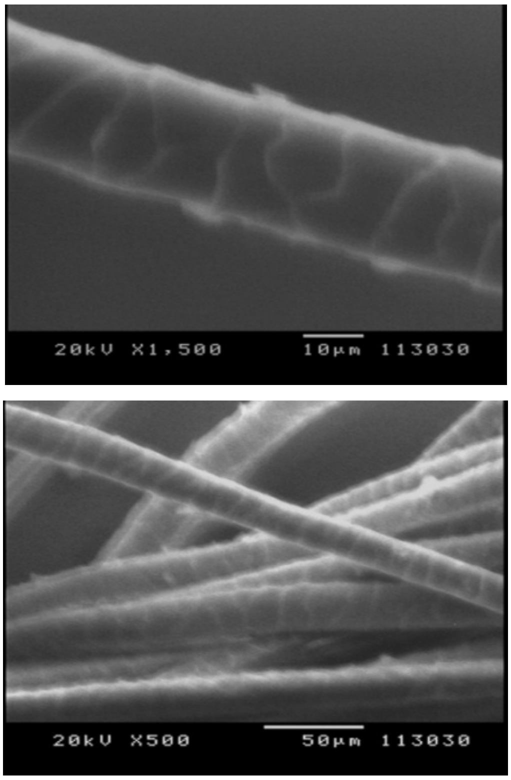


Plate 2: acid treated fibers

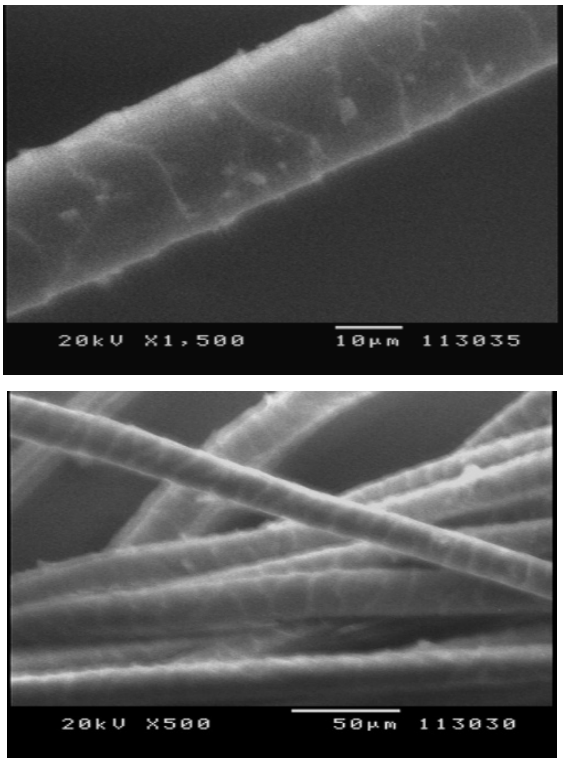


Plate 3: Alkaline treated fibers

Observation of Scanning Electronic Machine (Untreated & Treated Sample)

SEM test was carried out for untreated, and acid and alkaline enzyme treated and acid and alkaline treated with 10% microwave dyed samples fibers to study surface morphology of wool fibers.

SEM images showed that untreated wool fiber was rough and scales were sharp. And treated samples the surface scales of wool fiber got blunt and the smoothness of surface was found and subsequently resulted in improvement in the comfort factor of wool. The major changes seen in alkaline enzyme treated fibers.

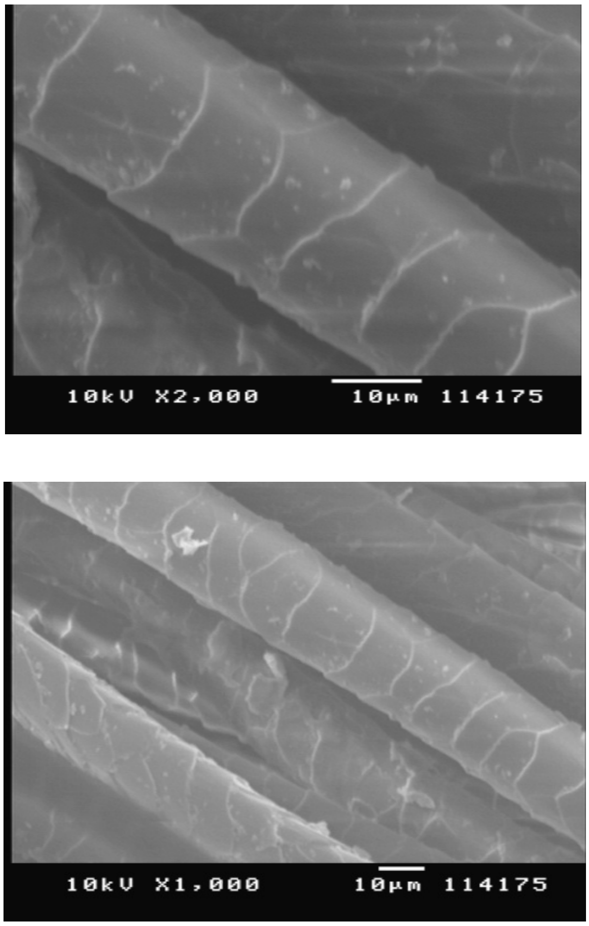
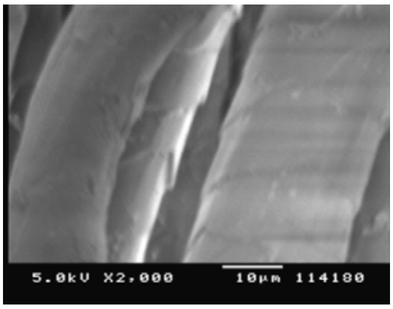


Plate 4: Acid Enzymes Treated Sample With 10% Microwave Dyeing



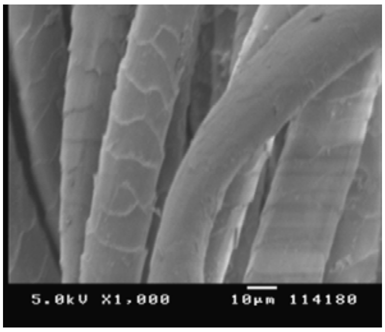


Plate 5: Alkaline Enzymes Treated Sample With 10% Microwave Dyeing

Observation of Scanning Electronic Machine (Microwave dyed Sample)

On close examination of the SEM images of acid and alkaline enzyme treated microwave dyed (10% shade) samples, it is observed that there is an increase in smoothness of the fibres with an apparent decrease in the scales on alkali enzyme treatment in comparison to acid treatment. Also on comparison of the undyed and dyed (10% shade) dyeing has further decreases the scales and the fibres appear smoother.

S. No.	Sample name	Colorfastness to washing		Colorfastness to rubbing				Colorfastness to perspiration			
	Sample name	Colour change	Colour staining	Dry		Wet		Change in colour		Staining (Cotton)	
				Change in colour	Staining	Change in colour	Staining	Acid	Alkaline	acid	Alkaline
Conventional	2% untreated	4	4	5	5	5	5	4	4	4	4
	10% untreated	3	2	4	3	5	5	4	4	4	3
Acid Enzyme Treatment	2% Shade conventional	4	4	4	3	5	5	5	5	5	5
	2% acid microwave	4	4	5	5	5	5	4	4	5	5
	10% acid conventional	4	3	4	3	5	5	5	5	4	4
	10% acid microwave	4	3	4	4	5	5	4	4	4	4
Alkaline Enzyme Treatment	2% alkaline conventional	4	3	5	5	5	5	4	4	5	5
	2%alkaline microwave	4	4	5	5	5	5	5	5	5	5
	10% alkaline conventional	4	3	4	4	5	5	4	4	4	4
	10%alkaline microwave	4	3	4	4	5	5	5	5	4	4

Weight Test: (ASTM D 2720-94 (2012))

S. No.	Sample name	Weight of sample
1	Untreated sample	2.097gm
2	Acidic enzyme treatment	1.999gm
3	Alkaline enzyme treated	2.236gm

It is evident from the table that untreated sample showed oven dried weight is average. Compared to acid enzyme treated samples showed minimum weight loss. And alkaline treated samples showed oven dried weight more compared to untreated and acid treated samples.

Colorfastness to washing

On observation of the ratings for colourfastness to washing, it ranges from good to excellent. There was slightly change in color seen and noticeable staining.

Results of Color Strength (K/S values) by Spectrophotometer

The 2% shade colour strength (K/S) increased on treatment with enzymes whether acid or alkaline. On comparing conventional and microwave dyeing acid enzyme treated samples showed a higher K/S Value.

2% Shade

Samples	Conventional dyeing K/S value	Microwave dyeing K/S value
Untreated Dyed	89.03	
Acid Enzyme Treated	134.87	143.23
Alkaline Enzyme Treated	166.79	114.06

10% Shade

The 10 % shade color strength (K/S) decreased on treatment with enzymes whether acid or alkaline.

Samples	Conventional dyeing K/S value	Microwave dyeing K/S value
10% untreated	381.54	
Acid treatment	334.66	317.39
Alkaline treatment	329.03	337.39

Conclusion

The fabric that was sourced for the study was identified as wool by using burning, microscopic and solubility tests. The given fabric was visually inspected and it was concluded that the fabric was plain weave (40x40 Fabric Count), off- white color and slightly rough and fuzzy textured compared to enzyme treated fabric that showed slight increase in smoothness.

The broad conclusion of the research study is that alkaline enzyme treatment gave optimum results when compared to acid enzyme treated samples. On overall comparison, the fastness properties of both conventionally dyed and microwave-assisted dyed samples showed similar results, though it may be noted that the time for dyeing is lesser in microwave assisted dyeing. Thus, microwave assisted dyeing may be suggested as an alternative energy conserving method of dyeing but may find limited commercial viability for dyeing large volumes of fabric.

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