THE HONG KONG POLYTECHNIC UNIVERSITY

INSTITUTE OF TEXTILES AND CLOTHING

# CHARACTERISATION AND APPLICATION OF NANO-SCALE FIBRE PARTICLES

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# Certificate of Originality

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April 2007

**TO MY PARENTS, FAMILIES,
TEACHERS AND FRIENDS**

**For Their Love, Encouragement and Support**

# Abstract

Fibre is a very important material in the textile industry. However, there is a large amount of wastage of fibres generated in textile industry daily. Scientists believe that the development of nanotechnology can allow the reuse of these discarded garments and fibres for creating a great market potential in the textile industry.

To achieve this goal, the newly innovative method was proposed for pulverising fibres into nano-scale particles. It was found that the rotate crusher, ultrasonic crusher, ultra-turrax® and nano-colliding machine were effective tools for pulverising the fibres including wool, cotton and silk fibres into micro-scale and nano-scale fibres particles. The pulverisation procedures were similar for various fibres except that the pulverisation duration was different. The size of the particles was confirmed to be in micro-scale and nano-scale by means of Scanning Electron Microscopy and Particle Size Analysis. The Fourier Transmittance Infrared Analysis illustrated that the chemical structures of micro-scale and nano-scale fibre particles were only slightly different from those of the original fibres. However, there is a gradual reduction in the crystallinity of the pulverised wool, silk and cotton particles with respect to the decreasing particle size.

Attempt had been conducted to coat nano-wool particles onto cotton and polyester fabrics aiming to modify their properties, functions and dyeing performance. The experimental results revealed that nano-wool particles could reduce the air permeability, thermal conductivity, keeping warmth ability, q-max value of both cotton and polyesters fabrics. On the other hand, there was a reduction in the OWTC, OMMC values, moisture management capability and hydrophility for the cotton fabrics, but there was an enhancement in these properties for the polyester fabrics after nano-wool treatment. In addition, the nano-wool treatment also caused the swelling, enhancement in wrinkle recovery and ultraviolet protection of cotton fabrics. In the nano-wool treatment, some parameters such as the amount of nano-wool added, dipping time, curing time and curing temperature did affect the properties and functions of different fabrics in various ways. As for the dyeing performance, it was found that the nano-wool treatment not only increased the colour depth of the dyed cotton and polyester fabrics, but also affected the tone of the dyed fabrics slightly.

# Publications

## <<Referee Journal>>

1. Y. F. Cheng, Y. Li, C. W. M. Yuen and J. Y. Hu, *“Enhancement of Ultraviolet Protection and Wrinkle Recovery of Cotton Fabrics by Nano-Scale Wool Powder”* AATCC Review of the Textile Dyeing, Printing, and Finishing Industry, 2006, vol. 6, no. 11, November 2006, p.41-43.

2. Y. F. Cheng, C. W. M. Yuen, Y. Li, S.K. A. Ku, C. W. Kan and J. Y. Hu, *“Characterisation of Nano-Scale Wool Particles”*, Journal of Applied Polymer Science, published online on 25 Jan 2007, vol. 104, issue. 2, p.803-808.

3. Y.F. Cheng, C.W.M. Yuen, Y. Li, S.K.A. Ku, C.W. Kan, and J.Y. Hu, *“Modification of Thermal and Liquid Transfer Properties of Cotton Fabric by Nano-Wool Powder”*, has been submitted to Fibers and Polymers.

4. Y.F. Cheng, C.W.M. Yuen, Y. Li, S.K.A. Ku and C.W. Kan, *“Influence of Nano-Wool Powder on the Dyeing Properties of Cotton Fabrics”*, will be submitted to Colouration Technology.

5. Y.F. Cheng, C.W.M. Yuen, Y. Li, S.K.A. Ku and C.W. Kan, *“Influence of Nano-Wool Powder on the Dyeing Properties of Polyester Fabrics”*, will be submitted to Colouration Technology.

6. Y. Li, C.W.M. Yuen, J.Y. Hu and Y.F. Cheng, *"Analysis of Structural Characteristics of Nano-Scale Silk Particles"*, Journal of Applied Polymer Science, 2006, vol. 100, no. 1, p. 268-274.

7. C.W.M. Yuen, Y. Li, J.Y. Hu and Y.F. Cheng, *"Characterization of Nano-Scale Cotton Particles"*, has been submitted to Journal of the Textile Institute and is under review.

## <<Patent>>

1. Y. Li, J.Y. Hu, Y.F. Cheng, K.W. YEUNG and C.W.M. Yuen, *"Method of Treating Fabrics and Fibers"*, US patent office, Filing Number: 11/053,291, Filling Date: 8th February 2005.

2. C.W.M. Yuen, Y.F. Cheng, S.K.A. Ku and C.W. Kan, *"Modified Textile Materials and Manufacturing Method thereof"*, Chinese Patent Office, Filling No: 200610121379.7, Filling Date: 21st August 2006.

3. C.W.M. Yuen, Y.F. Cheng, S.K.A. Ku, and C.W, KAN, *"Method for Pulverization of Polyester Filaments into Micro- and Nano-Scale Fibrous Materials"*, Chinese Patent Office, Filling No. 200510086043.7, Filling Date: 19th July 2005.

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# Chapter 1 Introduction

## 1.1. Background of Study

Fibre is a very important elemental material in the textile industry. Owing to the limitation and requirement of spinning, it is sometimes very difficult to spin too short or too weak fibres into yarns. As a result, these fibres have to be disposed during the spinning process. On the other hand, many garments are discarded daily all over the world. Reusing these discarded garments and fibres will create a great market potential for the textile industry.

Nowadays, textile fibres have gained a great deal of attention because of their great potential to be used as a multi-functional material in various application areas such as biotechnological and biomedical fields. In order to fulfil these new applications, pulverisation of fibres into particle form is necessary. Hitherto, some researchers have invented different ways to pulverise the natural fibres into particles and apply them to various fields. However, no one has ever tried to apply the fibre particles onto the fabrics to modify the properties of the fabrics. The aim of this project is to pulverise the fibres into nano-scale particles and then apply them onto the fabrics in order to study the nano-scale effect on the properties, function and dyeing performance of fabrics.

## 1.2. Research Aim and Objectives

Nowadays, some research works have been conducted to pulverise natural fibres into particles aiming to explore the feasibility of their new application. Moreover, the application of nanotechnology in the textile industry has been initiated. Hitherto, it seems that no one has ever attempted to combine nanotechnology with pulverisation of fibres into nano-scale for textile finishing. This project is thus proposed in an attempt to combine nanotechnology with pulverisation of fibres together.

The objectives of the project are:

i To invent special method for pulverising fibres into nano-scale particles.

ii To study and compare the characteristic and structure of micro-scale and nano-scale fibre particles.

iii To investigate the feasibility of using nano-scale fibre particles in textile finishing.

iv To explore the recipe and technology for treating fabrics with nano-scale fibre particles.

v To determine the dyeing properties of fabrics treated with nano-scale fibre particles.

## 1.3. Scope of Study

The scope of study is mainly divided into four areas which are summarised as follows.

Firstly, the knowledge of nanotechnology will be introduced first followed by the review of fibre particles manufacturing method and application.

Secondly, the newly invented procedures for pulverising wool, silk and cotton fibres into nano-scale particles will be proposed followed by the study of the morphology, chemical and structural characterisation of the pulverised particles.

Thirdly, attempts will be made to apply the nano-scaled fibre particles directly onto cotton and polyester fabrics for modifying the properties and functions of fabrics.

Finally, the effect of nano-wool treatment on the dyeing performance of cotton and polyester fabrics will be studied.

## 1.4 Significance and Values

Some of the project significance and value are identified as follows:

i The pulverisation methods used for converting cotton, wool and silk fibres into nano-scale particles have been successfully developed. These methods provide a way to recycle and reuse discarded and useless fibres.

ii The morphology, particle size, chemical and structural changes of the nano-scaled fibre particles have been systemically evaluated and studied for developing new nano-products.

iii The methods of applying the nano-wool particles onto the pure cotton and polyester have been explored to modify the properties and functions of the fabrics.

iv The methods of applying the nano-wool particles onto the pure cotton and polyester for making alteration of the dyeing properties of the treated fabrics have been investigated.

## 1.5 Arrangement of Thesis

This thesis comprises 7 chapters. Chapter 1 introduces the background, research aim and objectives, scope of study, significance and values of this research. A brief outline of the thesis is also provided in this chapter.

Chapter 2 is literature review that gives an introduction to nanotechnology and a review of fibre particles manufacturing method and application in textile industry. In this chapter, the definition, properties, advantages and applications of nanotechnology in textile field are briefly described. This chapter also mentions the fibre particles manufacturing method and its application in textile field.

Chapter 3 is focused on the study of the pulverisation method and characterisation of nano-scaled fibre particles. The newly invented procedures for pulverising wool, silk and cotton fibres into nano-scale particles will be proposed followed by the study of the morphology, chemical and structural characterisation of the pulverised particles.

Chapters 4 and 5 put emphasis on the study of the modification of properties and functions of cotton and polyester fabrics treated by nano-wool particles. The procedures of applying the nano-wool particles directly onto cotton and polyester fabrics are briefly introduced. The properties and functions of the nano-wool treated cotton and polyester fabrics were evaluated and compared with those of the untreated one. The preliminary experiments indicated that nano-wool particles were effective in modifying the properties of fabrics.

In Chapter 6, the dyeing performance of the untreated control and nano-wool treated cotton and polyester fabrics was compared using reactive dye and disperse dye for the dyeing of the cotton and polyester fabrics respectively.

Chapter 7 provides general conclusions of the present study. Recommendations for further study are also proposed in this chapter.

# Chapter 2 Literature Review

## 2.1. Nanotechnology

Recently, nanotechnology has raised attention and become popular all over the word. Many people from both the science and manufacture fields are willing to gain benefit from the unique properties of nano-materials. Nanotechnology has the impact on textiles all the way from fibres to end products. Fibres, finishes, dyeing and colouration system as well as textile composites have been influenced by nanotechnology either in current applications or are in research to develop new applications (Thiry, 2004). Nano-fibre is one of the nano-materials in textile industry. These nano-fibres are mainly manufactured during the electrospinning process. They are in the form of long fibres with one of the dimension, i.e. the diameter of the cross-section, less than 100nm. The nano-fibre materials mentioned in the project, however, are created by a new method. The materials generated by this new method are in particle form rather than those in long fibre form manufactured by the electrospinning method. In this section, the definition, properties and application of nano-materials and nanotechnology in textile industry are introduced.

### 2.1.1. Definition of Nano and Nanotechnology

The term ‘nanotechnolgy’ was first originated by Norio Taniguchi at the University of Tokyo in 1974. Nanotechnology refers to numerous rapid emerging technologies based upon the scaling down of the existing technologies to the next level of precision and miniaturisation (Ghosh T., 2005). Nanotechnology is an emerging area of research and development devoted to the increasing control over material structures of nanometre size (0.1 to 100nm) (Li Y. *et al*., 2003). Nanotechnology, therefore, deals with materials, process and technology on the scales of atoms and molecules, i.e. between 0.1 and 100 nanometres (Thiry M. C., 2004).

Nano is a derivation from the Greek word nanos, meaning dwarf, and in basic SI units. The prefix nano is used as a factor representing $10^{-9}$m. By making comparison, the diameter of a single human hair is about 80,000nm, a human red blood cell is about 7000nm wide, and a water molecule is approximately 0.3 nm wide (Holme I., 2005) as shown in Figure 2.1.

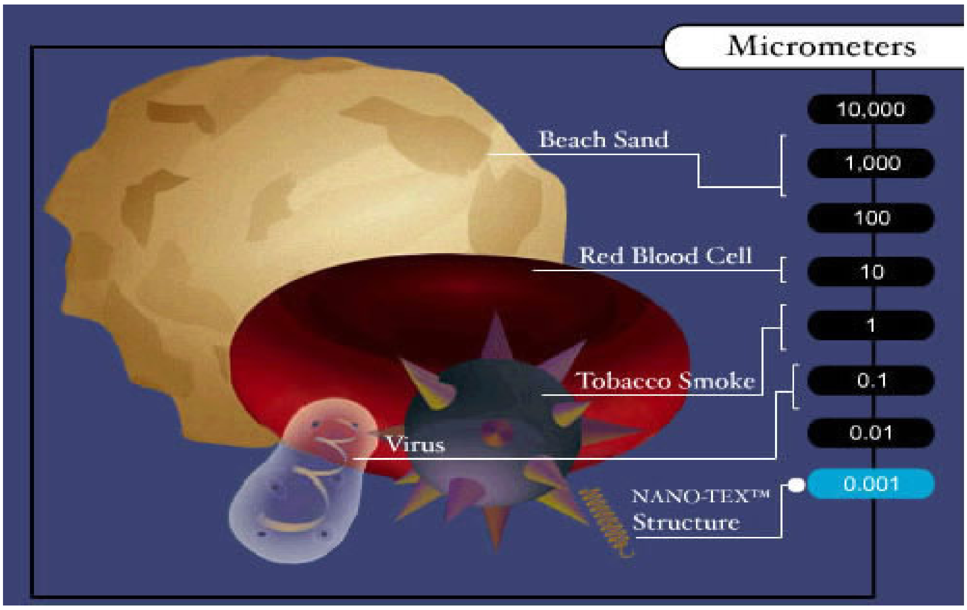

Figure 2.1. Comparison of the size of nano-scaled materials with different materials (Nano-tex, 2003)

### 2.1.2. Novel Properties of Nano

When materials reach the nano-scale, their physical properties will change. This is the reason why nanotechnology is so exciting to researchers and venture capitalists. The ability to alter the physical properties of materials gives nanotechnology an enormous potential impact across various disciplines (Thiry M.C., 2004).

The extraordinary behaviour of nano-scaled materials is due to the high surface to volume ratio, strong attractive forces and quantum effects etc, which will result in the development of novel mechanical, chemical, optical, electronic or magnetic properties of nano-materials that are not predictable from the bulk material. In textile materials, the lotus effect exhibited by the nano-materials also has great influence on the properties of the nano-treated materials.

The surface and volume effects are useful in implementing unique properties of nano-materials. For the surface effect, nano-materials have a relatively larger surface area when compared with the same mass of material produced in a larger form. Hence, surface effect is important in nano-materials, meaning that along with the reduction in the particle diameter of the materials, the scale of surface atoms will be enlarged while the surface energy will be heightened and activated (Li. Y. *et al*, 2003). The enhancement of surface activities and attractive forces by the high percentage of surface atoms affect many properties of materials in such nano size range. The special highly exposed surface structure stimulates and promotes chemical reaction (Subramanian M. *et al*, 2004). One consequence of the increase in surface area is that some materials which are normally inert in their larger form will become reactive in their nano-scale form (Holme I., 2005). For the volume effect, the micro and macro features of surface atoms of such materials will undergo abnormal changes caused by the separation of the electronic energy levels, widening of energy gaps, alternation of crystal patterns and decrease of the density of the surface atoms (Li Y, *et al*., 2003). Due to both surface and volume effects, it is possible that nanotechnology can be applied to produce various innovative products that have unique and superior functional properties (Subramanian M. 2004).

Apart from the surface and volume effects, the finite size of particles in nano-scale confines the spatial distribution of the electrons, leading to the quantised energy levels due to size effect. Materials exhibit new feature and function determined by the rules of the quantum world, i.e. rules that govern at the nanoscale. Quantum nanoscience is the application of quantum theory to the design of new nanoscale materials. Quantum theory is defined as the modern physical theory concerned with (i) the emission and absorption of energy by matter and (ii) the motion of material particles. The quantum theory and the theory of relativity together form the theoretical basis of modern physics. Similar to the theory of relativity which assumes the importance of the special situation where very large speeds are involved, the quantum theory is necessary for the special situation where very small quantities are involved, i.e. up to the scale of molecules, atoms, and elementary particles. Aspects of the quantum theory have provoked vigorous philosophical debates concerning, the uncertainty principle and the statistical nature of all the predictions of the theory (High Beam TM Encyclopedia Research Inc, 2007). Quantum Nanoscience explains the functionality and structure in natural or engineered nano-scaled systems through quantum mechanisms such as discretisation, superposition and entanglement. Quantum nanoscience is an enabling science for engineering and design of new nanotechnologies (Department of Physics, The University of Queensland, 2003).

The ‘lotus effect’ is an important feature of nanotechnology for textile materials. When nano-materials are applied onto textile materials, a very fine surface structure will be implemented on the textile surface, just like the lotus leaves which are coated with hydrophobic wax crystals of around 1 nm in diameter. The nano-textured surfaces will bring the modified surface tension to textile and enhance the roughness of the surface. The rough surfaces induced by nano-materials are more hydrophobic than smooth surfaces. Hence, when textile is coated with nano-substrates on its surface, water-resistant and self-cleaning functions can be obtained (Chan E., 2004) as shown in Figures 2.2(a) and (b).

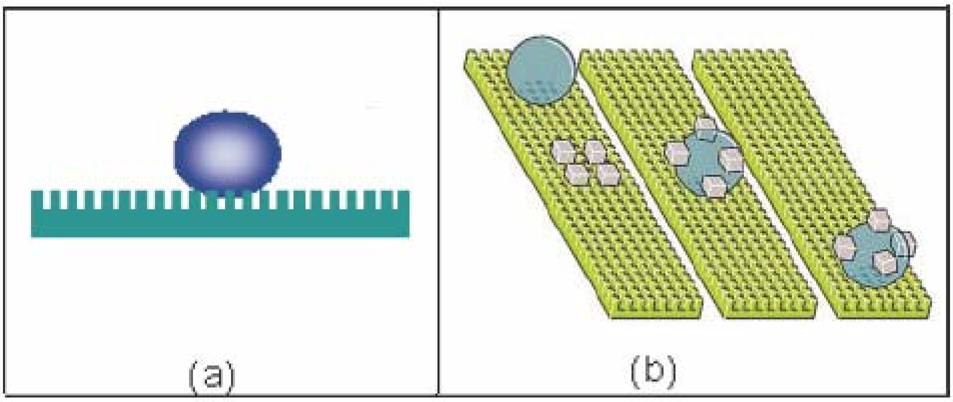

Figure 2.2 (a) Nano-scaled roughness inducing water repellency surface by lotus effect and (b) surface roughness and self-cleaning by rinsing with water (Baumann M. et. al., 2003)

## 2.1.3. Advantages of Nanotechnology

The unique properties of the nano-materials exhibit numerous advantages which make nanotechnology so popular. In this part, several advantages of the nanotechnology are listed and summarised in the following.

### *Reducing Manufacturing Costs and Wastes*

Nanotechnology enables manufacturers to reduce the cost involved in resources and waste during the production process because it permits the use of smaller amount of particles to create production materials which possess similar properties of the materials created through the conventional methods (Chan E., 2004). In addition, nano-products are more durable resulting in the reduction in manpower, time and required detergent for laundering (Sophie Lucie Dewulf 2004).

### *Light Weight*

Nano-fibres, nano-finishes and nano-fabrics are less heavy as compared to the traditional one due to its small size (Sophie Lucie Dewulf 2004). Since nanotechnology provides the potential for minimising the weight of materials, thus the US Army Research Office's has the goal of creating a new generation of uniforms for soldiers with the aid of nanotechnology to reduce the weight of uniform that a soldier must carry in the field (Thiry M.C., 2004).

### *Direct Application*

Chemical nano textile treatments can be applied directly around the fibre or agent (Sophie Lucie Dewulf 2004).

### *Permanent Bond and Easy-Care*

Nano-treatments are more highly durable and resistant to harsh washings and wearing than the standard finishes (Sophie Lucie Dewulf, 2004). Since the treatments bond to the fibres on a nano-level and become inherent in the fabric, thus the durability is increased tremendously and the performance lasts for the life of the garment (Thiry M.C., 2004).

### *Minimal Changes in Colour and Hand Feel*

After the nano treatment, each individual fibre was protected by the nano-materials through sophisticated molecular engineering on the nano-metre scale with minimal impact on the hand feel and drape of the fabric. (Thiry M.C., 2004) Nano-finishing, e.g., Texcote's finish, is a kind of unnoticeable, odourless and good colour fastness treatment (Sophie Lucie Dewulf 2004).

***No Need for New Equipment***

Traditional existing machinery can be used to apply the nano-finishes onto fabrics as the nano-finishes are water-based treatment. Mass production with nano-finishes is thus possible (Sophie Lucie Dewulf 2004).

***Environmentally Friendly and Non-Toxic***

Nanotech finishes are free of hazardous substances. They also require less energy in drying and save energy due to its rapid drying features (Sophie-Lucie Dewulf, 2004).

### 2.1.4. Application of Nanotechnology in Textile Field

The novel advantages and unique properties of nano-materials make nanotechnology become popular in different areas. In textile area, the exploration of nanotechnology has widely been developed (Wu Jinqian, 2005).

The textile sector is a promising area for the development and application of nanotechnology. At least 13 countries have initiated applications of nanotechnology in the textile industry with a continuous increase in the number of patents and publications. US and Europe are the major important research centres and markets for textile products made with nanotechnology. Some Asian countries have recently started to pay more focus on their development (Yuen C.W.M. *et al*., 2005 and Li Y. *et al*., 2003).

In recent years, US, Japan and Korea have invested US$982 million, US$875 million and US$237 million respectively in research and development of nanotechnology (Thiry M.C., 2004, Chan E. 2004).

In Hong Kong, investment on nanotechnology research is only about US$12.8 million. Two academic institutes, namely the Hong Kong University of Science and Technology and The Hong Kong Polytechnic University, have paid special attention to this research area (Chan E. 2004).

Although commercial nano textile products are already available in Europe, US, Japan, South Korea, Taiwan, Hong Kong and China, they are comparatively expensive. Some researchers reported that the global demand for nano textiles would experience a fast growth in the next nine years (Wu Jinqian, 2005). It is predicted that the worldwide demand for nano textiles will reach US$15 billion by 2007. Textiles experience the impact of nanotechnology all the way from fibres to end products. Fibres, finishes, dyeing and colouration system as well as textile composites have been affected by nanotechnology either in current applications or in exploration research stages. The application of nanotechnology in textile products is currently emphasised on the improvement of functionality, product value and quality (Wu Jinqian, 2005).

At present, the application of most developed nanotechnology for textiles is in the area of nano-finishes. Nano-emulsification is used in traditional finishes as it benefits the treatments by providing even more application of the chemistry to the fabric (Thiry M. C, 2004).

Nanotechnology presently has greatest impact on textile finishes in the areas of stain, oil and water repellency; soil resistance, stain release and wrinkle resistance. In addition, nano-finishes can render additional functionality to the manufacture of speciality fabrics such as antistatic, flame retardant, anti-ultraviolet ray, infrared absorbent or repellent, anti-microbial, anti-bacterial, deodorising, smear resistant, temperature and thermal regulation, electromagnetic shielding (wave absorption), dyeability, colour fastness and strength of textile materials with the maintenance of the breathability of fabric. Other nano-finishes change the surface properties of the fabric itself such as hand feel, moisture management and smoothness (Wu Jinqian, 2005, Thiry M. C, 2004 and Yuen C.W.M et. al., 2005).

Many companies have come out with finishes based on nanotechnology that are being promoted as superior finishes when compared with the conventional one, some examples of such companies are listed in the following.

Nano-Tex has developed finishes for garments including (i) Resists Spills – Enhancement in spill resistance for many kinds of fabrics and fibres for repelling various liquids, (ii) Coolest Comfort – Quick-absorbing fabric enhancement for providing superior wicking properties to synthetic fabrics by carrying perspiration away from the body and drying it quickly to keep the body cool and comfortable, (iii) Cotton Touch – Naturally soft fabric enhancement designed to make synthetic fabric look and feel like cotton. Nano-Tex has also developed the nanotechnology based products of Nano-care; Nano-Dry; Nano-Pel and Nano-Touch (Thiry M. C., 2004, Lee E., 2002 and Harper Tim, 2004).

BASF has the approach to develop super-hydrophobic materials and coatings by combining the nano-particles with strongly hydrophobic polymers (Thiry M. C., 2004, Lee E., 2002 and Harper Tim, 2004).

Bugatti (Germany) provides men jackets made of Schoeller’s NanoSphere-liquid and stain repellent fabrics called Schoeller Textil AG Bugatti. The NanoSphere® finishing technology is based on the self-cleaning principle and is almost a perfect copy of nature. This finishing prevents different stains like ketchup, honey, coffee, red wine, oil and grease, as well as water from remaining on the fabric surface, and makes these kinds of stains just simply run off the nano-surface of the fabrics. This stain repellency can be maintained even after numerous laundering. NanoSphere manages to transfer a lotus leaf finely-structured 3-dimensional surface, which has non-stick and self-cleaning characteristics, onto a textile for inducing the dirt and oil repellent properties for the textiles, yet being robust and abrasion-resistant (Sophie Lucie Dewulf, 2004, Thiry M. C., 2004, Lee E., 2002 and Harper Tim, 2004).

According to the Texnology Nano Textile Ltd (China), Texcote processing technology is used for imparting water repellency and stain resistance, guaranteeing breathability and colour fastness properties for different textile materials such as cotton, silk, wool, linen and polyester fabrics. Other benefits of Texcote-processing treatment include air permeability, easy of care and environmental friendliness (Sophie Lucie Dewulf, 2004, Thiry M. C., 2004, Lee E., 2002 and Harper Tim, 2004).

In the case of the Shin Han Industrial Co. Ltd (Korea), Nanoplus finish provides water/oil repellency properties and protection against spills and stains, delivering durable, long lasting easy care textile protection for the treated fabrics (Sophie Lucie Dewulf, 2004, Thiry M. C., 2004, Lee E., 2002 and Harper Tim, 2004).

New Lauch is the Gamex Compact range developed by Eschler (Switzerland) which provide nanotech stain repellent finishes for sportsgear (Thiry M. C., 2004, Lee E., 2002 and Harper Tim, 2004).

Nyacol ® Nano Technologies is the leader in colloidal dispersions and nano-particles technology. The products include flame-retardants for textiles and plastics, UV absorbers and acid scavengers, anti-block and abrasion resistance additives for films and colloidal dispersions for catalysts (Thiry M. C., 2004, Lee E., 2002 and Harper Tim, 2004).

Toray Industries Inc (Japan) offers nano-tech developed fabrics with effective moisture absorption properties (Thiry M. C., 2004, Lee E., 2002 and Harper Tim, 2004).

Hong Kong-based GP NanoTechnology Group Ltd has recently applied nanotechnology to create a soil resistant and water repellent finishing agent (Thiry M. C., 2004, Lee E., 2002 and Harper Tim, 2004).

Anson Nanotechnology Group Co. Ltd (ANG) is a Hong Kong-based company pioneering in the research and development of nanotechnology. Textile and non-woven fabrics include baby diaper, household cleaning materials, female hygiene products and filtering materials which are the four major products groups developed by the ANG's research group (Thiry M. C., 2004, Lee E., 2002 and Harper Tim, 2004).

Mammut (Switzerland) manufactures mountain and active outdoor wear made of Schoeller's NanoSphere textiles (Sophie Lucie Dewulf, 2004, Thiry M. C., 2004, Lee E., 2002 and Harper Tim, 2004).

Levi's (USA) introduces Docker's ProStyle line of casual business for men's wear by using the strain-repellent Nano-Tex fabrics (Thiry M. C., 2004, Lee E., 2002 and Harper Tim, 2004).

Eschler, a Swiss textile manufacturer, started using the nano chemical anti-dirt treatment a season ago. It is believed that the key advantages of the treatment are durability, easy and direct application without using the binding agent. The newly developed anti-dirt treatment has improved performance, e.g. soil release and advanced water repellency, as compared to the standard traditional treatments with increase in the fabric's regular price tag (Sophie Lucie Dewulf, 2004, Thiry M. C., 2004, Lee E., 2002 and Harper Tim, 2004).

Besides the application of nanotechnology in textile finishing with the advancement in the textile field, nanotechnology can be applied to Smart Textiles like:

(i) Self-cleaning fabrics - Robotic devices similar to mites can periodically scour or wash the fabric surfaces, and integral conveyers are responsible for transporting dirt to a common collection site where the dirt can be easily washed away. Recently, The Hong Kong Polytechnic University has reported its nano-coated clothing with self-cleaning functions. The self-cleaning fabric coated with titanium dioxide can remove dirt, pollutants and bacteria by itself under sunlight. This could be applied onto clothing and even furniture (Tirtha G.., 2005, Chan E., 2004 and Yuen C.W.M *et al*., 2005).

(ii) Self-repairing fabrics - In the self-repairing fabrics, sensors will detect discontinuities in the material via the loss of signal and send robotic ‘crews’ to repair the damage. Self-shaping fabrics will have the ability to return to their original shape around a tear until repairs are affected (Tirtha G.., 2005, Chan E., 2004 and Yuen C.W.M *et al*., 2005).

Furthermore, there are other research areas applying nanotechnology in textile field such as nano-fibre and nano-filtration. Nano-fibres are linear solid nano-materials having the flexibility characteristics and a diameter equal to or less than 100nm. Nano-fibre technology is an important branch of the growing nanotechnology. The enormous availability of surface area per unit mass is one of the most important properties of nano-fibre. The high surface area of nano-fibres provides a significant capacity for the attachment or release of functional groups, absorbed molecules, ions, catalytic moieties, and nano-meter scale particles of many kinds. Nano-fibres with high surface area and porosity have various applications; e.g. filter media and adsorption layers in protective clothing. Nanofibre materials are mainly produced by electrospinning and other methods such as phase separation (Sophie Lucie Dewulf, 2004 and Yuen C.W.M *et al.*, 2005).

Electrospinning is the best method for the preparation of polymer nano-fibres. The process involves the use of a strong electric field applied to a syringe-shaped die connected to a reservoir which contains a polymer solution or polymer melt, a ground plate and the counter electrode. By means of a proper selection of the processing parameter, the diameter of the fibres can be controlled within a broad range down to a few nm. The polymer solution is pulled by the charge through the air into a tiny fibre, which is collected on an electrical ground. The diameter of the fibre produced is less than 100 nm, which is 1,000 times smaller than that of the conventional spinning. The production of nano-fibres provides a good way to recycle materials at all phases of textile production and remove them from the waste stream. Hence, it is possible to produce a low cost, high-value, high-strength fibres from a biodegradable and renewable waste product (Subramanian M. 2004).

Nowadays, nano-fibre materials have been under very good development. For example, Kanebo Spinning Corp. (Japan) has created the polyester nano-fibre which has high water-absorption properties absorbing moisture 30 times more than the standard polyester. Teijin Fibres Ltd. (Japan) manufactures luminescent nanotech-polyester fibres. eSpin (Japan) provides custom-made nano-fibres with light weight, high-strength and multi-functional properties like fire retardant and anti-bacterial protection etc. (Sophie Lucie Dewulf 2004).

Besides the commercial company, government organisation also has interest in the application of nanotechnology in textile materials. For example, in the Fall of 2001, the US Army Research Office (ARO) announced its intention to set up a University Affiliated Research Centre (UARC) for developing nano-scale science and technology solution to help design new equipment and uniforms for the soldiers of the future (Thiry M. C., 2003).

In this research, works have been done to study the method and application of nano-scaled fibre materials. The nano-scaled fibre materials mentioned in this thesis are in particle form with the dimensions less than 100nm. The method to prepare the nano-scaled fibre particles is also different from the aforementioned electrospinning.

The concept of this project involves both the nanotechnology and fibre particles concept. After studying the usage of nanotechnology in textile field, the literature review of fibre particles manufacturing method will be reported in the following section.

## 2.2. Pulverisation of Natural Fibres

In addition to the application in clothing, textile fibres have been used as a multi-functional material in various application areas such as bio-technological and bio-medical fields. In order to fulfil these new applications, pulverisation of textile fibres is necessary. Hitherto, some researchers have invented different ways to pulverise the natural fibres into powders and applied them to various fields. However, no one has ever attempted to pulverise these natural fibres into nano-scaled particles. In the following sections, the pulverisation of natural fibres including wool, cotton and silk done by different researchers is reviewed.

### 2.2.1. Pulverisation of Wool Fibres

Wool is the fibre obtained from the fleece of sheep. It is a natural multi-cellular staple fibre composing of proteins and organic substances with the composition of carbon, hydrogen, oxygen, nitrogen and sulphur (The Hong Kong Cotton Spinners

Association 2000). The length of wool is dependent on the breed of sheep and the growing time of the fibre. In general, fibre length ranges from 20 to 300 millimetres with shorter fibres usually being finer and longer fibres usually being coarser. The diameter of wool fibres ranges from 8 to 70 microns. The width of Merino fleece fibres is usually 15 to 17 microns.

In cross section, wool fibre is oval or elliptical composing of three parts namely (i) Cuticle (ii) Cortex and (iii) Medulla. Cuticle is the outer layer consisting of a fine network of small overlapping scales. This scale structure is responsible for the felting and shrinkage behaviour of wool. Cortex makes up most of the fibre and is composed of microscopic cells that pack this area. Medulla is the section that provides air space for carrying the pigment or colourants. The majority of finer wool do not have a medulla (Collier 2001).

As shown in Figure 2.3, the structure of wool fibre includes microfibril (about 7 nm in diameter), marcofibril (about 200 nm in diameter) and cuticle (about 20,000 nm in diameter).

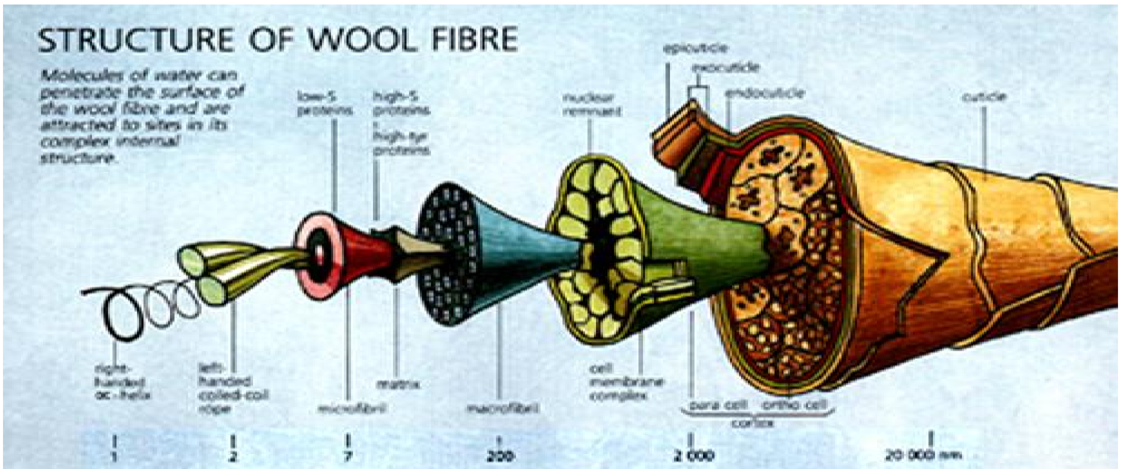

Figure 2.3. Structure of wool fibre (Apparel products group, IWS development centre)

Generating particles from animal hair, especially wool, may provide more new application for the fibre. In recent years, some researches are being conducted to explore this area. For example, Yamada *et al*. (1994) proposed the process for the solubilisation of animal hair. They mentioned that the animal hair had been used as a trapping agent for heavy metals, an additive for cosmetic and food, a hair tonic and an improver for clothing. They pointed out that more extensive uses of the animal hair had been expected. Miyamoto, T. *et al* (1982) also conducted a research concerning the preparation of wool powder by explosive puffing treatment. Xu *et al*. (2003) suggested a method to generate ultra fine wool powder by milling and then analysed the thermal properties of the powder.

## 2.2.2. Pulverisation of Cotton Fibres

Cotton, the natural purest form of cellulose, is the seed hair of the plants of the genus Gossypium. It is regarded as a natural, cellulosic, mono-cellular staple fibre. Cotton has been cultivated for at least 5,000 years. When cotton fibre is dry, it is almost entirely made up of cellulose (up to 96%). The normal length of cotton is between 12.7-63.5mm. The mature cotton fibre forms a flat ribbon-like structure with various width between 12 and 20 microns. It is highly convoluted and the number of convolutions ranges from 4 to 6 per millimetres, reversing in direction of about each millimetre along the fibre. The structure of cotton fibres is fibrillar. Their morphology shows the three main features namely (i) primary wall, (ii) secondary wall and (iii) lumen. The primary wall is composed of a network of cellulose fibrils covered with an outer layer of pectin, protein and wax. The wax causes the fibre to be impermeable to water and aqueous solutions unless a wetting agent is used. Although the primary wall accounts for only 5% by weight of the fibre, it contains the majority of the non-cellulosic constituents. The secondary wall constitutes the bulk of a mature fibre and is composed of cellulose fibrils arranged spirally around the fibre axis. The actual composition is dependent on the type of cotton, growing conditions and maturity (The Hong Kong Cotton Spinners Association 2000).

Many researchers, especially Japanese, have tried to develop new application areas for cotton fibres because of their excellent intrinsic properties in the development of new materials in bio-technological and bio-medical fields. For example, Banker *et al.*

(1995) suggested several methods to produce low crystallinity powder cellulose, bead cellulose and hydrated cellulose by reacting cellulose materials with phosphoric acid. They pointed out that these products were suitable for use in cosmetic, pharmaceutical, personal care and like products. Kumar *et al*. (2001) proposed the way of manufacturing low-crystallinity cellulose powder solution by treating cotton linter in phosphoric acid with the aid of agitation action. Sachetto *et al*. (1982) invented the method of obtaining micro-crystalline depolymerised cellulose from a combination of physical and chemical treatments. Turbak *et al*. (1983) proposed the methods of converting the cellulose into micro-fibrillated cellulose without substantial chemical change of the cellulose starting material. They finely divided the cellulosic fibres, mainly cotton, into powder for using as low calorie additives to foods and as thickeners in pharmaceutical products. They are even widely used as thickeners, extenders and carriers in the cosmetic and toiletry industry. Matsuda *et al*. also (2001) tried to produce the super micro-fibrillated cellulose having an arithmetic average fibre length of 0.05 to 0.1mm which was suitable for being used as a carrier for dye or pigment for the production of a tinted paper. Orlando *et al*. (1961) invented the methods of producing level off degree of polymerisation cellulose products by hydrolysis. Forand (1991) looked into the way of producing cotton fibre particles and using such particles in baked goods.

### 2.2.3. Pulverisation of Silk Fibres

Over the centuries, silk has been regarded as a highly valued textile fibre. Its qualities of strength, elasticity, softness, absorbency, affinity for dyes and adaptability to various forms of twisting continue to satisfy various market demands. In spite of facing keen competition from man-made fibres, silk has maintained its supremacy in the production of luxury apparel and specialised goods of the highest quality (Robson 1998). Silk has been used as a textile fibre for over 4000 years. Due to its high tensile strength, lustre and ability to bind dyes, silk is still considered as a premier textile material in the world today (Zarkoob 2000).

Silk is a continuous strand of protein filament cemented together and is obtained from the cocoon of silk worm Bombyx mori. The silk worm generates silk by forcing two fine streams of thick liquid out of tiny openings in its head. The streams of liquid harden into filaments during contacting with air (The Hong Kong Cotton Spinners Association 2000).

The silk fibre has a triangular cross section. The double silk filaments lie with the flat sides of the triangles together, encircled by the sericin coating. The fibre is smooth and transparent, and has rod-like shape with occasional swelling or irregularities along its length. It is fine with a diameter of 9 to 11 microns. The length of the filaments ranges from 300 to 1,000 metres. Individual filaments with the length of 3,000 metres have even been measured (Collier and Tortora 2001). These silk fibres are not only in fibre form but also in powder form which can also be obtained by many researchers. For example, Sano *et al*. (1998) introduced a method to produce the superfine silk powder with a particle size of around 10 microns, which could be used in plastics films or sheets, coating medium and fibre treatment agents. Otoi *et al*. (1980) invented a method to crush silk fibres into fine powder and obtained such particularly useful powder as an additive for cosmetic preparations. Taikyu Shoten also developed the technology to finish yarn-dyed cotton fabrics with silk powder and marketed these cotton fabrics under the ‘Powder Taste’ brand (2000).

Moreover, Tsubouchi (1998 and 2002) invented a way to generate superfine silk powder by using an alkaline aqueous solution. He proposed a method for manufacturing crystalline silk fibroin powder industrially by mechanically comminuting silk yarn. He also mentioned that this superfine crystalline silk fibroin powder could provide excellent formability, improve adhesive property to the skin or the like and enhance the extensibility and the sense of touch. It is also outstandingly suitable for making raw materials used in cosmetics, foundations, ink additives, resin composite and paints.

# Chapter 3 Characterisation of Nano-Scale Fibre Particles

## 3.1. Introduction

Hitherto, some researchers have tried to pulverise the natural fibres into particles in different ways and applied them in various fields. However, no one has ever attempted to pulverise these fibres into nano-scale particles. It is the aim of this thesis to invent an innovative method for pulverising wool, silk and cotton fibres into micro-scale and nano-scale particles. In order to prove that this newly developed method is effective in producing in such small size particles, the dimension of the particles has been evaluated and confirmed to be in micro-scale and nano-scale by means of scanning electron microscopy and particle size analysis. The chemical and crystallinity characteristics of the micro-scale and nano-scale particles have also been investigated in order to study how the particles can modify the properties and functions of the fabrics.

## 3.2. Research Methodology

In this section, the procedures used for pulverising fibres including wool, cotton and silk into micro-scale and nano-scale particles are introduced followed by the characterisation methods used for comparing the morphology, size distribution, chemical and structural properties of the pulverised micro-scale and nano-scale particles with the fibres.

### 3.2.1. Pulverisation of Fibres

The nano-scale fibre particles were prepared through three pulverisation processes. The rotate crusher, ultrasonic crusher, ultra-turrax® and nano-colliding machine were used for the three pulverisation processes. The pulverisation procedures for different fibres were similar with the exception that the pulverisation parameters such as pulverisation duration were different. In the following sections, the preparation of nano-scale wool, silk and cotton particles is introduced respectively.

#### 3.2.1.1. Preparation of Wool Particles

The nano-scale wool particles were prepared as follows:

1. Wool fibres were first washed by using deionised water. The excessive water

inside the fibres was removed by drying the wool fibres at 130 ℃ for 1 hour.

2. After cleaning and drying, the raw wool fibres were crushed into small pieces by a mechanical rotate crusher with the particle size of around 300μm in length. The particles obtained were termed as *‘First Pulverised Wool Particles’* in this thesis.

3. In the second pulverisation process, 20 grams of the coarse particles obtained from Step 2 were added into 800ml of deionised water to form a suspension and then pulverised into micro-scale wool particles by a professional ultrasonic crusher, e.g. Ultrasonic Crusher JY-99IID. During the second pulverisation process, the Ultrasonic Crusher JY99-II D was set to run for two seconds of ultrasonic operation under a power of 1800W with a one second gap in between, and the suspension was pulverised by this means for 40 minutes. After the ultra-sonic operation, the micro-scale wool particles so obtained were termed as *‘Second Pulverised Wool Particles’* in this thesis.

4. The suspension was then filtered by a woven wire microplate sieve with an aperture size of 20 microns. The filtrate suspension obtained was dispersed and further crushed into smaller scale by means of a high performance dispersing instrument such as IKA T25 Basic Ultra-turrax® for 10 minutes. After crushing

and filtering, the filtrate suspension obtained was pulverised into nano-scale wool suspension using a nano-colliding machine (Nano collide machine Model: NT1500/5, Beijing Nanomizer Technology Research Institute) under the pressure of 1000kgf/cm$^2$ with a speed of 300rpm. After this state, the nano-wool particles so called ‘*Third Pulverised Wool Particles*’ were obtained.

#### 3.2.1.2. Preparation of Silk and Cotton Particles

The pulverisation of silk and cotton fibres followed the similar ways as those of the wool fibres, while the ultrasonic pulverisation and ultra-turrax® operation durations of silk and cotton fibres were different from those of wool fibres. The ultrasonic pulverisation and ultra-turrax® operation parameters of wool, silk and cotton are illustrated in the Table 3.1.

**Table 3.1.** Pulverisation parameters of wool, cotton and silk

| Fibres | Ultrasonic pulverisation duration (mins) | Ultra-turrax® operation duration (mins) |
|---|---|---|
| Wool | 40 | 10 |
| Cotton | 20 | 10 |
| Silk | 50 | 15 |

## 3.2.2. Characterisation of Nano-Scale Fibre Particles

After successfully generating the micro-scale and nano-scale fibre particles, the morphology, size distribution, chemical and structural characterisation of the particles were compared with those of control fibre by means of scanning electron microscopy, particle size analysis, Fourier transmittance infrared analysis and X-ray diffraction analysis respectively.

### 3.2.2.1. Scanning Electron Microscopy (SEM)

The morphology of the pulverised particles was examined by the scanning electron microscopy using a JSM-6335F Field Emission Scanning Electron Microscope.

### 3.2.2.2. Particle Size Analysis

The particle size distribution of the pulverised particles was analysed by the Beckman Coulter LS 13320 Particle Size Analyser which was designed to be fully compliant with the ISO/DIN13320-1 method.

### 3.2.2.3. Fourier Transmittance Infrared Analysis (FTIR)

The FTIR spectra of the original fibre, micro-scale and nano-scale pulverised particles were analysed in order to study the chemical changes of fibres after pulverisation. In this research, KBr disc technique was used for the FTIR analysis. The technique consisted of mixing the finely divided 0.004g pulverised particles with 0.4g powdered potassium bromide. The disc was formed in a die under very high pressure and then analysed by means of the Perkin Elmer Fourier Transform Infrared Spectroscopy System 2000.

#### 3.2.2.4. X-Ray Diffraction Analysis

The crystallinity of the pulverised particles was determined by means of the XRD technique using the Philips Analytical PW3710 based X-Ray diffractometers with a Cu tube anode source at 40kV x 35mA. A 0.1mm pinhole collimator was employed. Scattering XRD data were recorded using the XRD Evaluation Programme (Bruker Advanced X-Ray Solutions Diffract Plus Release 2000 Eva 6.0 version).

## 3.3 Result and Discussion

The SEM images of the pulverised fibres particles of wool, cotton and silk as well as the results of the particle size analysis of the particles confirmed that the pulverisation method invented in this project was useful in the preparation of nano-scale fibre particles. The chemical and structural changes of the fibres after pulverisation were illustrated in the following sections.

## 3.3.1. SEM Image of the Pulverised Fibres Particles

### 3.3.1.1. SEM Image of Pulverised Wool Fibre Particles

In this project, wool fibres were successfully pulverised into particles with the aid of a rotary blade, ultra-sonic crusher, ultra-turrax® machine and nano-colliding machine. The ultra-sonic and ultra-turrax® operation durations used for pulverising wool fibres were 40 and 10 minutes respectively.

Figure 3.1 shows that the length of the original wool fibres was in millimetre scale with the diameter of around 8–20μm. The surface of wool fibres consisted of a fine network of small overlapping scales. Figure 3.2 shows that after the first pulverisation process, the fibres were smashed into the combination of large rod-like particles of around 300μm together with a large amount of less than 10μm superfine particles. The scales of the wool fibres were also removed during the first pulverisation process. Figure 3.3 shows that the second pulverised wool particles of 0.1–7μm were obtained after the second pulverisation process. Most of the second pulverised wool particles were in sheet form. Figure 3.4 shows that most of the third pulverised wool particles were in nano-scale spherical shape, while some were in rod-like shape.

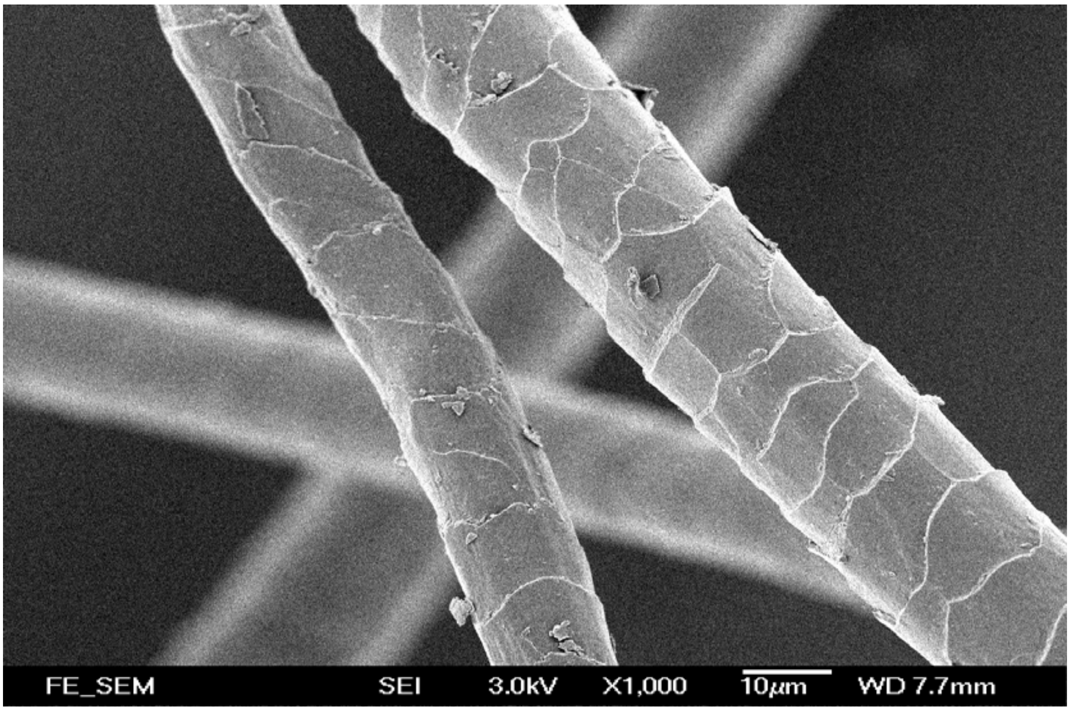

**Figure 3.1** SEM image of wool fibres with the fibre diameter of around 8–20μm of 1,000x

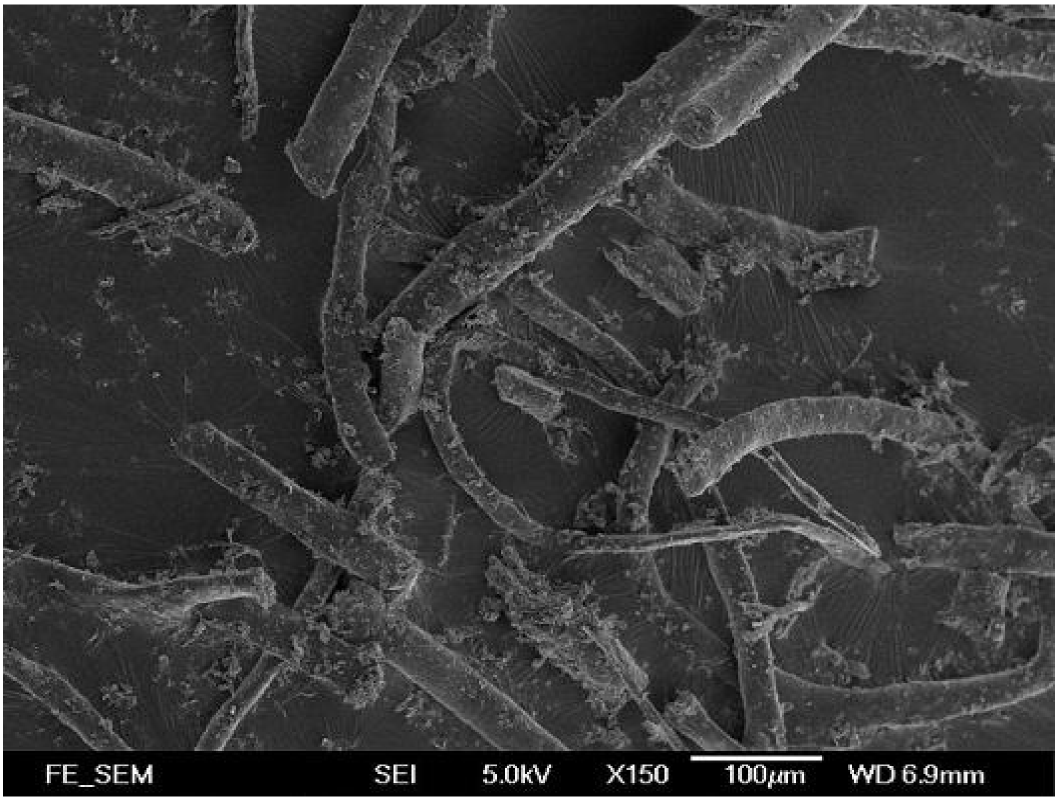

**Figure 3.2** SEM image of the first pulverised wool particles of 150x

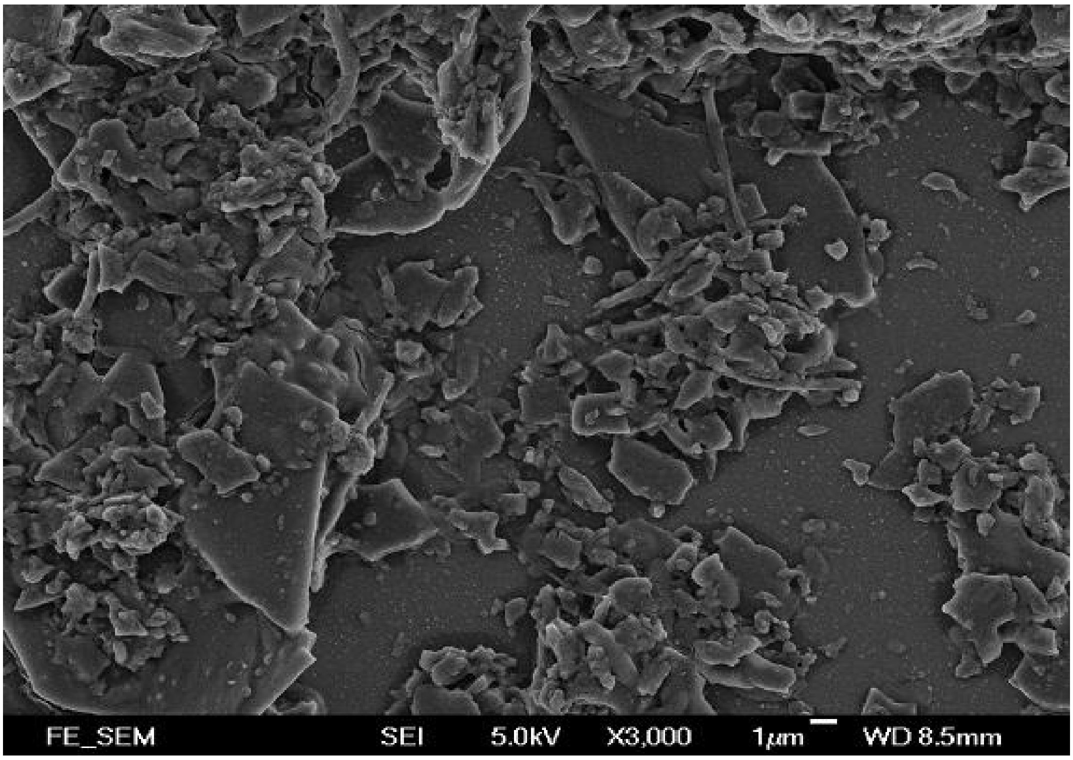

**Figure 3.3** SEM image of the second pulverised wool particles of 3,000x

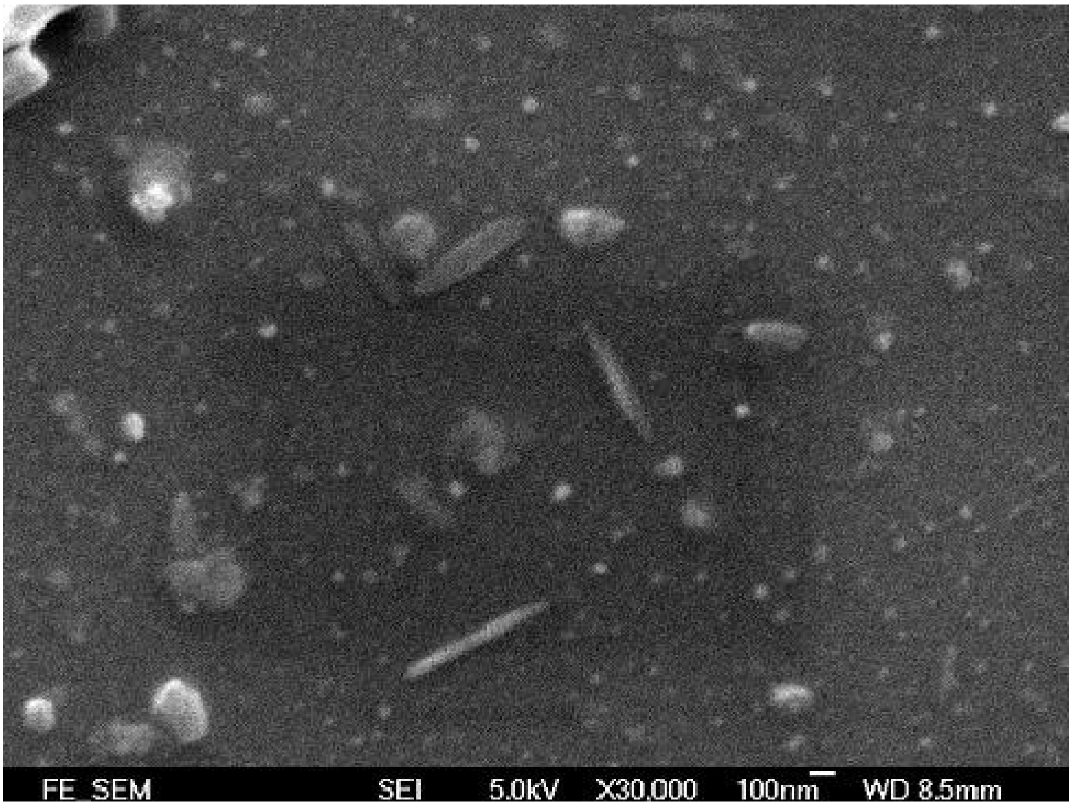

**Figure 3.4** SEM image of the third pulverised wool particles of 30,000x

### 3.3.1.2. SEM Image of Pulverised Silk Fibre Particles

Silk fibres were also successfully pulverised into particles with the aid of a rotary blade, ultra-sonic crusher, ultra-turrax® machine and nano-colliding machine. The duration of ultra-sonic and ultra-turrax® operation used for pulverising silk fibres was 50 and 15 minutes respectively, which were much longer than those of wool fibres and cotton fibres due to the higher strength of the silk fibres.

Figure 3.5 shows that the length of the original silk fibres was in millimetre scale with the diameter of around 9 to 11μm. Figure 3.6 shows that after the first pulverisation process, the fibres were smashed into the combination of large rod-like particles of around 100 to 300μm together with a small amount of less than 10μm superfine particles. The surface of the silk fibres became rough after the first pulverisation process. Figure 3.7 shows that the second pulverised silk particles of around 0.1 to 10μm were obtained after the second pulverisation process. Most of the second pulverised silk particles were either in ribbon or spherical shape. Figure 3.8 shows that all the third pulverised silk particles were in nano-scale spherical shape.

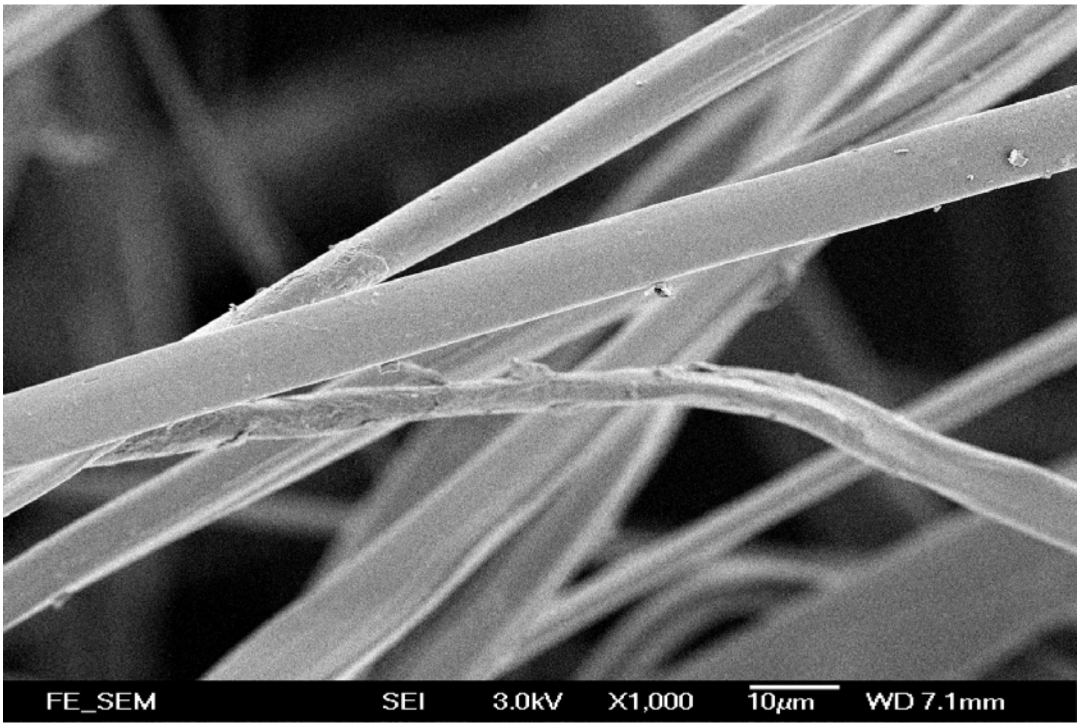

**Figure 3.5** SEM image of silk fibres of 1,000x with the diameter of around 9-11μm

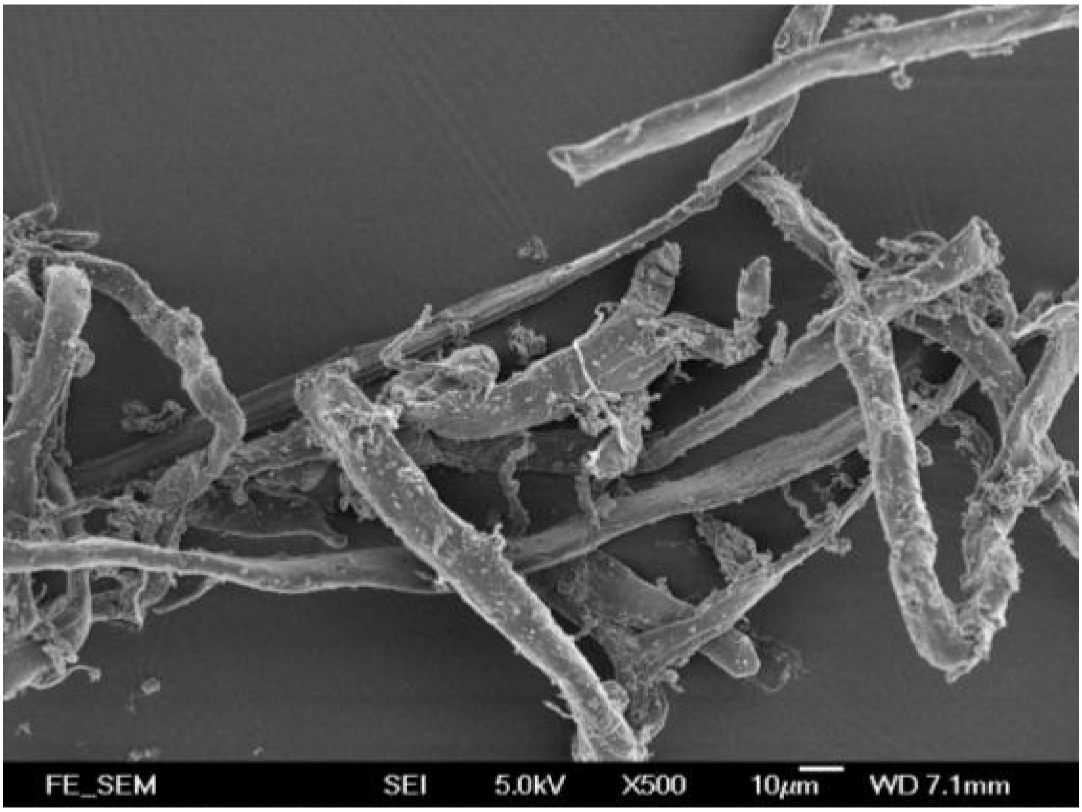

**Figure 3.6** SEM image of the first pulverised silk particles of 500x

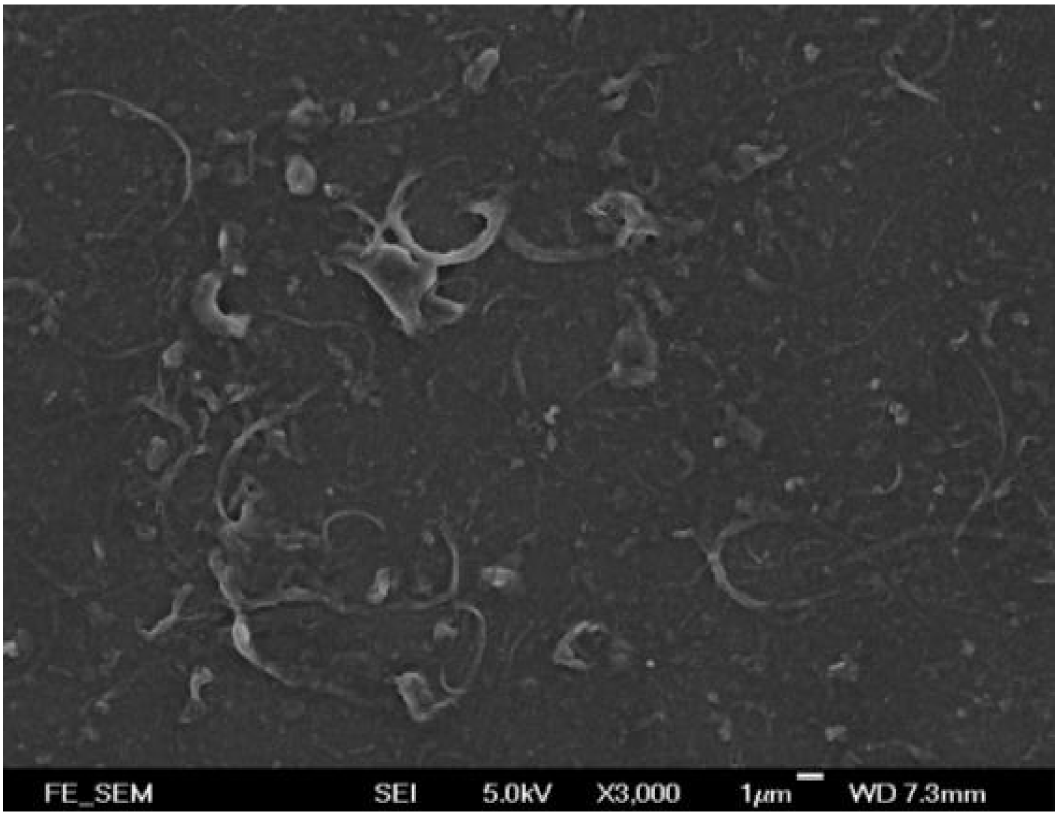

**Figure 3.7** SEM image of the second pulverised silk particles of 3,000x

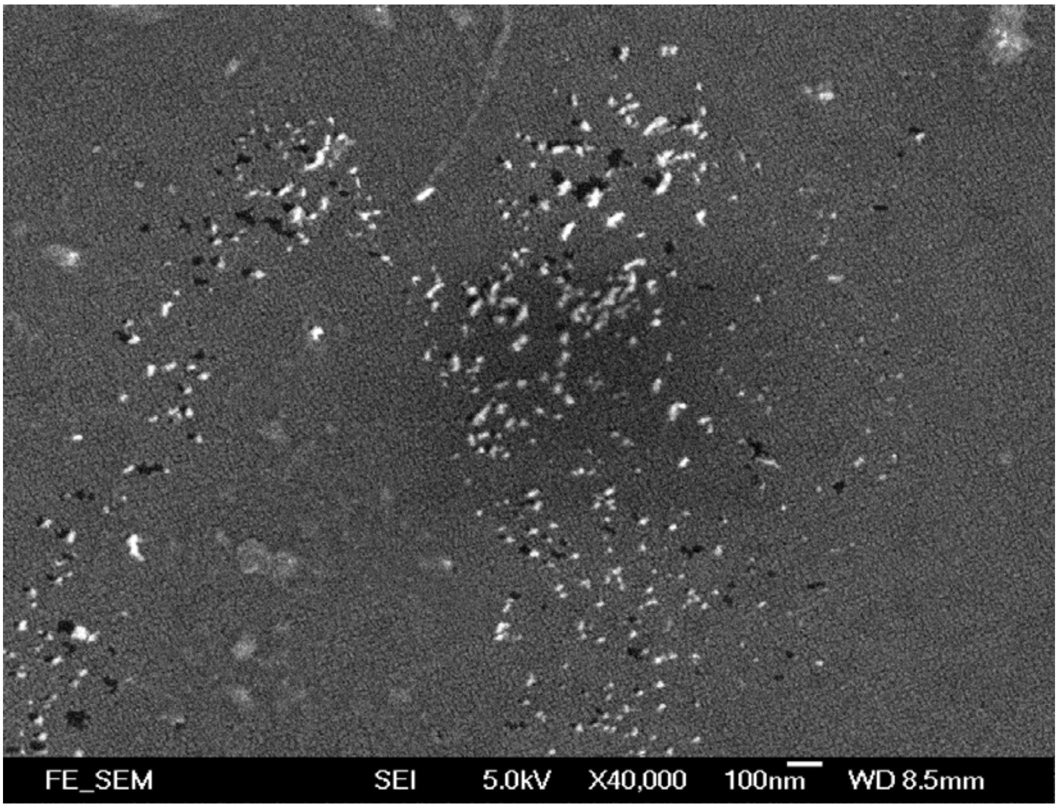

**Figure 3.8** SEM image of the third pulverised silk particles of 40,000x

### 3.3.1.3. SEM Image of Pulverised Cotton Fibre Particles

In this project, cotton fibres were also been pulverised into particles with the aid of a rotary blade, ultra-sonic crusher, ultra-turrax® machine and nano-colliding machine. The duration of ultra-sonic and ultra-turrax® operation used for pulverising cotton fibres was 20 and 10 minutes respectively with the shortest duration among the three types of fibres when compared.

Figure 3.9 shows that the length of the original cotton fibres was in millimetre scale. The cotton fibres were highly convoluted with their diameters ranging from around 10 to 20µm. Figure 3.10 shows that after the first pulverisation process, the cotton fibres were pulverised into particles of around 2 to 3µm. The first pulverised cotton particles was no longer in fibre form, and the convolution of the fibre was not observed after the first pulverisation process. Figure 3.11 shows that after the second pulverisation process, the majority of second pulverised cotton particles were less than 1µm in size. Most of the second pulverised cotton particles were in non-spherical shape. Figure 3.12 shows that most of the third pulverised cotton particles were in nano-scale spherical shape with the size of less than 100nm.

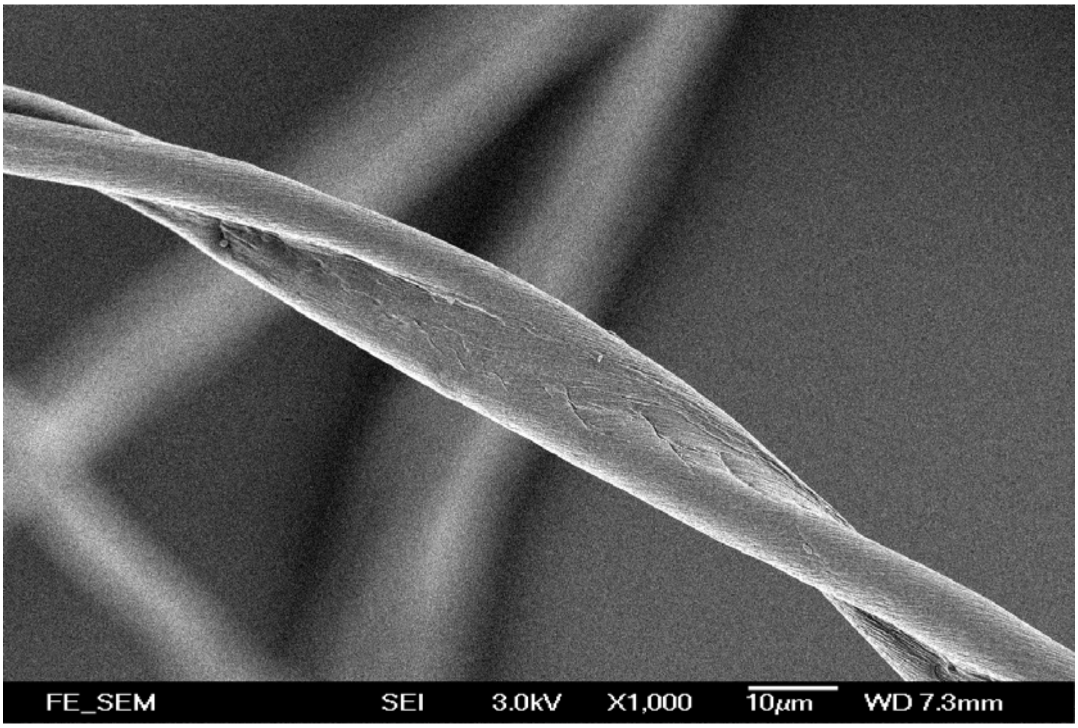

**Figure 3.9** SEM image of longitudinal section of original cotton fibre of 1,000x with the diameter ranging from 10 to 20μm

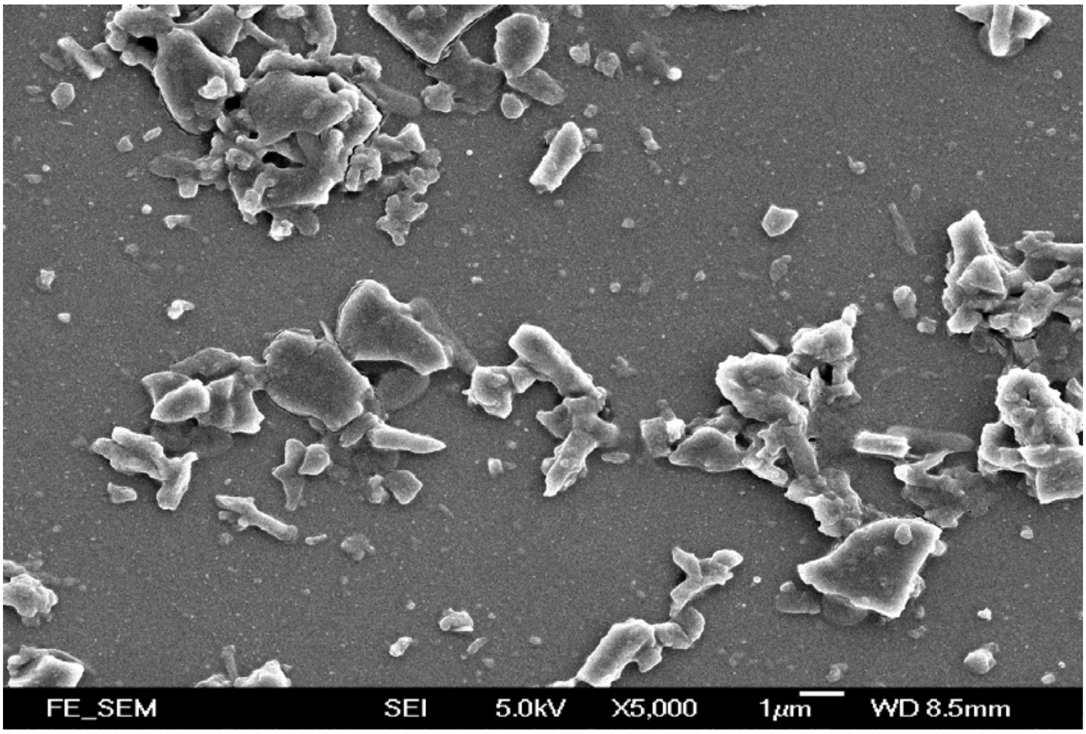

**Figure 3.10** SEM image of the first pulverised cotton particles of 5,000x

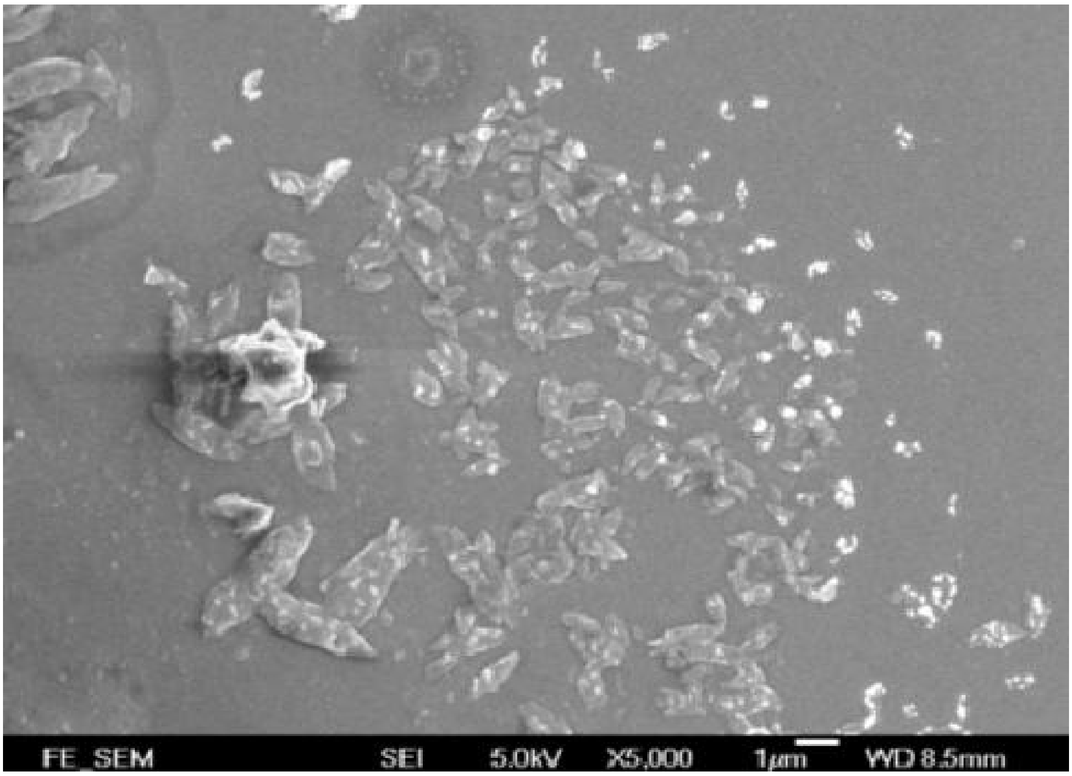

**Figure 3.11** SEM image of the second pulverised cotton particles of 5,000x

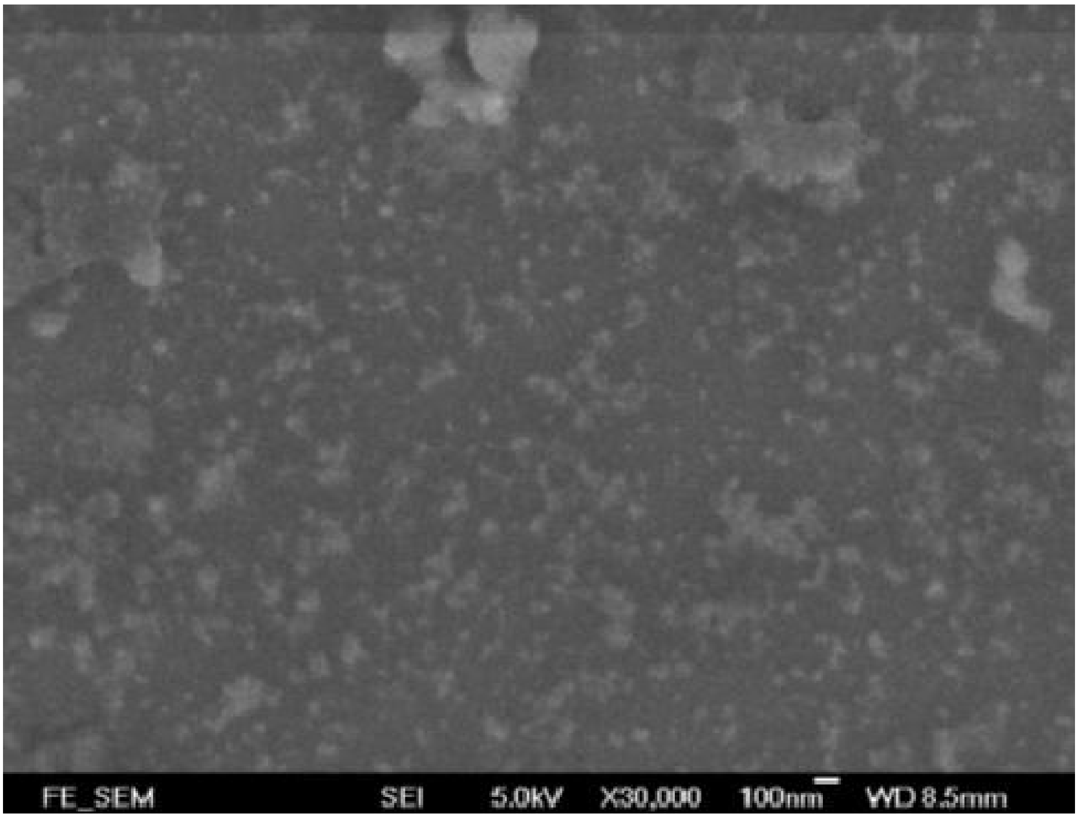

**Figure 3.12** SEM image of the third pulverised cotton particles of 30,000x

## 3.3.2 Particle Size Analysis with Laser Particle Size Analyser

The SEM images shown in Figure 3.1 to 3.12 demonstrated that after the second and third pulverisation processes, the micro-scale and nano-scale of wool, silk and cotton particles were generated respectively. Although, these SEM pictures could only exhibit the images of wool, silk and cotton particles located in a specific area, yet they did not provide a quantitative analysis of the actual particle size distribution of the particles. As a result, it was necessary to analyse the particle size distribution of the first, second and third pulverised wool, silk and cotton particles using the Beckman Coulter LS 13320 particle size analyser.

### 3.3.2.1. Principle of Particle Size Analysis

The laser diffraction technique of the LS 13320 particle size analyser is based on the fact that particles scatter light in all directions with an intensity pattern depending on the particle size. All these instruments assume that the particles are spherical. For non-spherical particles, an equivalent-sphere size distribution is obtained because the technology makes the assumption of spherical particles in its optical model. Hence, the resulting size distribution may differ from those obtained by other physical methods, such as sedimentation and sieving. This deconvolution was found according to the Fraunhofer theory of light scattering (International Organisation for

Standardisation. Particle Size Analysis 1999). Xu and Di Guida (2003) pointed out that the diameters of nonspherical particles were ill defined and the theoretical models of scattering patterns were seldom available. The results obtained from the application of any spherical model would certainly have unexpected bias or error.

In practice, most people neglect the difference between the real shape of particles and the model. They customarily take the reported mean values and size distribution for granted without realising that these are only apparent sizes which are actually not the equivalent sizes. In certain extreme cases, the results obtained by the application of the spherical model to non-spherical particles will be very different from the truth.

### 3.3.2.2. Particle Size Analysis Result

The particle size distribution of the first, second and third pulverised wool, silk and cotton are shown in Figures 3.13, 3.14 and 3.15 respectively. The particle size ranges of the pulverised wool, silk and cotton particles are recorded in Table 3.2.

**Table 3.2.** Particle size distribution of pulverised wool, silk and cotton particles

| | Particle size of wool particles (μm) | Particle size of silk particles (μm) | Particle size of cotton particles (μm) |
|---|---|---|---|
| 1st pulverised particles | 1 to 15 | 1 to 20 | 1.5 to 50 |
| 2nd pulverised particles | 0.15 to 7 | 0.1 to 5 | 0.1 to 2.5 |
| 3rd pulverised particles | < 0.1 | < 0.1 | < 0.1 |

Table 3.2 demonstrates that the size of the third pulverised wool, silk and cotton particles was less than 0.1μm. The particles with a diameter of less than 0.1μm were classified as nano-scale particles. This confirmed that the nano-scale wool, silk and cotton particles were successfully obtained after the pulverisation processes.

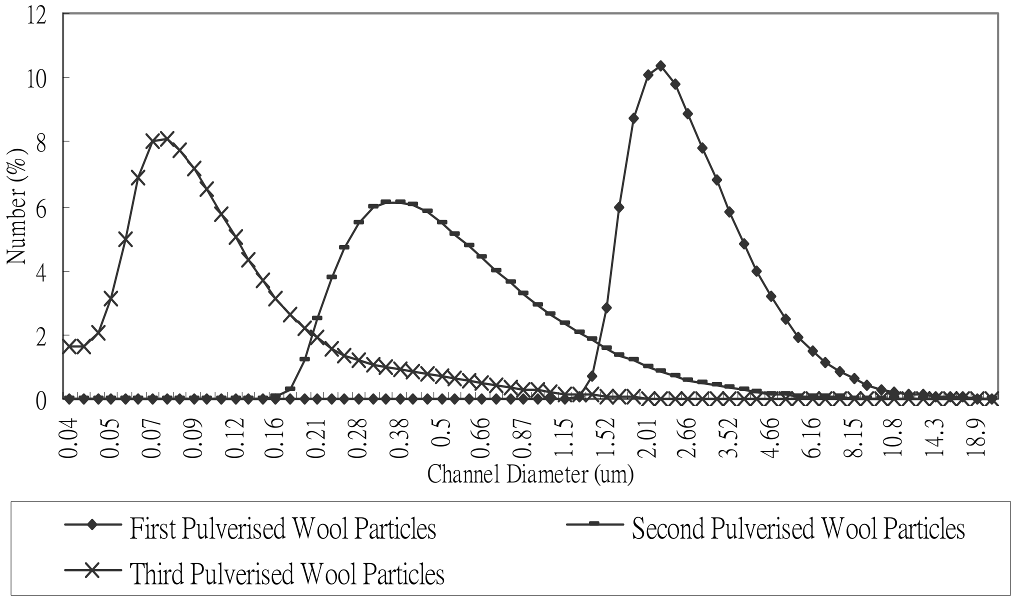

**Figure 3.13** Particle size distribution of the first, second and third pulverised wool particles

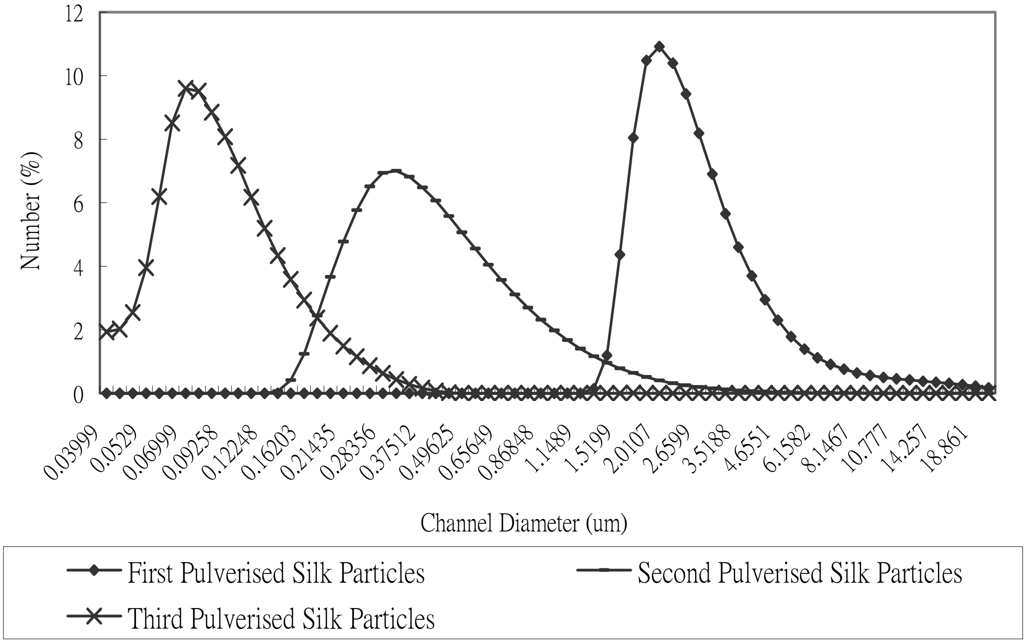

**Figure 3.14** Particle size distribution of the first, second and third pulverised silk particles

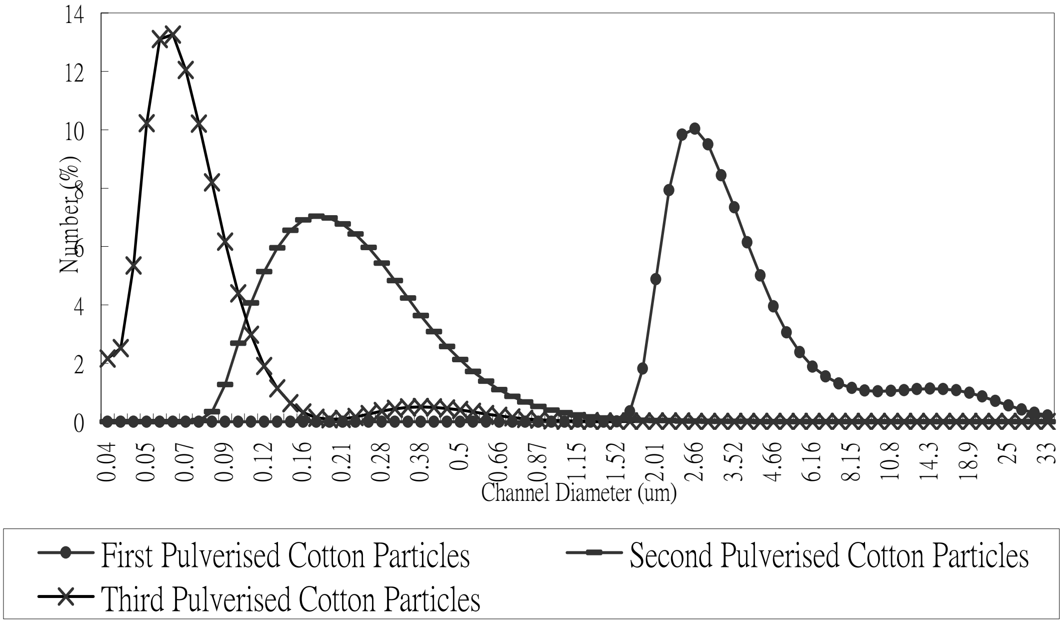

**Figure 3.15** Particle size distribution of the first, second and third pulverised cotton particles

### 3.3.2.3. Contradiction Between SEM Images and Particle Size Analysis

Apart from the particle size analysis, the particle size distribution of the pulverised wool, silk and cotton particles was also confirmed by the observation of SEM images aiming to determine the morphology of the particles.

The SEM images of the first pulverised wool, silk and cotton particles are shown in Figures 3.2, 3.6 and 3.10 respectively. These figures showed that the particle sizes of the first pulverised wool, silk and cotton particles were around 300µm, 100 to 300µm, and 200 to 500µm respectively. However, the particle size distribution shown in Table 3.2, could hardly identify such particles. The difference might be attributed to a number of potential causes namely (i) the particles present in a specific area of the SEM images were randomly selected, (ii) the particles were assumed to be spherical in the particle size analyser, and (iii) the particle size analysis was based on the counting of the number percentage of the particles. The first pulverised wool, silk and cotton particles might contain only a limited number of large particles. Such an amount was considered negligible for counting the number percentage as most of the first pulverised particles were in very small size.

The SEM images of the third pulverised wool, silk and cotton particles showed that most of the particles were almost spherical and their diameters were less than 100nm.

This was consistent with the measurement results obtained from the laser particle size analyser.

### 3.3.3 Theory Behind the Pulverisation Method

The aforementioned SEM images and particle size analysis did provide evidence about the successful development of pulverisation method for preparing nano-scale fibre particles. The pulverisation method involved the use of mechanical rotate crusher, ultrasonic crusher, microplate sieve, high performance dispersing instrument and nano-colliding machine.

Mechanical rotate crusher was simply used for cutting the fibres into particles form by means of the rotary blades. The particles obtained were then pulverised by using ultrasonic crusher.

Ultrasonic crusher was important in the preparation of nano-scale fibre particles as it could provide acoustic energy for pulverisation. According to the article of Simona Vajnhandl and Alenka Majcen Le Marechal (2005), acoustic energy is a mechanical energy which is not absorbed by molecules. When ultrasound transmits through a liquid, it induces vibration of the intermolecular bonds and energy is thus transferred through the medium in the propagation direction. Ultrasound waves comprise expansion and compression cycles. Compression cycles introduce a positive pressure on the liquid and push molecules together, while expansion cycles initiate a negative pressure and pull molecules apart. Cavities can be generated during the expansion of a sound wave cycle and therefore ‘voids’ are created in the liquid and cause the formation of cavitations. Kaully *et al.* 2003 indicated that the generation of a high frequency, ultrasonic vibration field in liquids caused the formation of cavitations and the production of high local pressure. The high pressure in the cavities near the particles suspended in the partial solvent normally induces grinding effects, causing sharp reduction in the particle size. Ultrasonic vibrations in the appropriate liquid produce a sharpening effect, imparting to the particles a rounded, almost spherical shape. The shape is associated with a grinding effect, viz. the reduction in the particle size. This shows that ultrasonic vibration is useful in the pulverisation of fibre into small size particles.

After pulverising the fibre particles into micro-scale with the aid of ultrasonic crusher, the microplate sieve was used for filtrating the large size particles. A high performance dispensing instrument, Ultra-turrax ®, was then applied. It was used not only for dispersing the suspension, but also for pulverisation purpose. Hsu *et a.l* (2004) patent mentioned that the high performance dispensing instrument, ultra-turrax ®, was required to separate the coagulated particles into dispersed nano-particles. Moreover, the stirring action created by the instrument provided kinetic energy for grinding media to collide with each other, thereby driving the suspension to generate shearing force and colliding force between fluid and solid or between solid and solid. The collision of the particles was useful for further pulverising process.

After the usage of ultra-turrax®, the final instrument used for the pulverisation process was the nano-colliding machine. The action of the nano-colliding machine was based on the method called “self-abrasion”. In the machine, the fluid jet captured the particles and brought them upwards to the separation zone at the velocity near the transonic speed. The thin fraction, i.e. the smallest particles, was separated off by the centrifugal separator. Heavy and large particles, however, came back to the pounding zone. The streams met each other with different velocities, i.e. they flew up at a high speed and fell down at rather slower speed, along with the non-ground material layer, which was constantly poured into the device. The particles broke to pieces by blowing each other and the friction of particles (Simona Vajnhandl and Alenka Majcen Le Marechal 2005).

## 3.3.4. Fourier Transmission Infrared (FTIR) Spectra Analysis

In order to study whether there were any chemical changes in the fibres after pulverisation, the FTIR spectra of the first, second and third pulverised wool, silk and cotton particles were studied. The FTIR spectra analysis could check the functional groups of the wool, silk and cotton fibres.

### 3.3.4.1. FTIR Spectra Analysis of Wool Particles

The FTIR spectra of the pulverised wool particles shown in Figure 3.16 confirmed that there was no great change in the chemical structure of wool after the pulverisation processes. However, both the FTIR spectra of the wool fibre and the first pulverised wool particles were only slightly different from those of the second and third pulverised wool particles within the ranges of 1000-1800cm$^{-1}$ and 3230-3670cm$^{-1}$.

Within the 1000-1800 cm$^{-1}$ range, the FTIR spectra illustrated that the second and third pulverised wool particles had stronger absorbance band within 1490-1580cm$^{-1}$ and a stronger peak at 1655cm$^{-1}$ than those of the wool fibre and the first pulverised wool particles. This might be due to the presence of a greater number of the secondary amine group and amide group in the second and third pulverised wool particles.

The second and third pulverised wool particles also had higher absorption in the region of 1030-1230cm$^{-1}$. This might be due to the fact that the second and third pulverised wool particles have greater amount of C=S stretching vibration than the wool fibre and the first pulverised wool particles. The second and third pulverised wool particles also have stronger absorbance band within the region of 3230-3670cm$^{-1}$ than the wool fibre and the first pulverised wool particles. This indicated that the second and third pulverised wool had greater amount of O-H bond than the wool fibre and the first pulverised wool particles (Socrates, G., 2001).

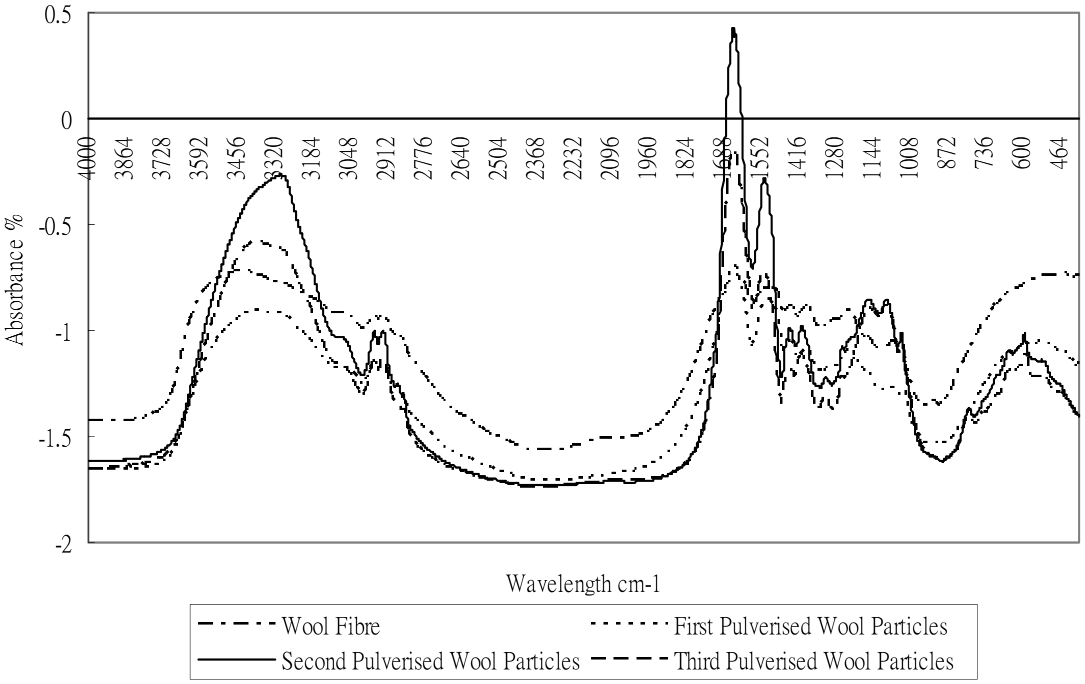

**Figure 3.16** FTIR absorption spectra of the first, second and third pulverised wool particles

### 3.3.4.2. FTIR Spectra Analysis of Silk Particles

The FTIR spectra of the pulverised silk particles shown in Figure 3.17 confirmed that there was no great change in the chemical structure of silk after the pulverisation processes. The FTIR spectra of the silk fibre and the pulverised silk particles were only slightly different from each other within the range of 1020-1240cm$^{-1}$, 1200-1305 cm$^{-1}$ and 1350-1475cm$^{-1}$. The FTIR spectra illustrated that the second and third pulverised silk particles had stronger absorbance band within 1020-1240cm$^{-1}$ than that of the silk fibre and the first pulverised silk particles. This might be due to the presence of a greater amount of the primary aliphatic amines in the second and third pulverised silk particles. The silk fibre as well as the first and second pulverised silk particles had medium absorption in the region of 1200-1305cm$^{-1}$, while the third pulverised silk particles did not have it. This might be due to the presence of the secondary amides in the silk fibre as well as the first and second pulverised silk particles, but they were absent in the third pulverised silk particles. The third pulverised silk particles had stronger absorbance band within the region of 1350-1475cm$^{-1}$ than that of the silk fibre as well as the first and second pulverised silk particles. This might be due to the presence of a greater amount of $C(CH_3)_3$ group in the third pulverised silk than that of the silk fibre, as well as the first and second pulverised silk particles (Socrates, G., 2001).

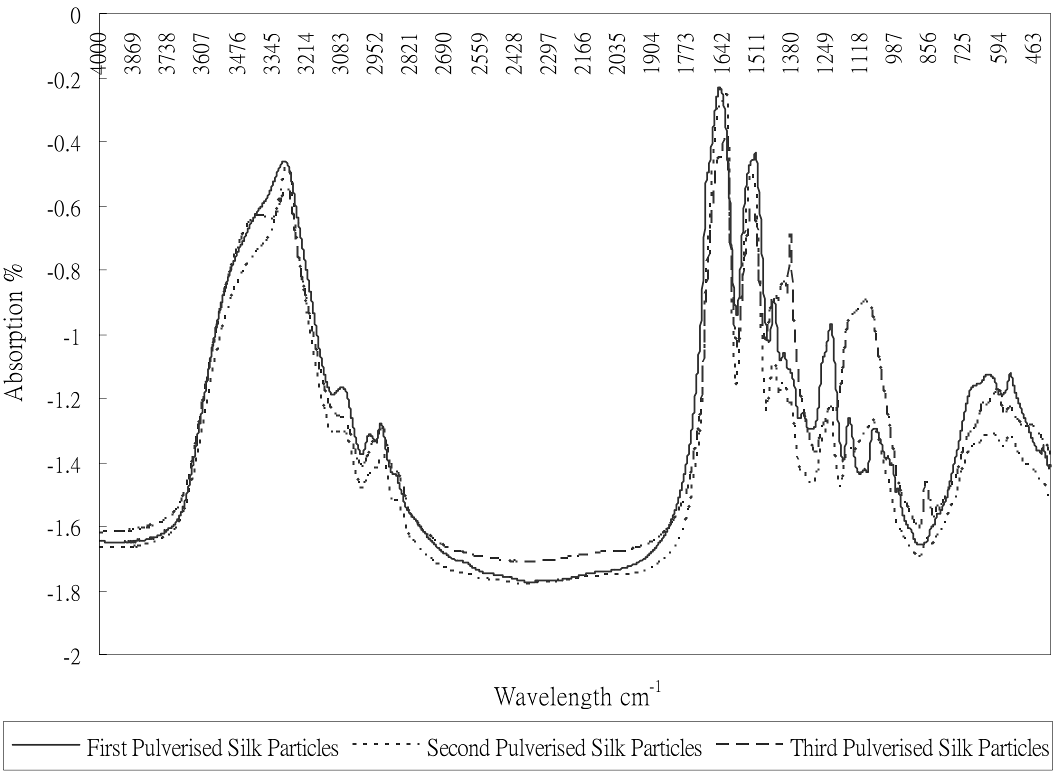

**Figure 3.17** FTIR absorption spectra of the first, second and third pulverised silk particles

### 3.3.4.3. FTIR Spectra Analysis of Cotton Particles

The FTIR spectra of the pulverised cotton particles shown in Figure 3.18 confirmed that there was no significant change in the chemical structure of cotton after pulverisation processes. However, the FTIR spectrum of the third pulverised cotton particles were different from that of the first and second pulverised cotton particles in the region of 1380cm$^{-1}$. The difference in this region was due to greater amount of symmetric methyl groups (C-H) present in the third pulverised cotton particles (Socrates, G., 2001).

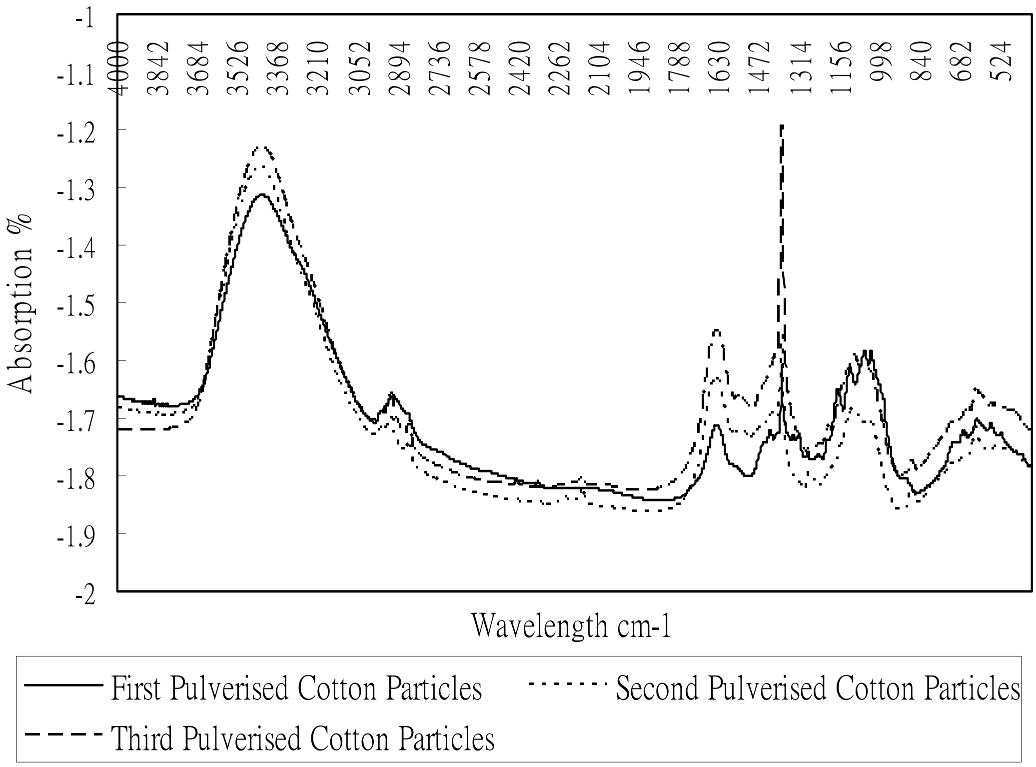

**Figure 3.18** FTIR absorption spectra of the first, second and third pulverised cotton particles

### 3.3.5 X-Ray Diffraction (XRD) Analysis

In the present study, the XRD analysis was used for assessing the physical change in the wool. The XRD analysis determined the crystallinity of fibres and was used to indicate a dramatic change in the crystallinity of the wool, silk and cotton fibres. The XRD evaluation programme (Advanced X-Ray Solutions Diffract Plus Release 2000 Eva, version 6.0, Bruker) allowed convenient data transfer and analysis. The method used for determining the crystallinity percentage was based on the estimation ratio of the crystalline material to the amorphous material in the sample. It was obtained from the modified XRD intensity profiles by the drawing of a smooth curve through the minimal of the principal diffraction effects and the comparison of the areas below and above the curve over an angular range of 2θ = 10–40°. The smoothing curve was generated with the function, “Background”, of the programme Eva.

The XRD patterns of the samples were divided into crystalline and amorphous regions with a smooth curve as shown in Figure 3.19. The area above the curve corresponded to the scattering caused by the crystalline region of the sample, whereas the area below the curve corresponded to the amorphous region.

#### 3.3.5.1 X-Ray Diffraction (XRD) Analysis of Wool, Silk and Cotton Particles

Figures 3.19 to 3.27 show the XRD intensity of the first, second and third pulverised wool, silk and cotton particles with 2θ ranging from 10 to 40° respectively. The area above the smooth curve represented the scattering caused by the crystalline region of the samples. There was a decrease in the area above the smooth curve with respect to the smaller particle size of the particles, meaning that the crystallinity of the particles also decreased as a result of smaller particle size.

Figure 3.22 is a graph presenting the crystallinity and amorphous percentages of the wool, silk and cotton particles. They were determined by a comparison of the areas above and below the smooth curve with the aid of the XRD evaluation programme. The results showed that the crystallinity percentage of the first pulverised wool particles decreased from 47.942% to 12.087% and 5.379% for the second and third pulverised wool particles respectively. As for the first pulverised silk particles, its crystallinity percentage reduced from 58.281% to 27.085% and 21.821% for the second and third pulverised silk particles respectively. The crystallinity percentage of the first pulverised cotton particles also decreased from 63.822% to 25.344% and 8.927% for the second and third pulverised cotton particles respectively.

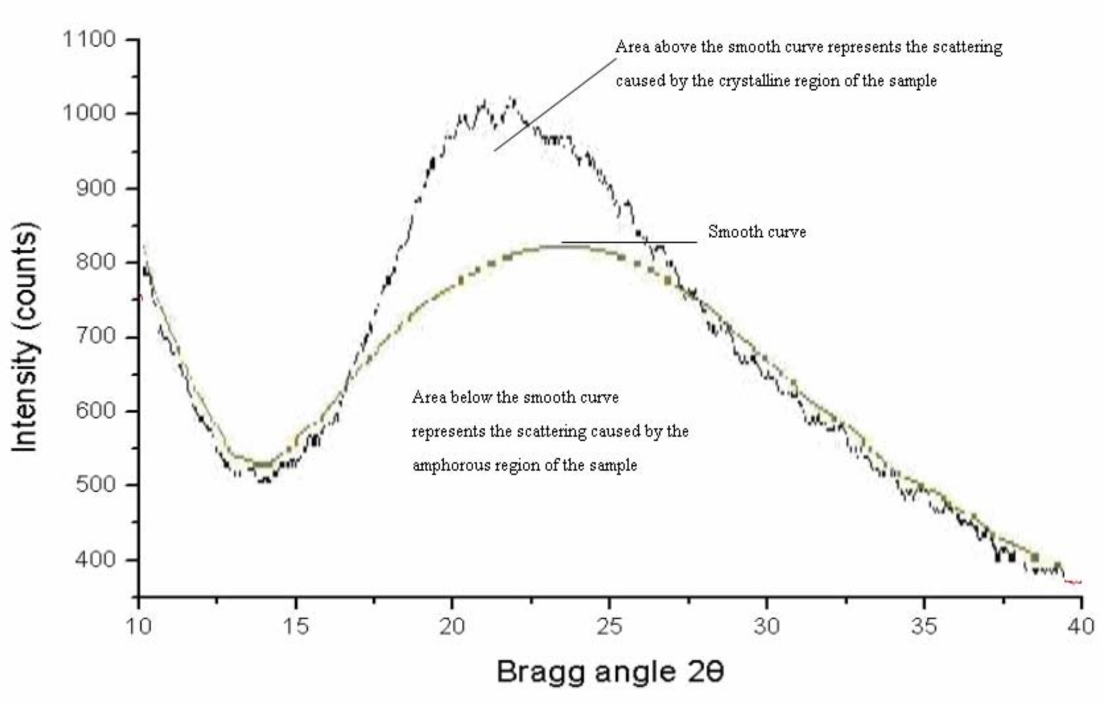

**Figure 3.19** XRD pattern of the first pulverised wool particles with 2θ ranging from 10 to 40˚

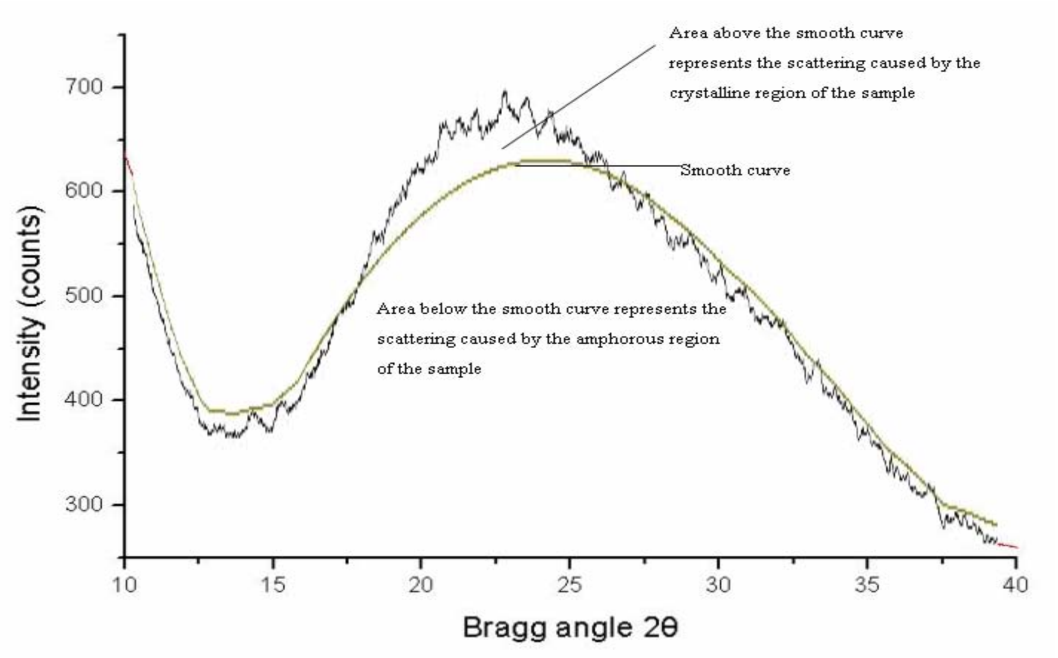

**Figure 3.20** XRD pattern of the second pulverised wool particles with 2θ ranging from 10 to 40˚

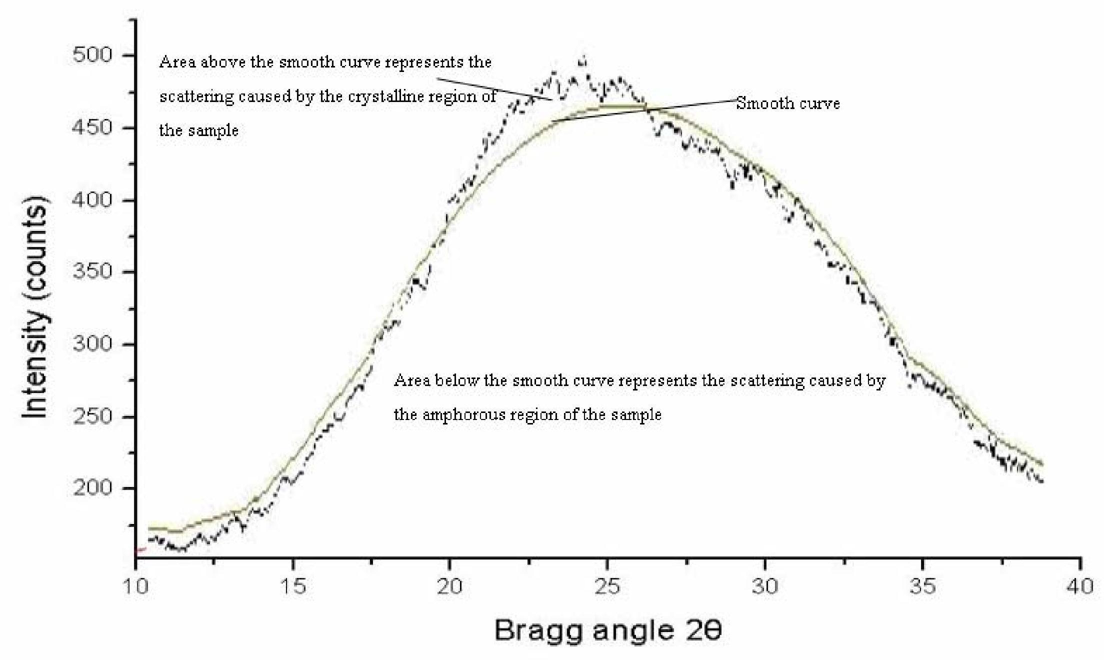

**Figure 3.21** XRD pattern of the third pulverised wool particles with 2θ ranging from 10 to 40˚

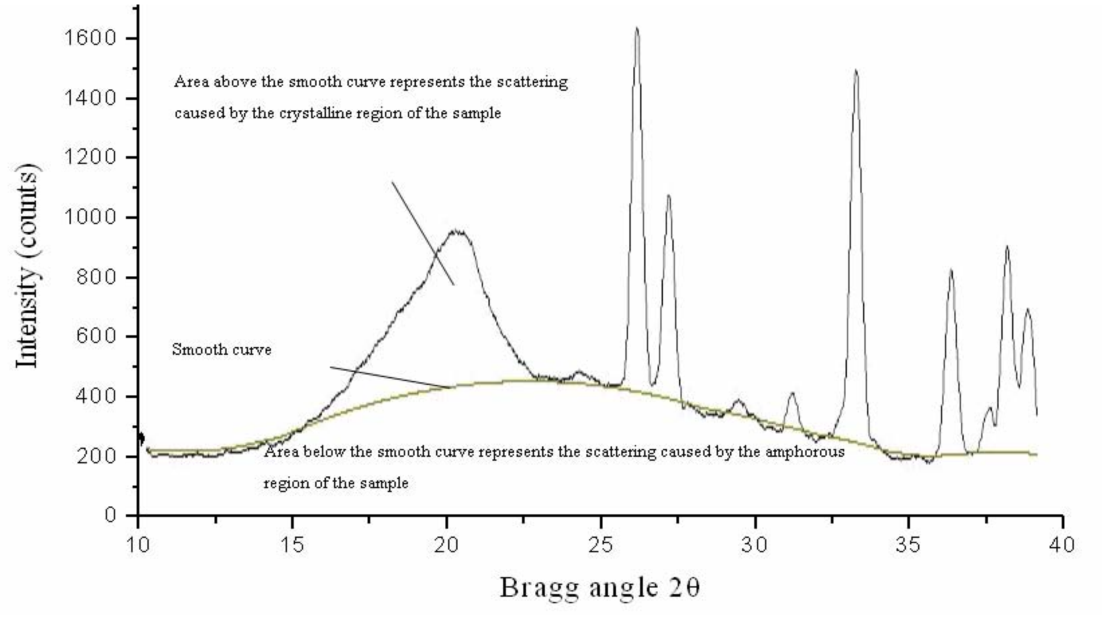

**Figure 3.22** XRD pattern of the first pulverised silk particles with 2θ ranging from 10˚ to 40˚

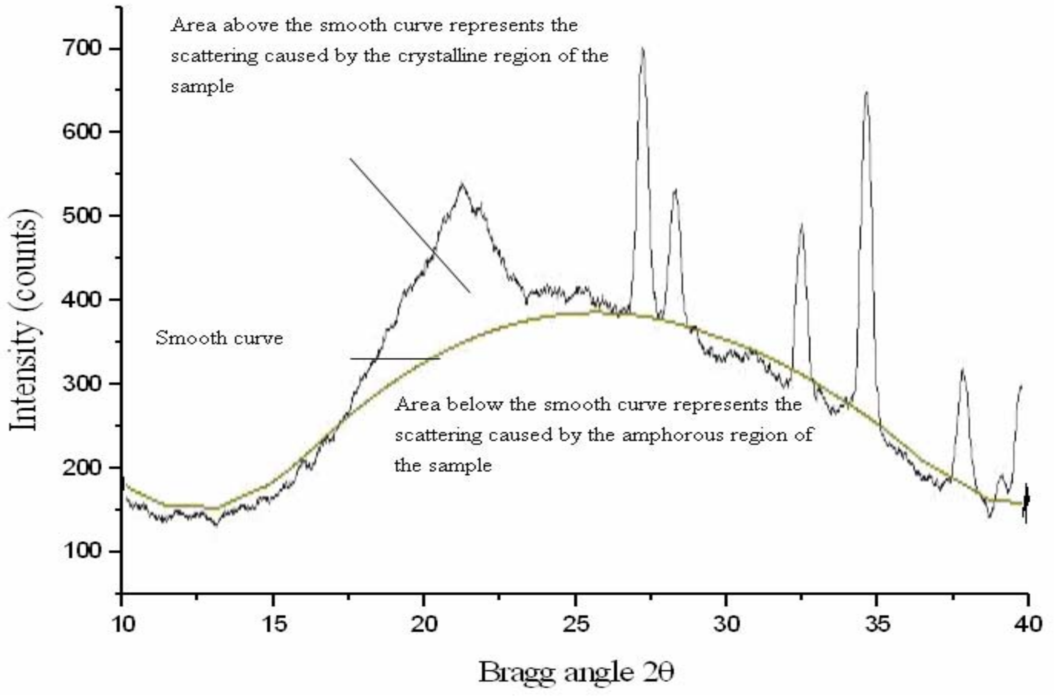

**Figure 3.23** XRD pattern of the second pulverised silk particles with 2θ ranging from 10˚ to 40˚

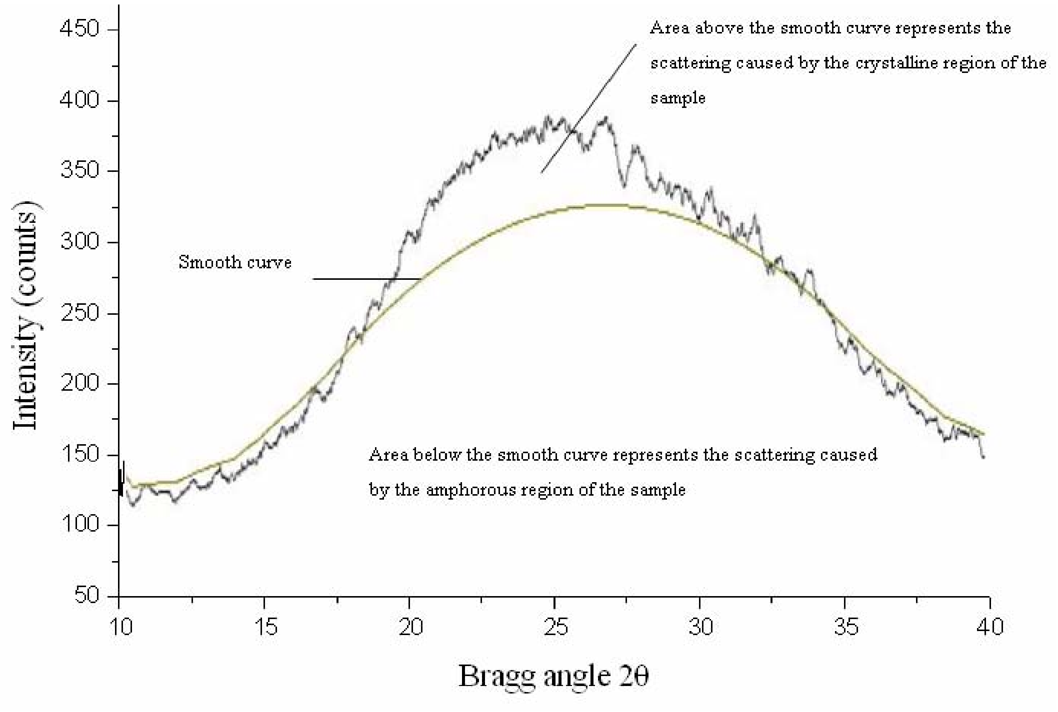

**Figure 3.24** XRD pattern of the third pulverised silk particles with 2θ ranging from 10˚ to 40˚

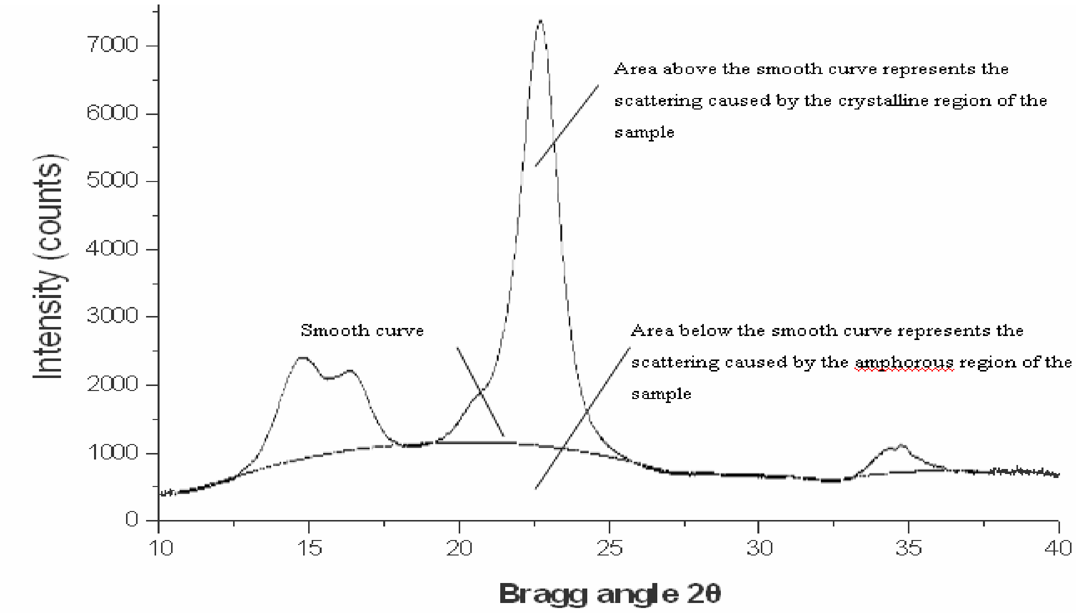

**Figure 3.25** XRD pattern of the first pulverised cotton particles with 2θ ranging from 10 to 40˚

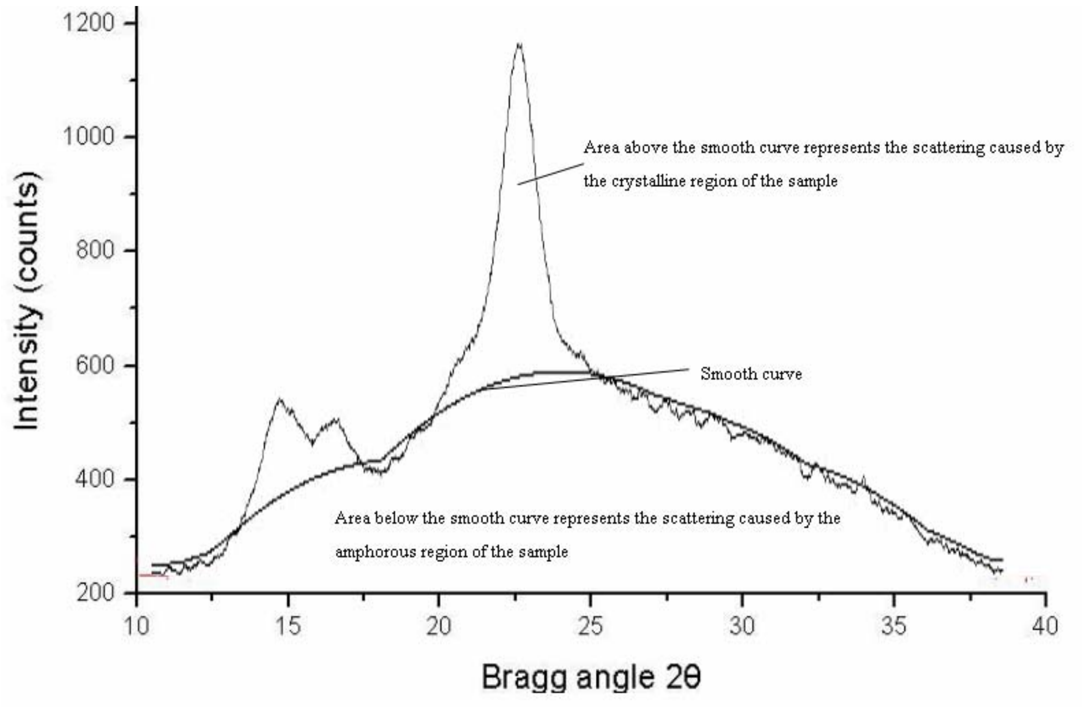

**Figure 3.26** XRD pattern of the second pulverised cotton particles with 2θ ranging from 10 to 40˚

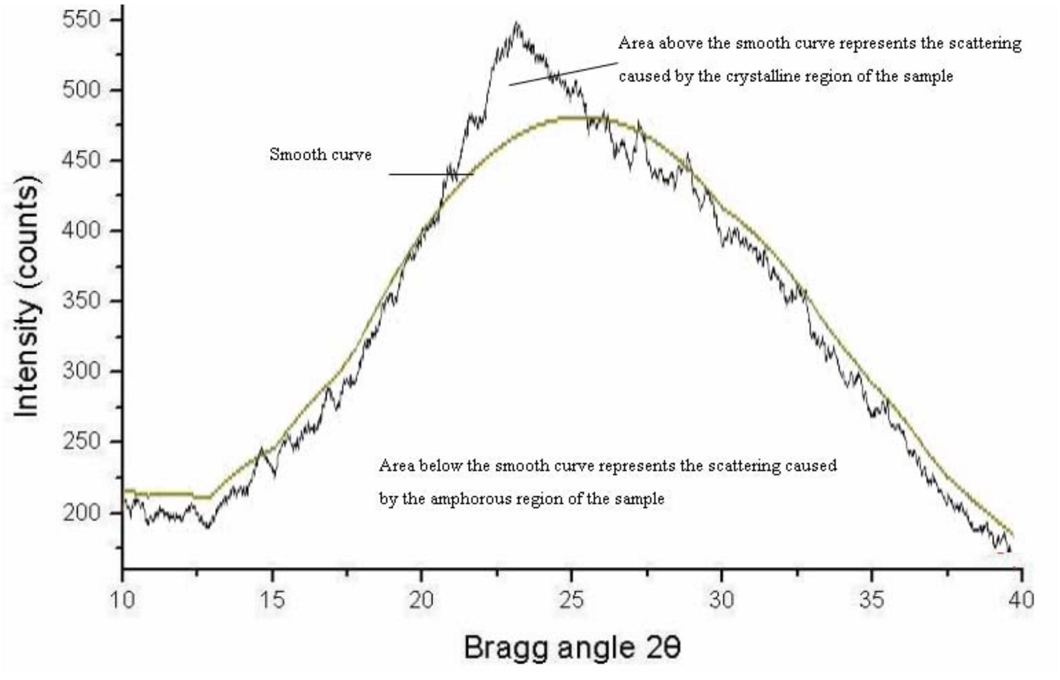

**Figure 3.27** XRD pattern of the third pulverised cotton particles with 2θ ranging from 10 to 40˚

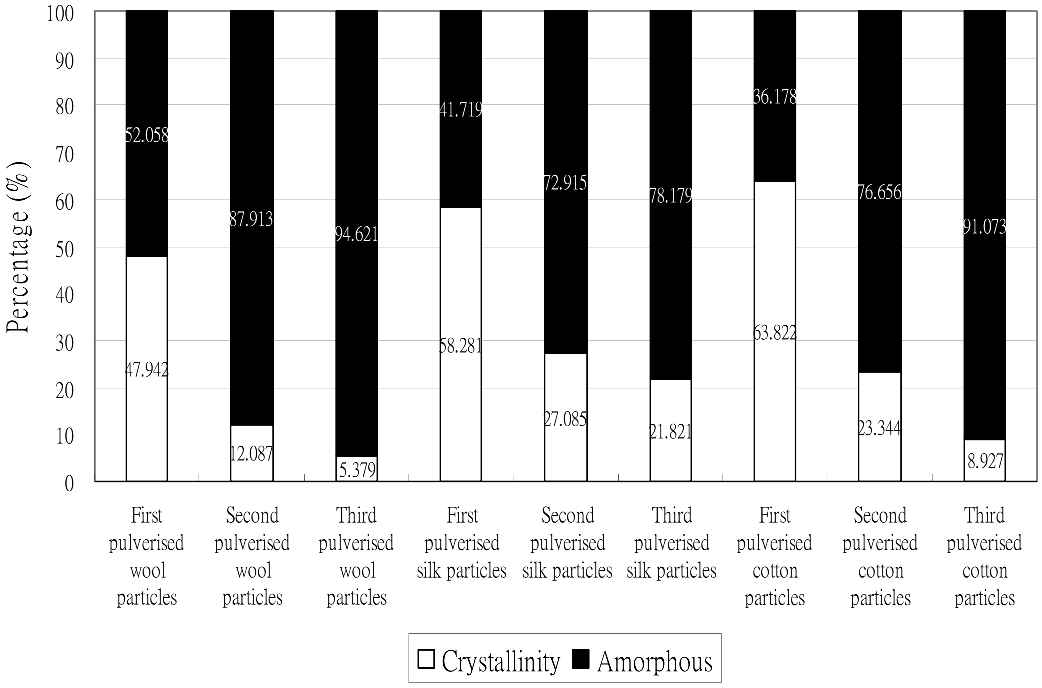

**Figure 3.28** Crystallinity and amorphous percentage of the first, second and third pulverised wool, silk and cotton particles

## 3.4 Summary

The results of particle size analysis and SEM images of the pulverised wool, silk and cotton particles showed that the particle size of these fibres particles decreased from a micro-scale to nano-scale during the progress of the three stage pulverisation. The FTIR spectra of the pulverised wool, silk and cotton particles illustrated that there was no significant change in the chemical structure of the fibres after the pulverisation processes. For the second and third pulverised wool particles, they contained more secondary amine group, amide group, C=S stretching vibration and

O-H bond than those of the wool fibre and the first pulverised wool particles. For the second and third pulverised silk particles, they had more primary aliphatic amines than those of the silk fibre and the first pulverised silk particles. The silk fibre as well as the first and second pulverised silk particles contained the secondary amides, while the third pulverised silk particles did not have them. Furthermore, there were larger amount of the $C(CH_3)_3$ group present in the third pulverised silk particles when compared with the silk fibre, the first and second pulverised silk particles. The third pulverised cotton particles was also different from those of the first and second pulverised cotton particles at the position of $1380cm^{-1}$. This might be due to the presence of the methyl group (C-H) in the third pulverised cotton particles sample. The XRD analysis showed that there was a decrease in the crystallinity of both the pulverised wool, silk and cotton particles with respect to the decreasing particle size.

# Chapter 4 Modification of Cotton Fabric Properties by Nano-Wool Particles

## 4.1 Introduction

Cotton is the most widely used and comfortable materials in the textile industry. Nowadays, many researchers have explored the application of nanotechnology for modifying the properties of cotton fabric in the area of stain, oil and water repellency, wrinkle resistance and ultraviolet protection etc. However, no one has ever attempted to apply nano-scale fibre particles onto the cotton fabric. In this research study, a new usage of nanotechnology in textile industry has been proposed by applying the nano-wool particles onto the cotton fabric. This invention does not only introduce the concept of recycling and reusing the fibre wastes for modifying the properties and performances of textile fabrics, but also helps explore the broad opportunity for applying the nanotechnology to the textile industry.

# 4.2 Methodology

## 4.2.1. Materials

Woven cotton fabrics were treated with nano-wool suspension and the properties of the treated fabrics were compared with those of the untreated one.

The nano-wool particles used were prepared according to the pulverisation procedures mentioned in Chapter 3 except that the amount of nano-wool added was dependent on the experimental conditions shown in Table 4.2. The treatment solution used in the experiment was in suspension form and prepared by adding the acrylic based Ciba Alcoprint PBA binder to the nano-wool suspension at the liquor ratio (volume/volume) of 1:100 correspondingly.

Totally, 16 different experimental conditions were employed to treat the 100% cotton fabrics with 10cm x 10cm in size. In each condition, 3 pieces of cotton fabrics were used. The fabrics were first washed with deionised water and then dried before the treatment. In addition, 3 extra pieces of cotton fabrics were also prepared in the same way and used as control untreated fabrics.

### 4.2.2. Designed Experimental Conditions

16 different experimental conditions were designed based on the fractional factorial design generated by means of the statistical software Minitab Release 14. The design of the experimental conditions was useful to study the effect of four experimental factors on the modification of cotton fabric properties. These four experimental parameters included (i) the amount of nano-wool added, (ii) dipping time of cotton fabrics in the nano-wool suspension, (iii) curing temperature and (iv) curing time. By using 2 levels of the experimental factors, different conditions were designed for treating the fabrics.

In the design of the experiment, two levels of each factor were used as shown in Table 4.1.

**Table 4.1** Two level of factors studied

| Factors | Level 1 | Level 2 |
|---|---|---|
| Amount of nano-wool added | 20g | 40g |
| Dipping time of cotton fabric | 15min | 30min |
| Curing temperature | 100℃ | 130℃ |
| Curing time | 5min | 10min |

For each experimental condition, the amount of nano-wool added, dipping time of cotton fabric, curing temperature and curing time were different as shown in Table 4.2.

**Table 4.2** Experimental condition of nano-wool treatment.

| Samples | Amount of nano-wool added (g) | Dipping time (min) | Curing temperature (℃) | Curing time (min) |
|---|---|---|---|---|
| 1 | 20 | 15 | 100 | 5 |
| 2 | 40 | 15 | 100 | 5 |
| 3 | 20 | 30 | 100 | 5 |
| 4 | 40 | 30 | 100 | 5 |
| 5 | 20 | 15 | 130 | 5 |
| 6 | 40 | 15 | 130 | 5 |
| 7 | 20 | 30 | 130 | 5 |
| 8 | 40 | 30 | 130 | 5 |
| 9 | 20 | 15 | 100 | 15 |
| 10 | 40 | 15 | 100 | 15 |
| 11 | 20 | 30 | 100 | 15 |
| 12 | 40 | 30 | 100 | 15 |
| 13 | 20 | 15 | 130 | 15 |
| 14 | 40 | 15 | 130 | 15 |
| 15 | 20 | 30 | 130 | 15 |
| 16 | 40 | 30 | 130 | 15 |

### 4.2.3. Appling Nano-Wool Particles Onto Fabrics

The nano-wool treated fabrics were prepared by placing the 3 pieces of cotton fabrics into 800ml of the pulverised nano-wool suspension for different duration and then padded 5 times with the suspension as shown in Table 4.2. Finally, the fabrics were dried and cured in an oven at specific temperatures and duration.

### 4.2.4. Data Analysis Method

The changes in properties including air permeability, wrinkle recovery, ultraviolet protection factor, thermal and liquid water transfer properties of the nano-wool treated fabrics were compared with those of the untreated cotton fabric by means of boxplot generated by the SPSS. These boxplots were used to compare the distribution of the aforementioned properties of the three pieces of treated specimens with the three pieces of untreated cotton specimens. Each distribution was represented by a rectangular box. The longer the length of the rectangular box, the greater the variation of the properties among the specimens would be.

The primary goal of screening the fractional factorial design is to identify the ‘vital’ few factors of key variables that influence the response. The Pareto chart provided by the Minitab Release 14 is used for identifying these influential factors by comparing the relative magnitude of the effects. This chart displays the absolute value of the effects and draws a reference line on the chart. Any effect that extends beyond this reference line is potentially important. The reference line corresponds to 0.1. In the experiment, some of the variables between the effects may be too small and so the effect cannot be extended beyond the reference line. However, the Pareto chart is still plotted for providing more ideas about the factors that have the greater effect on the change in the properties of the treated fabrics than the others.

In order to visualise the effects, the main effects plot is used. The main effects plot is actually a plot of the means at each level of a factor so as to compare the magnitude of various main effects.

### 4.2.5. Evaluation of Properties and Functions of Treated Fabrics

The properties of the nano-wool treated cotton fabrics being studied included the morphology, air permeability, wrinkle recovery, ultra-violet protection, thermal properties and liquid water transfer properties behaviour.

#### 4.2.5.1. Scanning Electron Microscopy

The morphology of the treated samples was examined by means of a JSM-6335F Field Emission Scanning Electron Microscope (SEM).

#### 4.2.5.2. Air Permeability

The air permeability of the treated cotton and polyester samples was tested according to the ASTM D737-1996 method by using the Shirley Development Limited Air Permeability Tester.

#### 4.2.5.3. Wrinkle Recovery

The wrinkle recovery of the nano-scale wool particles treated cotton fabrics was determined according to the AATCC Text Method 66-1998.

#### 4.2.5.4. Ultraviolet Protection

The Ultraviolet protection of treated polyester and cotton fabrics was tested by the Cary 300 Conc UV-Visible Spectrophotometer according to the Australian/New Zealand Standard AS/NZS 4399:1996.

#### 4.2.5.5. Liquid Water Transfer Properties

The liquid water transfer properties of the treated cotton fabrics were studied by means of the Moisture management tester (Ref USA Patent 6454814) accompanied by the Moisture Management Tester Programme Version 1.

#### 4.2.5.6. Thermal Properties

The thermal properties of the treated cotton fabrics were evaluated with the aid of the KES-F7 Thermal Labo II (Precise and Prompt Thermal Prosperity Measurement Instrument) which could evaluate not only the warm/cool feeling (q-max value), but also the thermal conductivity and insulation value (keeping warmth ratio) precisely and quickly.

#### 4.2.5.7 Data Analysis

The data analysis of all of these experimental results was evaluated by means of the Statistical Software Minitab Release Version 14 and SPSS Version 11.

## 4.3. Result and Discussion

According to the results shown in Sections 4.3.1 to 4.3.6, the nano-wool treatment could modify the morphology and properties of fabric such as air permeability, wrinkle recovery, ultraviolet protection, thermal properties and moisture management properties. In addition, the Statistical Software Minitab Release Version 14 and SPSS Version 11 were used for analysing the importance and effect of different experimental conditions, i.e. (i) amount of nano-wool added, (ii) dipping time, (iii) curing time and (iv) curing temperature, on the properties of the treated fabrics. The experimental results are discussed in detail in the following.

### 4.3.1. Morphology of Nano-Wool Treated Cotton Fibres

The SEM image of nano-wool treated cotton fabric is shown in Figure 4.1. It was obvious that after the nano-wool treatment, there were numerous nano-wool particles present on the fibre surface blocking the pores of the cotton fibres. By comparing the SEM picture of the untreated cotton fabric with that of the nano-wool treated cotton fabric at the magnification of 1,500x as shown in Figures 4.2 and 4.3, it was found that swelling of cotton fibre did occur after the nano-wool treatment.

Wool is a very absorbent fiber because of its numerous hydrophilic chemical groups and amorphous molecular structure. Wool is also water-repellent. Spilled liquids run off wool because the scale structure of wool inhibits wicking of moisture along the fibre surface (Tortora and Collier 1997).

When wool was pulverised down to nano-scale, the scale on the wool fibres which inhibited moisture was removed. In addition, the experimental result illustrated in Section of 3.3.4.1 of Chapter 3 revealed that nano-wool particles exhibited greater amount of hydrophilic hydroxyl (OH) group and amorphous region than wool fibres. Therefore, nano-wool might perhaps exhibit better hydrophilic properties than wool fibre. In dry cotton, high interchain forces firmly link the molecular chains together. However, after nano-wool treatment, the nano-wool particles present inside the cotton fibres could absorb water. When water was absorbed, it would position itself between the molecular chains. As a result, both water molecules and nano-wool particles present between the molecular chains of cotton reduced the bonding forces of the fibres, making the fibres more flexible and generally inducing swelling (Wallenberger 1978).

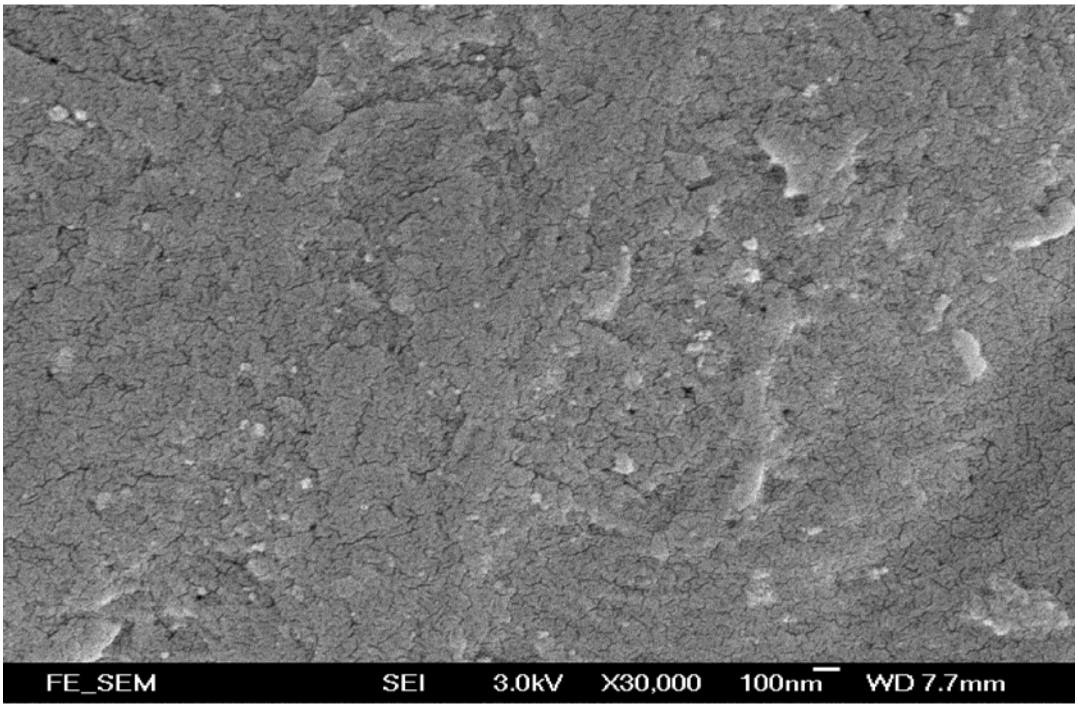

**Figure 4.1** SEM image of nano-wool treated cotton fibre surface of 30,000x

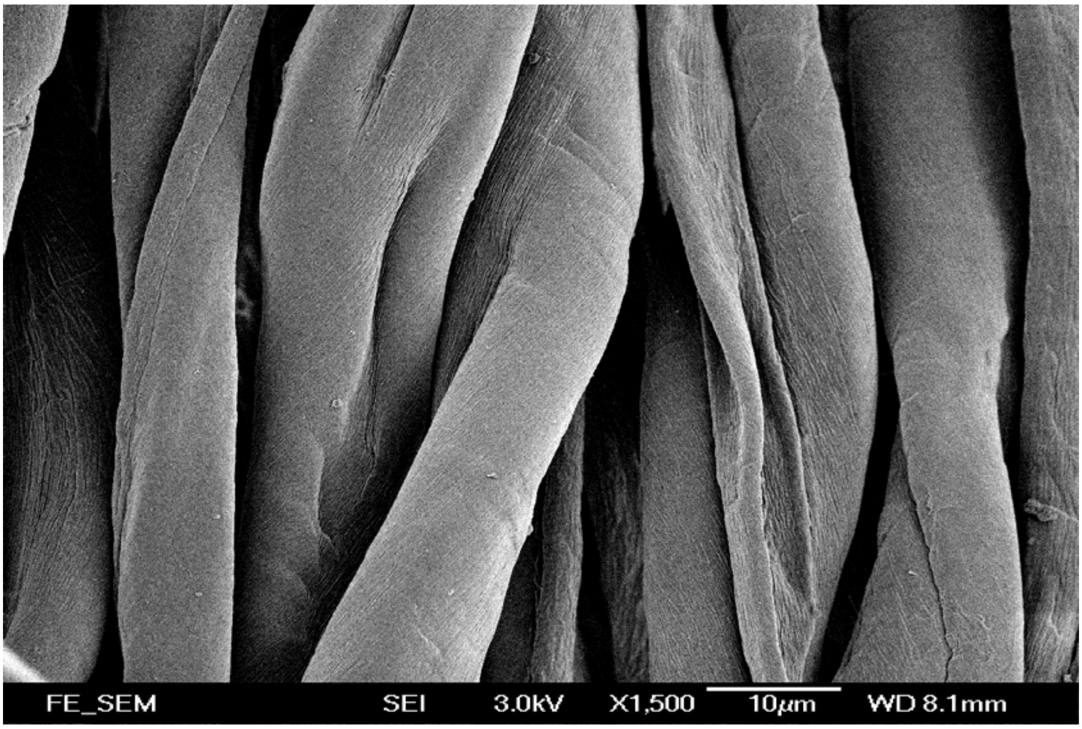

**Figure 4.2** SEM image of untreated cotton fibre of 1,500x

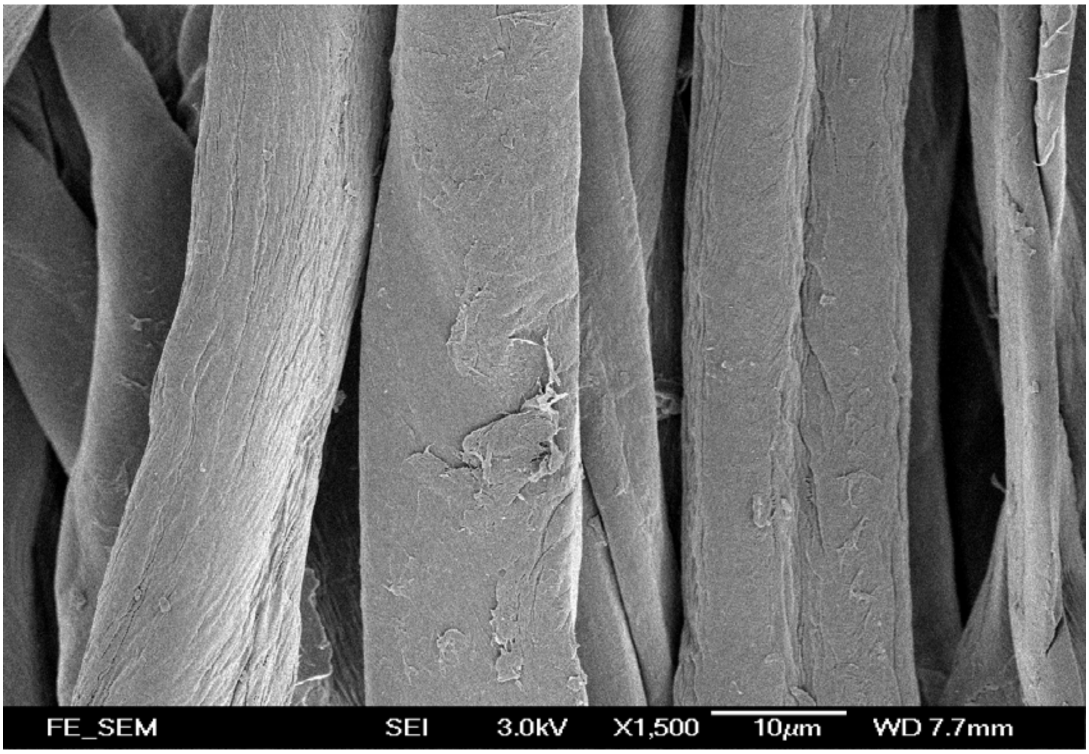

**Figure 4.3** SEM image of nano-wool treated cotton fabric of 1,500x

### 4.3.2. Air Permeability

Figures 4.2 and 4.3 show that after the nano-wool treatment, the cotton fibres would swell thereby reducing the volume of the inter-yarn spacing. Moreover, Figure 4.1 shows that the pores of the cotton fibre surfaces were blocked by the nano-wool particles. It was obvious that both the swelling of fibre and blocking of the pores by nano-wool particles would probably decrease the fabric porosity.

Since the parameters that influenced the air flow per unit fabric area included the fabric porosity, yarn diameter and inter-yarn pores etc. (Yoon and Buckley 1984), thus the decrease in fabric porosity caused by the swelling of fibres and blocking of the pores of cotton fibres might cause the reduction of air flow per unit fabric area. This would result in the decrease in air permeability of the fabrics after the nano-wool treatment as shown in Figure 4.4.

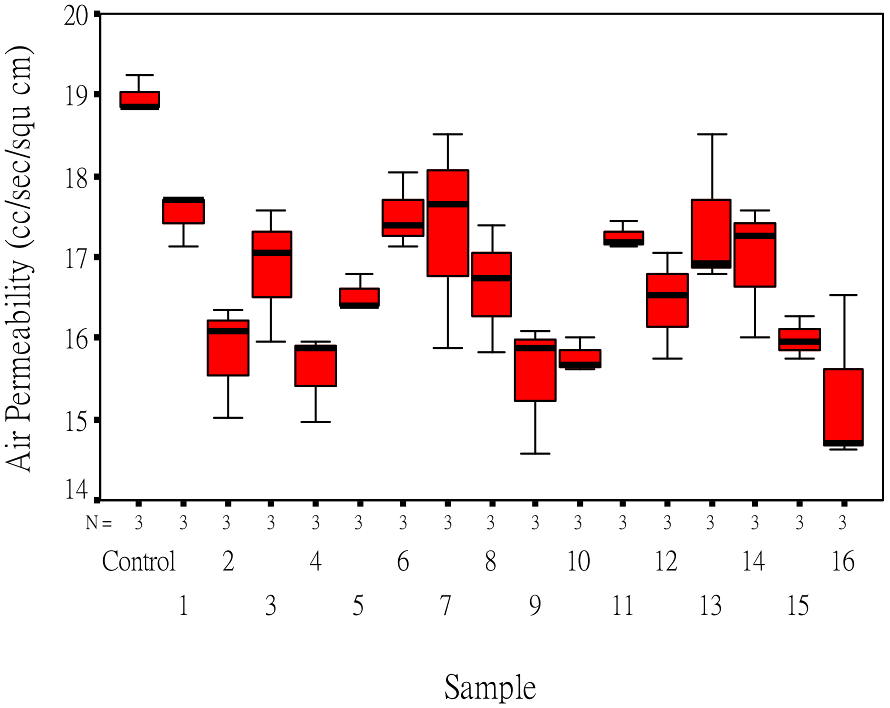

**Figure 4.4** Comparison of air permeability of the untreated cotton fabrics and nano-wool treated cotton fabrics

The Pareto chart with respect to air permeability as shown in Figure 4.5 illustrates that the amount of nano-wool added was the only significant factor that affected the air permeability of the nano-wool treated cotton fabrics.

When a larger amount of nano-wool particles were applied onto the cotton fabrics, the effect of both the swelling of cotton fibres and the blocking of the pores of cotton fibres was enhanced. As a result, there was a decrease in the air permeability of the nano-wool treated cotton fabrics with respect to the increase in the amount of nano-wool added as illustrated in Figure 4.6. Figure 4.6 also demonstrates that there was a reduction of the air permeability of the treated fabric with respect to the increase of dipping time and curing time. On the contrary, there was an increase in air permeability with respect to the elevated curing temperature.

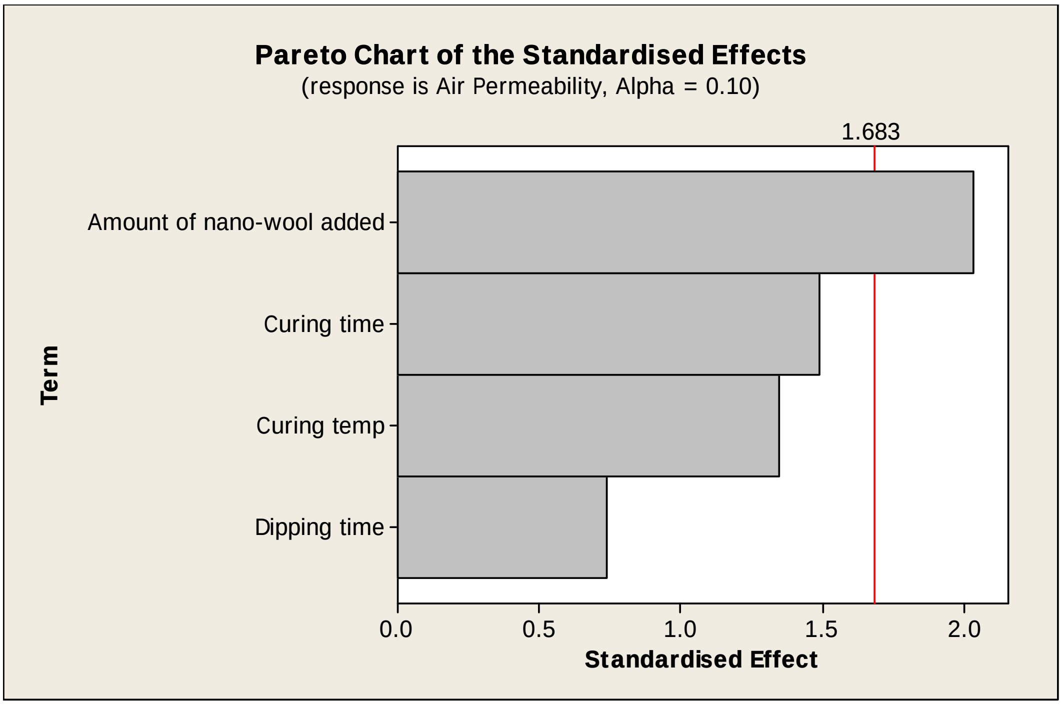

**Figure 4.5** Pareto chart in response to the air permeability of nano-wool treated cotton fabric

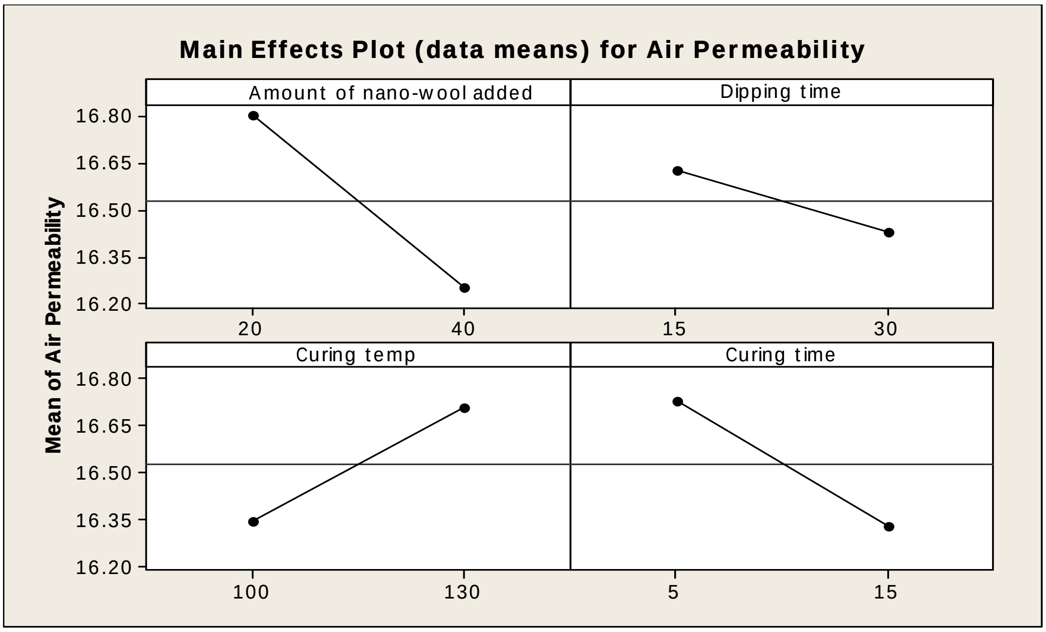

**Figure 4.6** Main effects plot for air permeability of nano-wool treated cotton fabrics

### 4.3.3. Wrinkle Recovery

The wrinkle recovery of the treated cotton fabrics in both warp and weft directions is shown in Figures 4.7 and 4.8 respectively. After the nano-wool treatment, the wrinkle recovery angle of the treated cotton fabrics was enhanced.

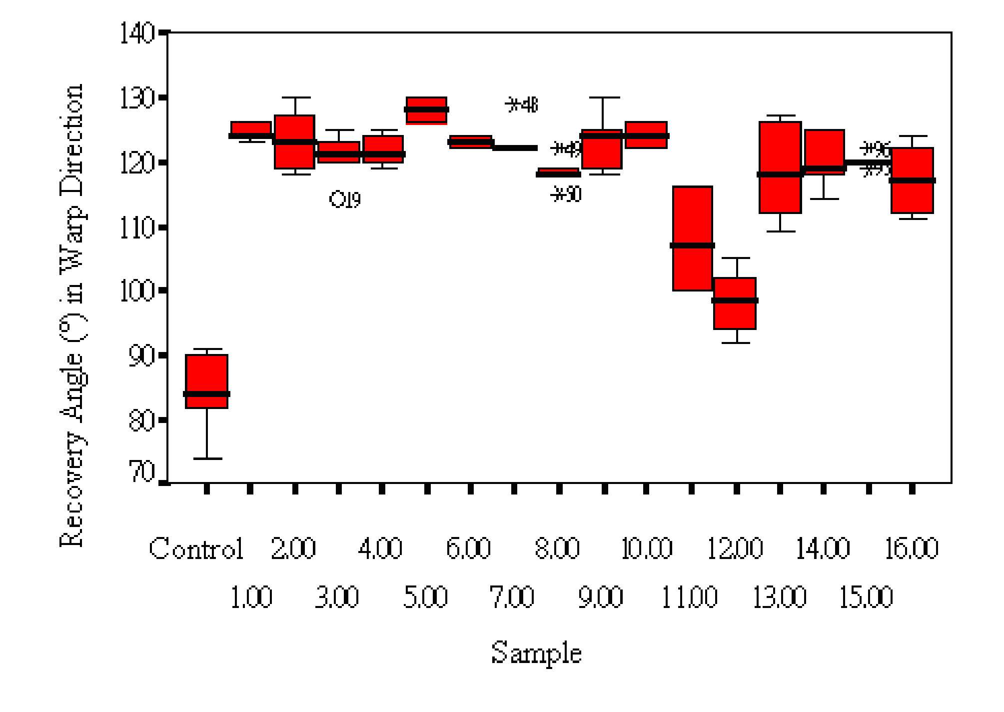

**Figure 4.7** Wrinkle recovery angle of nano-wool treated cotton fabrics in warp direction

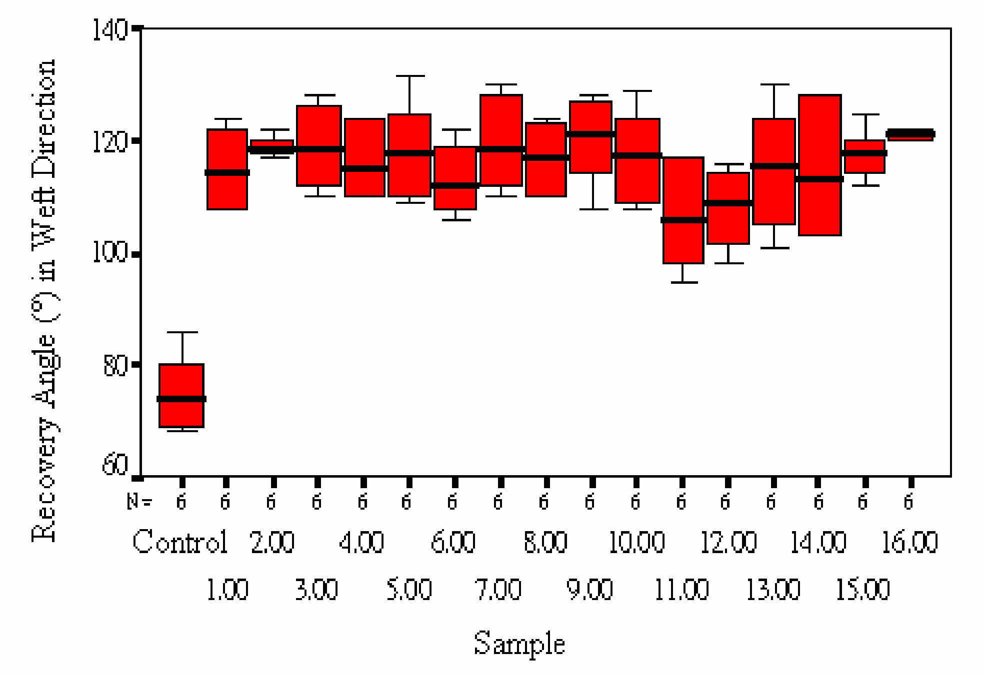

**Figure 4.8** Wrinkle recovery angle of nano-wool treated cotton fabrics in weft direction

Cotton consists of cellulose, a polysaccharide. The cellulose molecules present in cotton fibre are arranged linearly, passing in and out of the crystalline and amorphous regions, and held in place by hydrogen bonds between the molecules. Slippage between the cellulose chains or between larger structural units of the fibre occurs when a force of sufficient magnitude is placed on the fibre. The hydrogen bonds tend to resist or prevent the slippage. However once the slippage occurs, the bonds reform at new locations and tend to maintain the fibre in the bent or wrinkle state. In addition, cotton fibre is hydrophilic and absorbs water, which can break hydrogen bonds and allow the fibre or fabric to shrink. Hence, 100% cotton wrinkles easily and has the potential to shrink upon laundering (Soane *et al.*, 2002).

When nano-wool particles are applied to the fabrics, their atomic level of nano size might allow it to be present in the space between the cellulose chains or between the larger structural units of fibre, thereby enhancing the wrinkle recovery angle of cotton fabric as a result of the unique recovery property of wool particles. The unique recovery property of the wool fibres is due to the helical structure of the protein chains. In nano-scale, the helical structure still exists in the wool particles with a diameter of about 1 nm, and the spacing in the direction of the axis of the helix is 0.15nm (Asquith, 1977 and Lewin *et al.,* 1985). In nano-materials, the surface/interface-to-volume ratio is a very important parameter that affects their properties. When the material is in nano-scale, there is a decrease in the particle diameter of the materials. As a result, the scale of surface atoms will become enlarged, and the surface energy will also be heightened and activated (Li Yi *et al.*, 2003). This phenomenon may also occur in the nano-wool particles, i.e. the interface of the helical structure of wool to volume ratio is so large that it will cause greater effect on the wrinkle recovery of the nano-wool particles.

The sum of the wrinkle recovery angle of the nano-wool treated cotton fabric in both warp and weft directions was used to analyse the effect of different factors on the wrinkle recovery of the treated cotton fabrics. Figure 4.9 shows that curing time was the only significant factor that affected the wrinkle recovery of the nano-wool treated cotton fabric. Figure 4.10 shows that there was a decrease in the sum of wrinkle recovery in both warp and weft directions of the nano-wool treated cotton fabric with respect to the increase in (i) the amount of nano-wool added, (ii) dipping time and (iii) curing time. However, there was an increase in the sum of wrinkle recovery with respect to the elevated curing temperature.

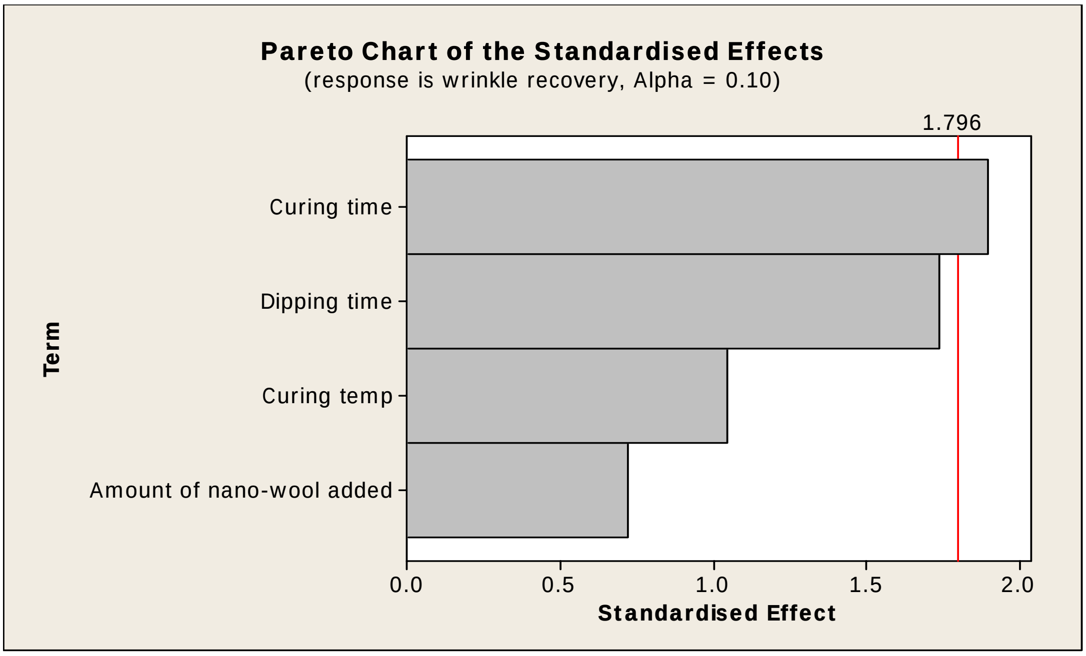

**Figure 4.9** Pareto chart with respect to the wrinkle recovery of nano-wool treated cotton fabric

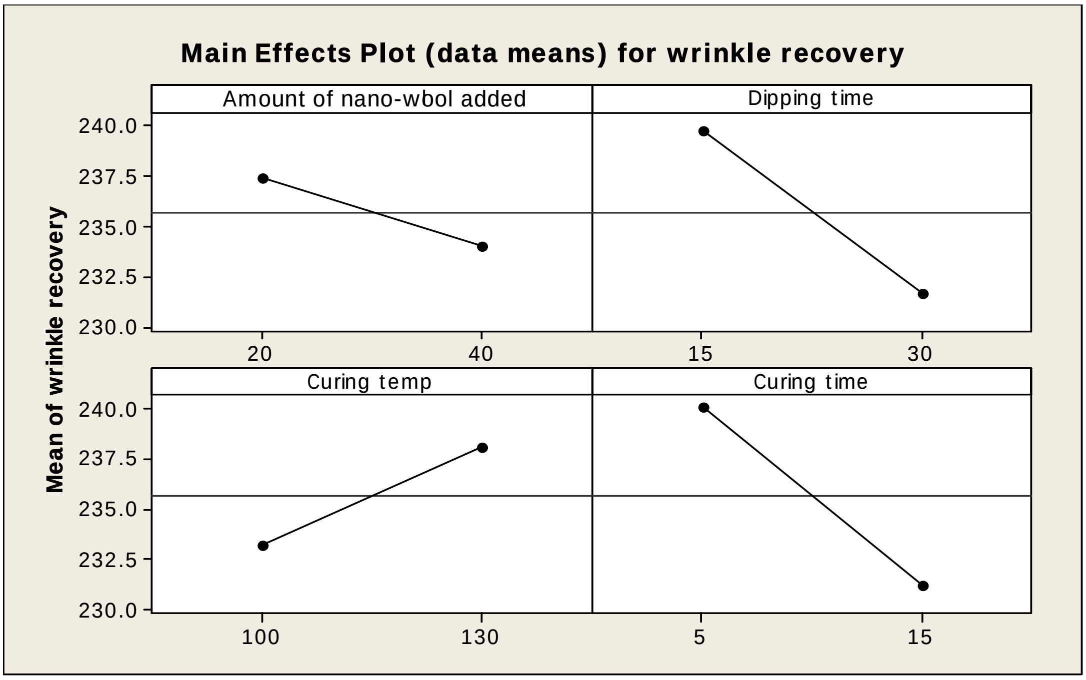

**Figure 4.10** Main effects plot for wrinkle recovery of nano-wool treated cotton fabrics

### 4.3.4. Ultraviolet Protection

The sun protection of the nano-wool treated samples was evaluated in terms of the ultraviolet protection factor (UPF). UPF is the ratio of the average effective UVR irradiance calculated for the unprotected skin to the average effective UVR irradiance calculated for the skin protected by the test fabric. According to the standard, the sun protective clothing shall be categorised according to its UPF for the purpose of labelling as given in Table 4.3.

**Table 4.3.** UPF Classification System

| UPF Range | UVR protection category | Effective UVR transmission, % |
|---|---|---|
| 15 to 24 | Good protection | 6.7 to 4.2 |
| 25 to 39 | Very good protection | 4.1 to 2.6 |
| 40 to 50, 50+ | Excellent protection | ≦2.5 |

The UPF rating of the untreated cotton was non-rateable. Figure 4.11 shows that after the nano-wool treatment, the UPF protection of the treated fabrics was still non-rateable although it was enhanced by at least 45%. For example, for the lowest UPF value of the treated Sample 2, its mean UPF was 10.5, while the mean UPF value of the untreated cotton fabrics was 7.2. Hence, the enhancement of the UPF value of Sample 2 was equal to (10.5 – 7.2) / 7.2 x 100% = 45%.

The Ultraviolet Protection Factor (UPF) value of the cotton fabrics is shown in Figure 4.11. After the nano-wool treatment, the UPF of the treated cotton fabrics was enhanced. This might be due to the swelling of fibres and the blocking of the surface pores by the nano-wool particles, which would inhibit the penetration of ultraviolet ray through the fabric. The enhancement of the ultraviolet protection of the fabric might also be due to the chemical interaction of ultraviolet light with wool particles (Banker *et. al.* 1995, Bradley *et. al.* 1993, Bradley and Mathieson 1997, Smith 1995). This phenomenon might also occur in nano-wool particles causing the ultraviolet light to lose some energy when passing through the nano-wool treated cotton fabric. As a result, the ultraviolet protection of the cotton fabric was enhanced by the nano-wool particles.

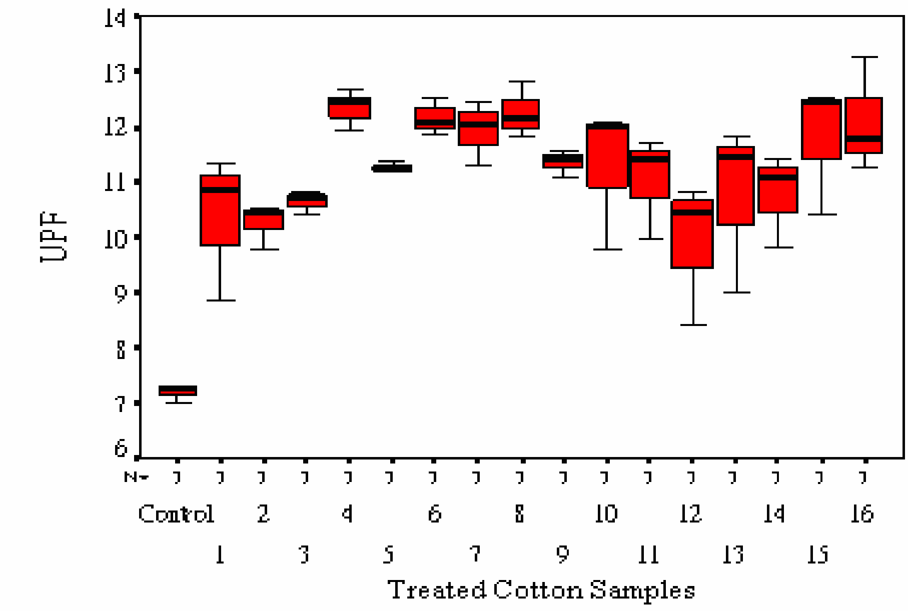

**Figure 4.11** UPF of nano-wool treated cotton fabrics

The Pareto chart with respect to UPF is shown in Figure 4.12 which illustrates that the curing temperature and dipping time were the two significant factors that affected the ultraviolet protection of the nano-wool treated cotton fabric. The curing temperature was the most affected one, while the dipping time was the second main factor. The main effects plot for the ultraviolet protection of nano-wool treated cotton fabrics is shown in Figure 4.13. The ultraviolet protection of the fabrics was enhanced with respect to the increase in the amount of nano-wool added, dipping time and curing temperature. On the contrary, there was a decrease in ultraviolet protection with respect to the prolonged curing time.

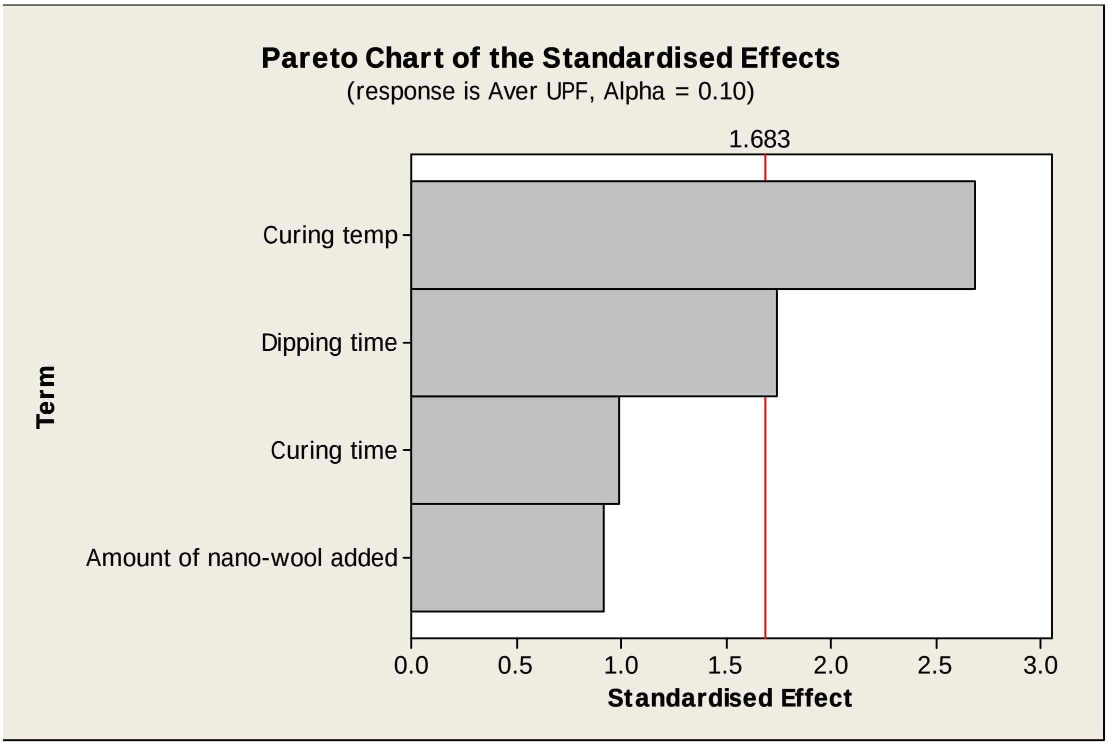

**Figure 4.12** Pareto chart of the UPF value of nano-wool treated cotton fabric

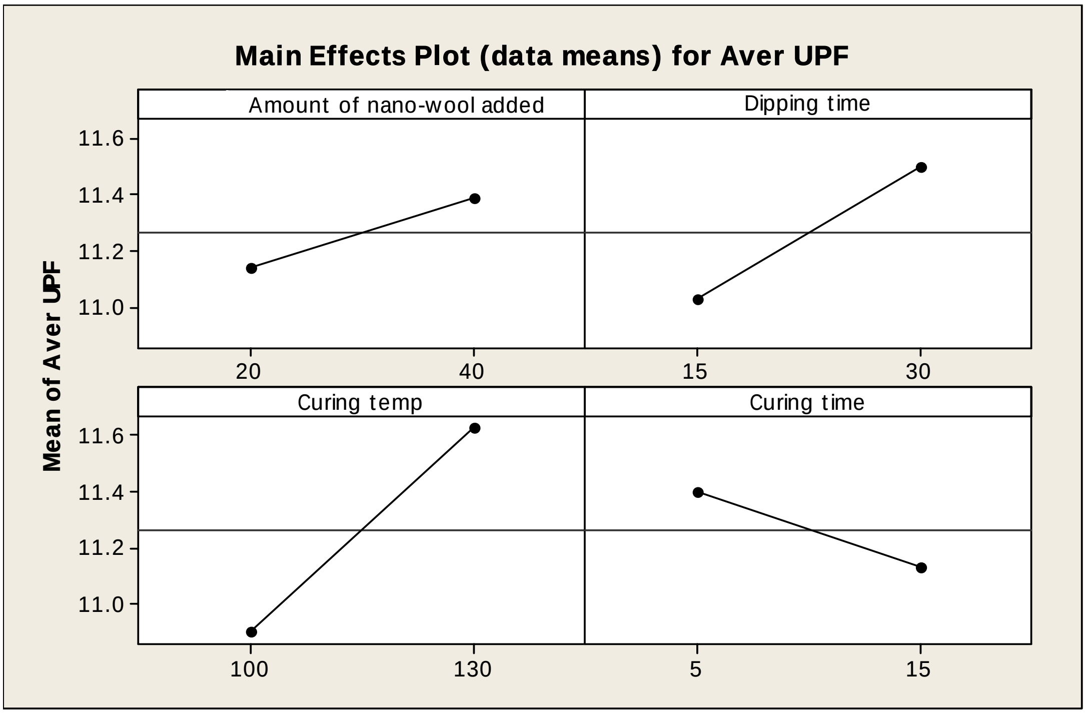

**Figure 4.13** Main effects plot for ultraviolet protection of nano-wool treated cotton fabrics

## 4.3.5. Thermal Properties

The thermal properties of the treated fabrics were evaluated using the KES-F7 Thermal Labo II (Precise and Prompt Thermal Prosperity Measurement Instrument) which could measure not only the warm/cool feeling (q-max value), but also the thermal conductivity and insulation value (keeping warmth ratio) precisely and quickly. The changes in the thermal properties of the treated cotton fabrics are shown in Figures 4.14, 4.17 and 4.20 respectively.

### 4.3.5.1. Thermal Conductivity

Figure 4.14 demonstrates that the nano-wool treatment could lower the thermal conductivity of cotton fabrics. As mentioned in Section 4.3.1, the pores of cotton fibres were blocked by the nano-wool particles after the treatment. According to the experimental results obtained by Schneider *et al.* in 1992, the thermal conductivity of wool (41.4mW/(mK)) was lower than that of cotton fabric (59.7 mW/(mK)). Based on this fact, it was assumed that the nano-wool particles could lower the thermal conductivity of the cotton fabrics. In addition, the Baxter's 1945 paper also mentioned that the thermal conductivity of wool was not only lower than that of cotton, but also lower than that of the air. As a result, when the air space between the cotton fibres was replaced by the nano-wool particles, the thermal conductivity of the cotton fabrics was tremendously reduced. Moreover, as mentioned in Section 4.3.2, the air permeability of the cotton fabrics was reduced after the nano-wool treatment. The reduction in air permeability of the nano-wool treated polyester fabrics implied that less heat could be conducted by air, resulting in poor thermal conductivity of cotton fabrics after nano-wool treatment.

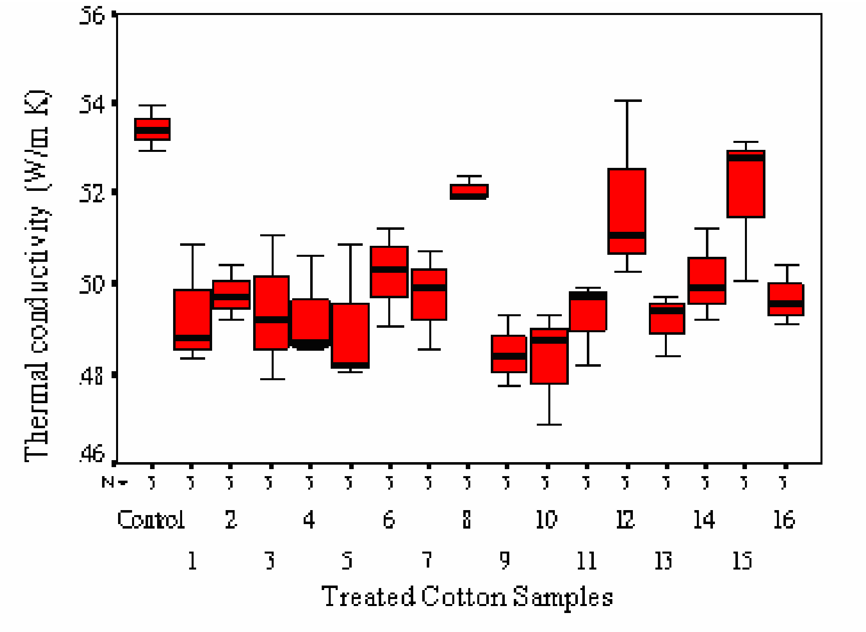

**Figure 4.14** Thermal conductivity of nano-wool treated cotton fabrics

The Pareto chart in response to the thermal conductivity is shown in Figure 4.15 which illustrates that the dipping time and curing temperature were the two significant factors that affected the thermal conductivity of the nano-wool treated cotton fabrics. The thermal conductivity of the treated cotton fabrics was affected mostly by the dipping time followed by the curing temperature.

The main effects plot shown in Figure 4.16 demonstrates that the thermal conductivity of the nano-wool treated cotton fabrics was enhanced with respect to the increase in the amount of nano-wool added, dipping time and curing temperature, but it almost remained unchanged when the curing time was prolonged.

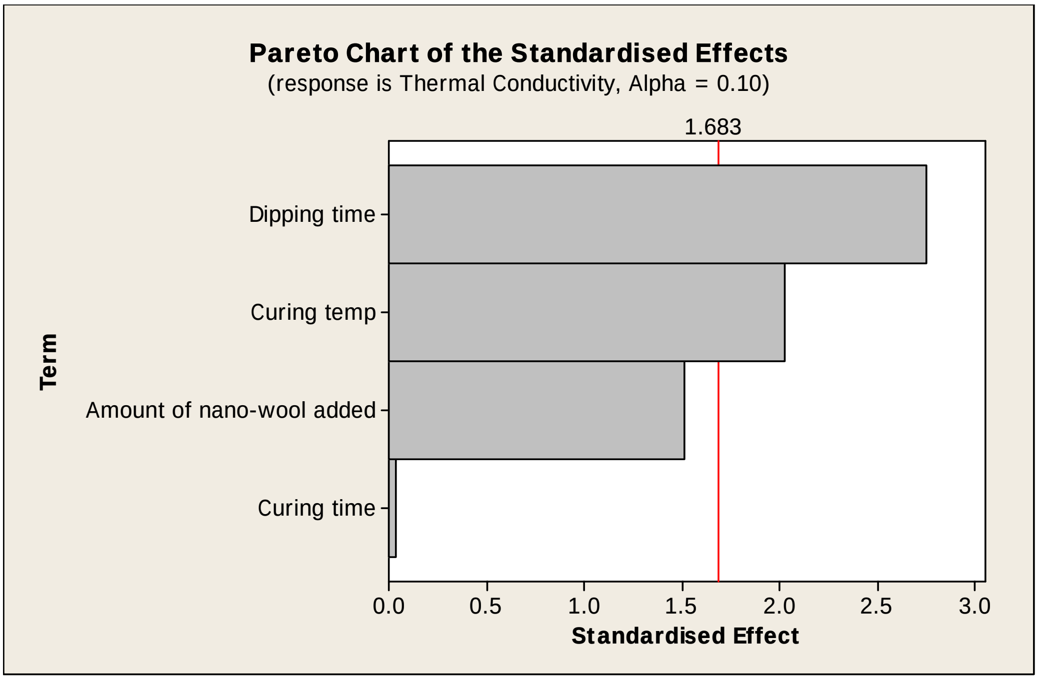

**Figure 4.15** Pareto chart in response to the thermal conductivity of nano-wool treated cotton fabric

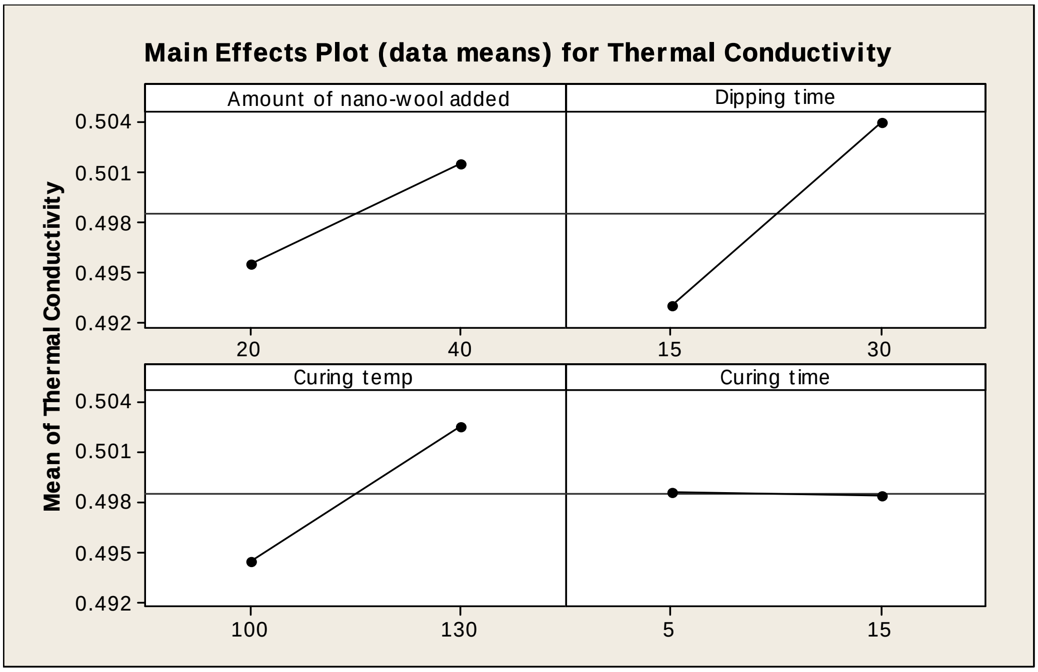

**Figure 4.16** Main effects plot for thermal conductivity of nano-wool treated cotton fabrics

#### 4.3.5.2. Keeping Warmth Ratio

Figure 4.17 illustrates that there was a decrease in the keeping warmth ratio of cotton fabric after the nano-wool treatment. It was probably due to the swelling of cotton fibres after the nano-wool treatment. As mentioned in Section 4.2.1, nano-wool treatment could cause the swelling of cotton fibres. When the cotton fibres swelled, the inter-fibre spacing would decrease causing less amount of air being trapped between the fibres. Since air was a very good heat insulator, thus less amount of air being trapped between the fibres might lead to the reduction in keeping warm ability after the nano-wool treatment.

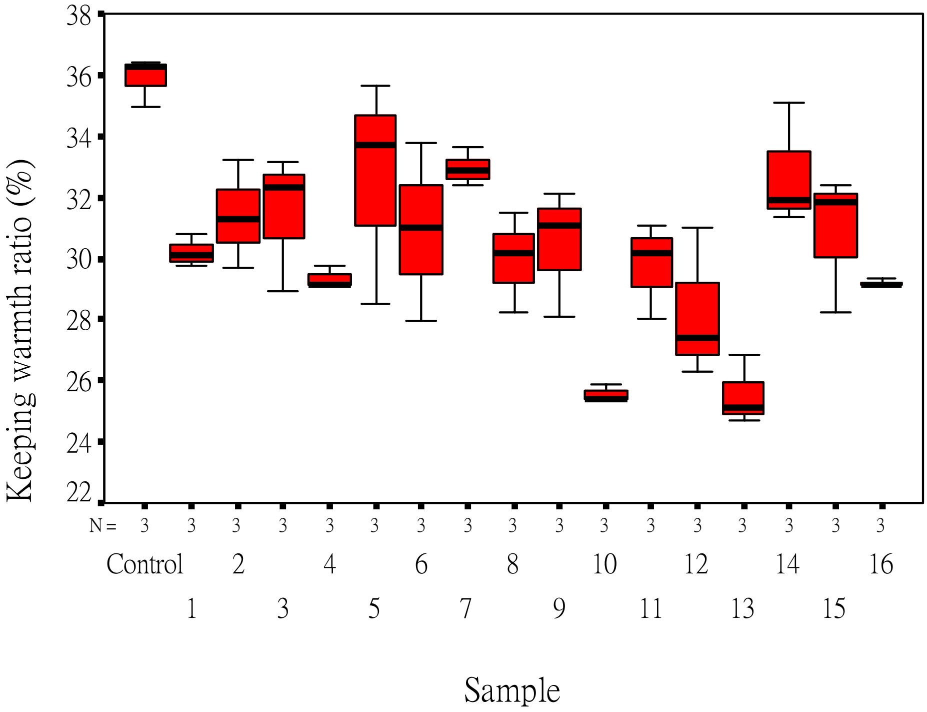

**Figure 4.17** Keeping warmth ratio of nano-wool treated cotton fabrics

The Pareto chart shown in Figure 4.18 revealed that the keeping warmth ratio of the nano-wool treated cotton fabric was significantly affected by the curing temperature followed by the curing time. The main effects plot shown in Figure 4.19 illustrates that when the amount of nano-wool added and the curing time were increased, the keeping warmth ratio of the nano-wool treated cotton fabric was also enhanced. However, there was a decrease in the keeping warmth ratio of the treated cotton fabrics with respect to the prolonged dipping time and elevated curing temperature.

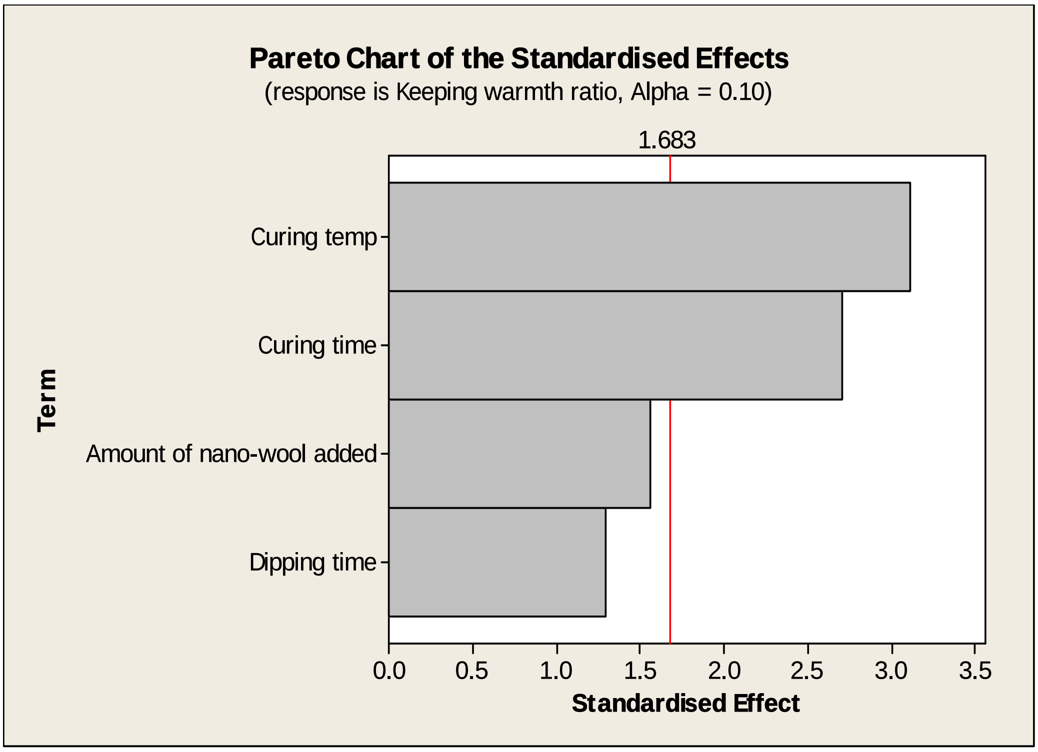

**Figure 4.18** Pareto chart in response to the keeping warmth ratio of nano-wool treated cotton fabrics

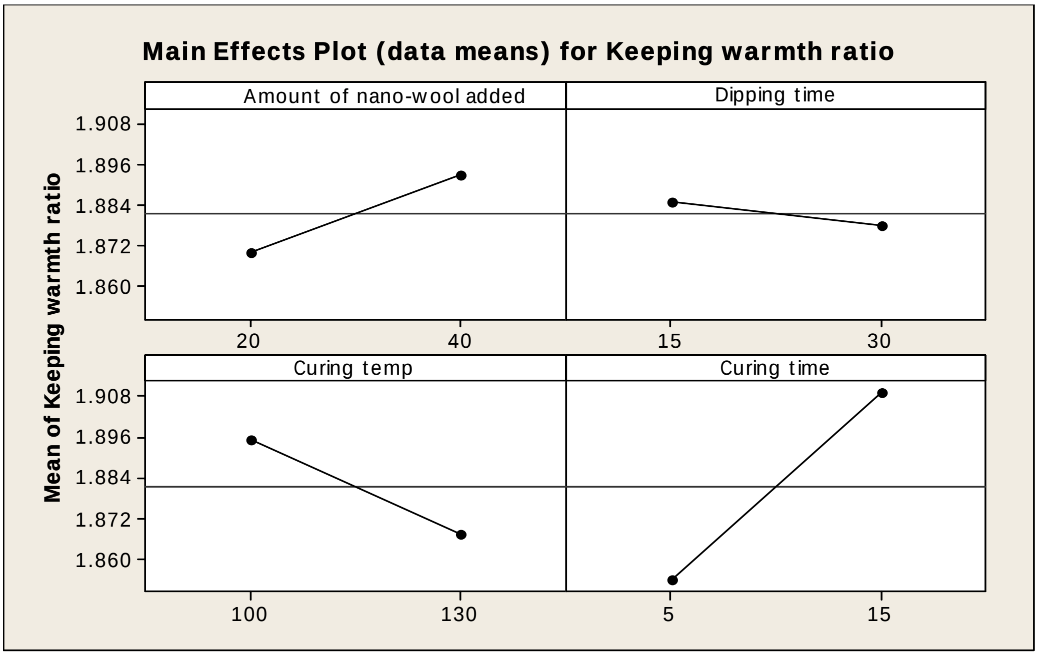

**Figure 4.19** Main effects plot for keeping warmth of nano-wool treated cotton fabrics

### 4.3.5.3. Q-Max Value

Q-max value is an index used to represent the ability of fabrics to provide cool feeling when touching the fabrics. The higher the q-max value, the cooler the feeling will be when touching the fabrics. Figure 4.20 demonstrates that after the nano-wool treatment, the q-max value of the treated cotton fabrics was much lower than that of the untreated cotton fabrics. This meant that when touching the treated cotton fabrics, the feeling was not as cool as that of the untreated cotton fabrics. It was probably due to the fact that the treated cotton fabrics had lower thermal conductivity than that of the untreated one. Therefore, when touching the treated cotton fabrics, the heat generated from our body could not be conducted so easily as that of the untreated one. As a result, the heat trapped by the treated cotton fabrics could provide us with warmer feeling when touching.

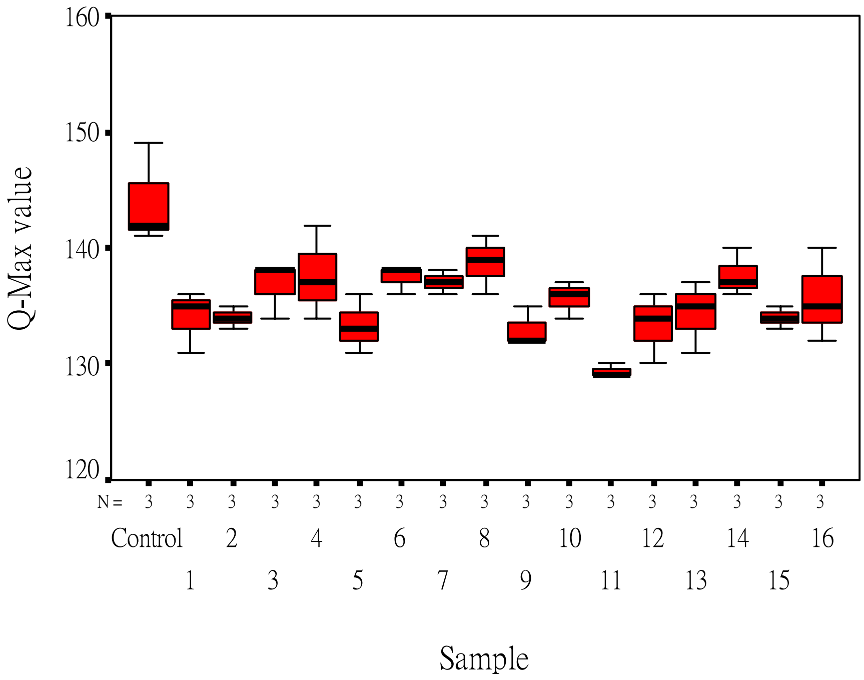

**Figure 4.20** Q-max value of nano-wool treated cotton fabrics

The Pareto chart demonstrated in Figure 4.21 revealed that the q-max value of the nano-wool treated cotton fabric was significantly affected by the amount of nano-wool added followed by the curing time and curing temperature respectively. The main effects plot shown in Figure 4.22 illustrates that there was an increase in the q-max value of the nano-wool treated cotton fabrics with respect to the increasing amount of nano-wool added, dipping time and curing temperature. On the contrary, there was a decrease in the q-max value with respect to the prolonged curing time.

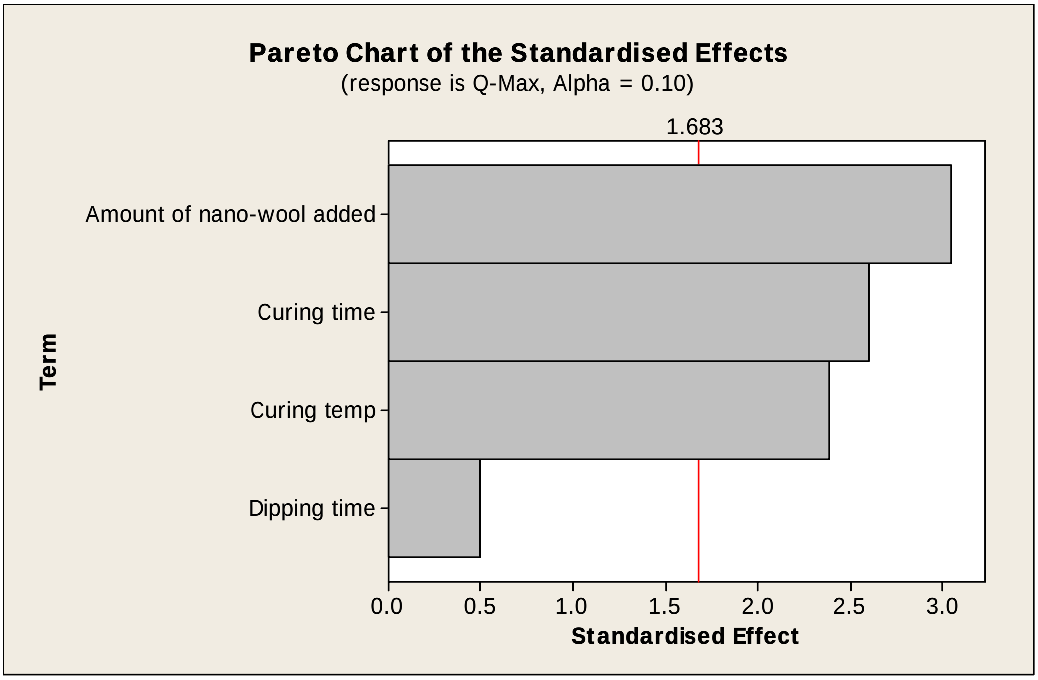

**Figure 4.21** Pareto chart in response to the q-max value of nano-wool treated cotton fabrics

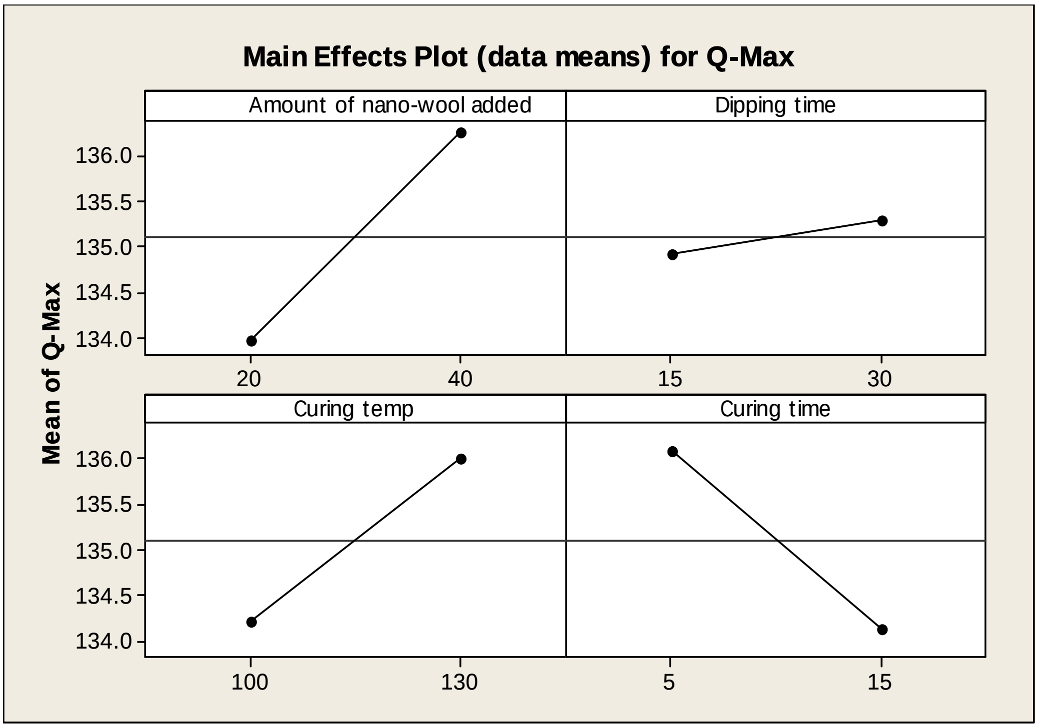

**Figure 4.22** Main effects plot for q-max value of nano-wool treated cotton fabrics

### 4.3.6. Liquid Water Transfer Properties

The moisture management capacity of nano-wool treated cotton fabrics was tested by the moisture management tester. The tester evaluates the moisture management capacity in terms of the Accumulative One-Way Transport Capacity (OWTC) and Overall Moisture Management Capacity (OMMC). OWTC is the difference of the accumulative moisture content between the two surfaces of the fabric in unit testing time period, which is equal to the subtraction of the water content on the fabric top surface from that on the fabric bottom surface in unit testing time. OMMC is an index to indicate the overall capability of the fabric to manage the transport of liquid performance:

1. Moisture absorption rate at bottom side,
2. One way liquid transport capability, and
3. Moisture drying speed at bottom side as represented by the maximum spreading speed.

The larger the OMMC, the higher the overall moisture management capability of the fabric will be.

Figures 4.23 and 4.24 show that nano-wool treated cotton fabrics had lower OWTC and OMMC than those of the untreated cotton fabric respectively. This meant that cotton became more hydrophobic and had poorer moisture management capability after the treatment.

Hydrophobic surfaces can be created mainly in two ways, i.e. (i) by creating a rough structure on a hydrophobic surface, and (ii) by modifying a rough surface using materials with low surface free energy. The enhancement in surface roughness provides a large geometric area for a relatively small projected area. The rough surface in general takes the form of a substrate membrane with a multiplicity of micro-scale to nano-scale projections or cavities.

When nano-wool particles were applied onto the cotton fabric, these tiny particles would crosslink with cellulose and cover the fabric so densely that no liquid could pass through it. This would create nano-scale projections and enhance the roughness of the cotton fibre surface. The water repellency of rough surface was induced by the air trapped between the gaps of the surface. This in turn would enlarge the water/air interface while the solid/water interface was minimised. Hence, the water did not spread out; but formed a spherical droplet instead while remaining on the rough surface (Gulrajani, M. L. 2006).

This concept is similar to the water-repellency effect of the lotus leaves. The ‘lotus effect’ is a feature of nanotechnology. When nanotechnology is applied to textiles, the nano-textured surfaces would bring modified surface tension to textile. The lotus leaves are coated with hydrophobic wax crystals which are around 1 nanometre in diameter. These wax crystals can induce a very fine surface structure to the lotus leaves making the lotus leaf surfaces become rough. This rough surfaces so formed can make the lotus leaves become more hydrophobic. Hence, when cotton was coated with nano-wool particles on its surface, the nano-wool particles acted like the nano-scale wax crystals of the lotus leaves, making the cotton fabrics surface become rough. As a result, the hydrophobility and water-resistant functions of the treated fabric could be obtained (Harper 2004 and Chan 2004).

In addition, when water transmits through fabrics, it does not only transport through the fibres, but also through the inter-yarn spacing between cotton fibres. In fact, it is more easy and faster for water to transport through the inter-yarn spacing than through the fibres. As mentioned in Section 4.3.1, the swelling of cotton fibres occurred after the nano-wool treatment. This in turn reduced the inter-yarn spacing between fibres for water to pass through. The swelling of cotton fibres was the most important factor for causing the hydrophobicity of cotton fabrics after the nano-wool treatment.

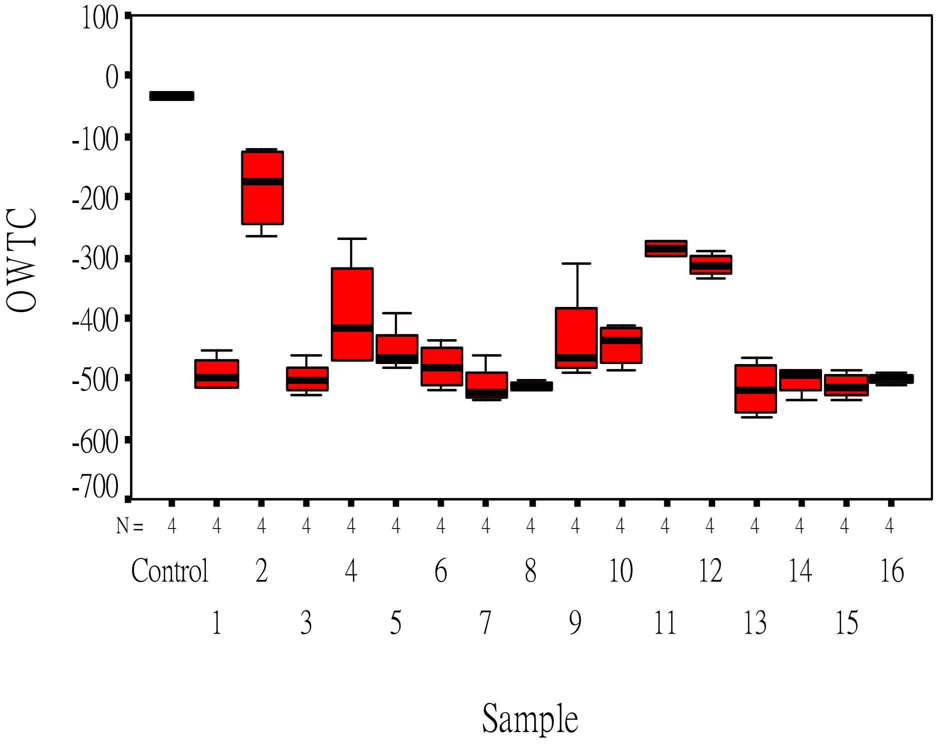

**Figure 4.23** OWTC of nano-wool treated cotton fabrics

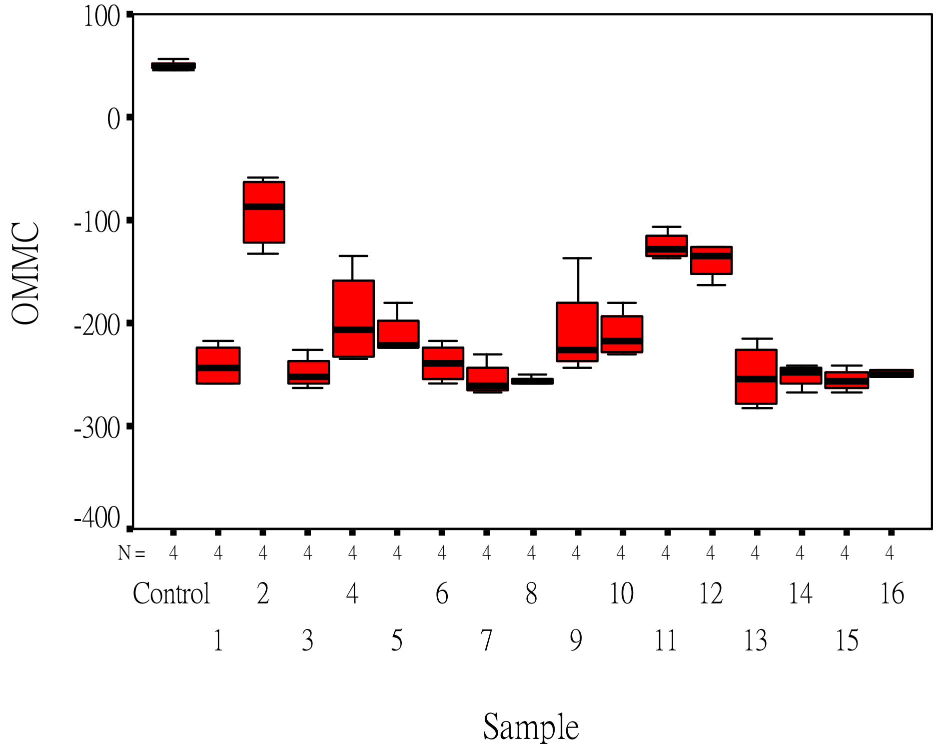

**Figure 4.24** OMMC of nano-wool treated cotton fabrics

The Pareto charts shown in Figures 4.25 and 4.26 revealed that the amount of nano-wool added and curing temperature were the two significant factors that affected the OWTC and OMMC of the nano-wool treated cotton fabrics. When compared, the curing temperature had greater effect than that of the amount of nano-wool added.

The main effects plots shown in Figures 4.27 to 4.28 illustrate that the OWTC and OMMC of the nano-wool treated cotton fabric were enhanced with respect to the increasing amount of nano-wool added, but decreased with respect to the elevated curing temperature. The OWTC and OMMC of the treated cotton fabric almost remained unchanged irrespective of the prolonged dipping time and curing time.

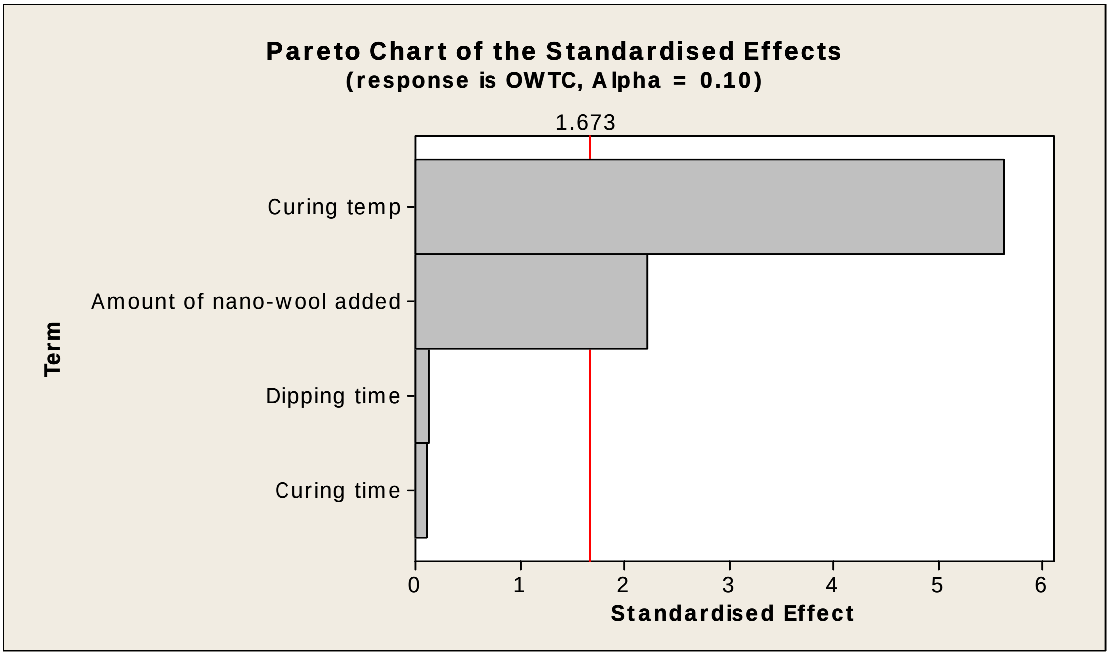

**Figure 4.25** Pareto chart in response to the OWTC of nano-wool treated cotton fabrics

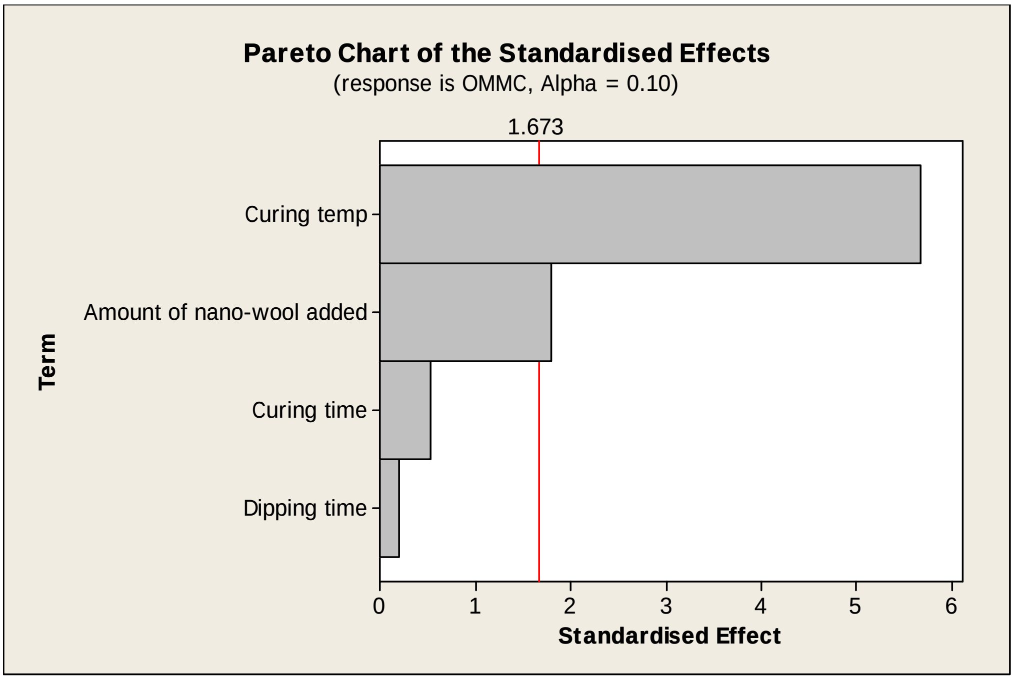

**Figure 4.26** Pareto chart in response to the OMMC of nano-wool treated cotton fabrics

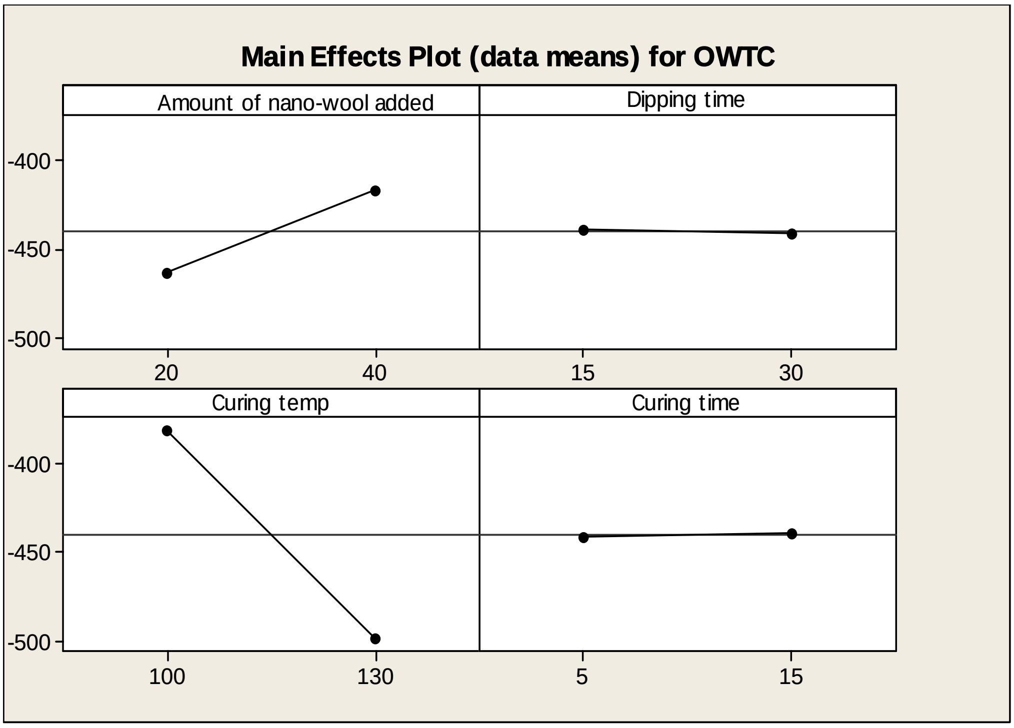

**Figure 4.27** Main effects plot for OWTC of nano-wool treated cotton fabrics

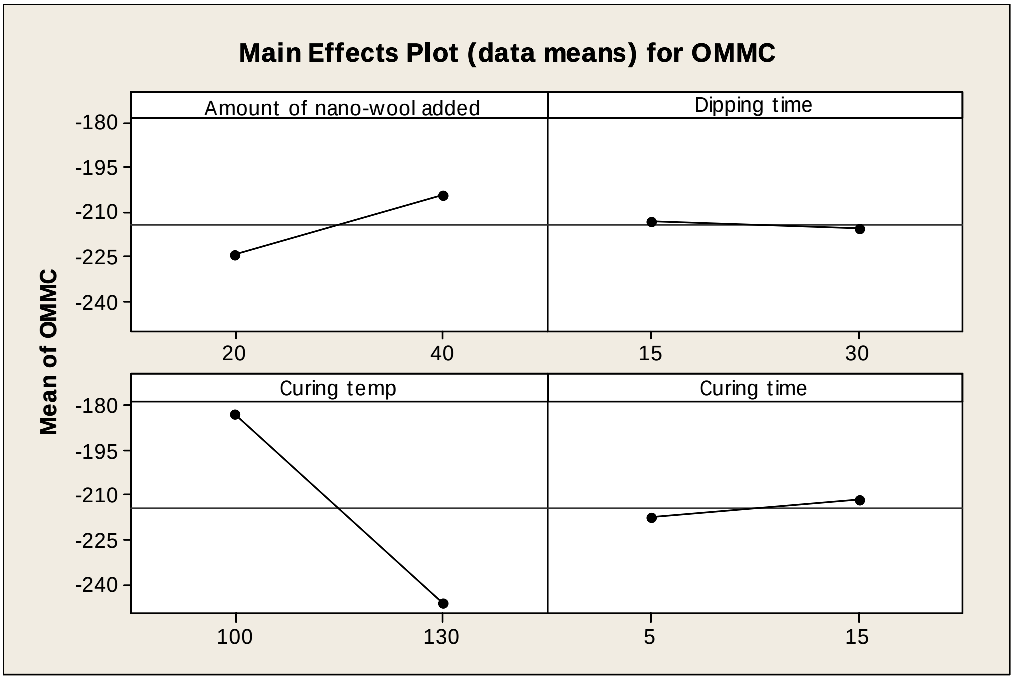

**Figure 4.28** Main effects plot for OMMC of nano-wool treated cotton fabrics

## 4.4. Summary

Attempt to apply nano-wool particles onto cotton fabrics had been done by the pad-dry-cure method. It was found that the thermal properties, liquid water transport properties, ultraviolet protection, air permeability of both the cotton and polyester were modified by the nano-wool treatment.

After the nano-wool treatment, the fibre swelling together with an increase in wrinkle recovery and ultraviolet protection of cotton fabrics did occur. Furthermore, the nano-wool treatment also caused the reduction in air permeability, thermal conductivity, keeping warmth ability, q-max, OWTC and OMMC value of the cotton fabrics. The reduction in q-max values meant that when touching the treated cotton samples, the feeling was not as cool as that of the untreated cotton fabrics. On the other hand, the lower in OWTC and OMMC values after the treatment meant that the cotton fabrics became more hydrophobic and had poorer moisture management capability.

In this research, the fractional factorial design generated by means of the Minitab Release 14 was used is to identify the effect of ‘vital’ few variables on the properties of the cotton fabrics. These valuables included (i) the amount of nano-wool added, (ii) dipping time, (iii) curing time and (iv) curing temperature.

The summary of the effect of these factors on the properties of the nano-wool treated cotton fabrics is recorded in Table 4.4. The one with double marking of arrows represents the most significant factor that affects the properties of the cotton fabric.

Table 4.4 illustrates that the increase in the amount of nano-wool added would result in the reduction of air permeability, wrinkle recovery and hydrophobicity. On the contrary, there was an increase in the UPF value, thermal conductivity, keeping warmth ratio and q-max value of the treated cotton fabrics.

It was also demonstrated that the increase in dipping time could cause the rise of

UPF value, thermal conductivity and q-max value. On the contrary, there was a decline in the air permeability, wrinkle recovery and keeping warmth value of the nano-wool treated cotton fabrics with respect to the prolonged dipping time.

When the curing time was prolonged, there was a decrease in air permeability, wrinkle recovery, UPF, q-max of the treated cotton fabrics, but an increase in keeping warmth ratio. Higher curing temperature could cause the enhancement in all the properties of the treated fabrics except the keeping warmth ratio.

In summary, the variety of properties as well as the performance of the nano-wool treated cotton fabrics were greatly affected by (i) the amount of nano-wool added, (ii) dipping time, (iii) curing temperature and (iv) curing time of the treatment in different ways.

**Table 4.4.** Comparison of the effect of different parameters on the properties of nano-wool treated cotton fabrics

|  | Air permeability | Wrinkle recovery | UPF | Thermal conductivity | Keeping warmth ratio | Q-max value | Hydrophobicity |
|---|---|---|---|---|---|---|---|
| Increase in amount of nano-wool added | ↓ ↓ | ↓ | ↑ | ↑ | ↑ | ↑ ↑ | ↓ |
| Increase in dipping time | ↓ | ↓ | ↑ | ↑ ↑ | ↓ | ↑ | - |
| Increase in curing time | ↓ | ↓ ↓ | ↓ | - | ↑ ↑ | ↓ | - |
| Increase in curing temperature | ↑ | ↑ | ↑ ↑ | ↑ | ↓ | ↑ | ↑ ↑ |

Remark: The double marking of arrows represents the most significant factor that affects the properties of the nano-wool treated cotton fabrics.

# Chapter 5 Modification of Polyester Fabric Properties by Nano-Wool Particles

## 5.1 Introduction

Polyester is the most widely used synthetic fibre today. It is commonly used in a wide range of wearing apparel, home furnishings and industrial products, either alone or in blends (Collier and Tortora, 2001). The major advantages of polyester fibre are its ease of care and wrinkle resistance. It also has very good ultraviolet protection property. However, it is hydrophobic and has a very low moisture regain and liquid transfer properties, making it uncomfortable to wear (Peyton B. Hudson *et al.*, 1993). In this chapter, the modification of liquid water transfer properties by nano-wool treatment is proposed. Furthermore, the effect of nano-wool particles on the morphology, air permeability, thermal properties of polyester fabrics will also be studied.

## 5.2 Methodology

In the research work, polyester woven fabrics were treated with nano-wool particles using the same treatment method as that of the cotton fabrics as mentioned in Section 4.2 of Chapter 4. The properties of the nano-wool treated polyester fabrics were compared with those of the untreated polyester fabrics. The properties being studied included morphology, air permeability, thermal properties and liquid water transfer properties. The methods used for the evaluation of these properties and data analysis were also the same as those mentioned in Chapter 4.

## 5.3. Result and Discussion

The experimental results confirmed that the nano-wool treatment could modify the morphology and properties of polyester fabric such as air permeability, thermal and liquid water transfer properties as shown in Sections 5.3.1 to 5.3.4. In addition, the Statistical Software Minitab Release Version 14 and SPSS Version 11 were used for analysing the importance and effect of different experimental conditions, such as (i) amount of nano-wool added, (ii) dipping time, (iii) curing time and (iv) curing temperature, on the properties of the treated polyester fabrics.

## 5.3.1. Morphology of Nano-Wool Treated Polyester Fibres

The SEM image of nano-wool treated polyester fabric at the magnification of 1,000x is shown in Figures 5.2. It was obvious that after the nano-wool treatment, there were a lot of nano-wool particles present on the polyester fibre surface. Figures 5.1 and 5.2 also demonstrate that nano-wool particles did not cause the swelling of polyester fibres.

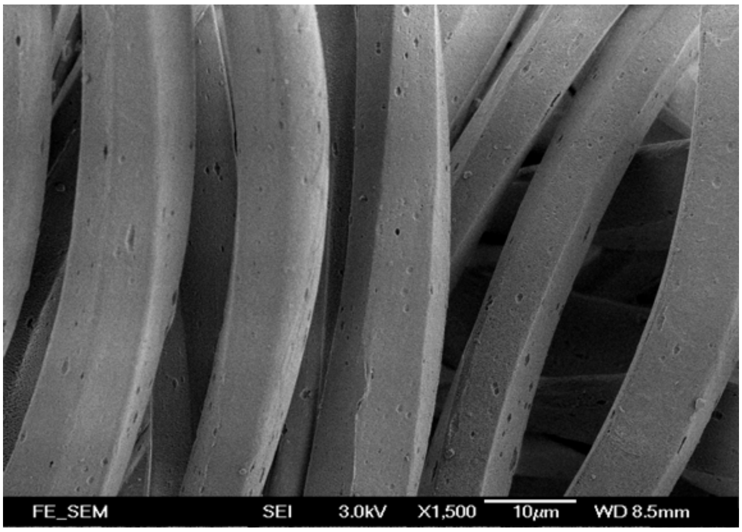

**Figure 5.1** SEM image of untreated polyester fibres of 1,500x

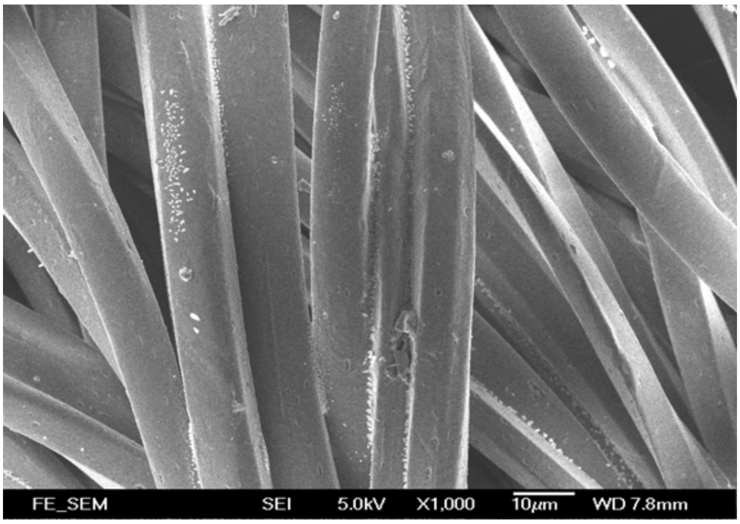

**Figure 5.2** SEM image of nano-wool treated polyester fibres of 1,000x

### 5.3.2. Air Permeability

As mentioned in Section 4.3.2 of Chapter 4, the fabric porosity is one of the factors affecting the air flow per unit fabric area, i.e. the air permeability of the fabrics. Figure 5.1 shows that the pores of the polyester fibre surfaces were blocked by the nano-wool particles. This would reduce the fabric porosity and might cause the reduction of air flow per unit fabric area, resulting in the decrease in air permeability of the nano-wool treated polyester fabrics. Moreover, as mentioned in Chapters 3 and 4, nano-wool particles exhibited better hydrophilic properties when compared with wool fibres because it possessed greater amount of hydroxyl group and amorphous region. When nano-wool was present on the polyester surface, it would attract more water molecules from the environment. Both the water molecules and nano-wool particles present on the polyester fibre surface would occupy inter-yarn spacing of polyester fabrics and reduce the spacing for air to pass through. Hence, the air permeability of polyester was declined after nano-wool treatment as shown in Figure 5.3.

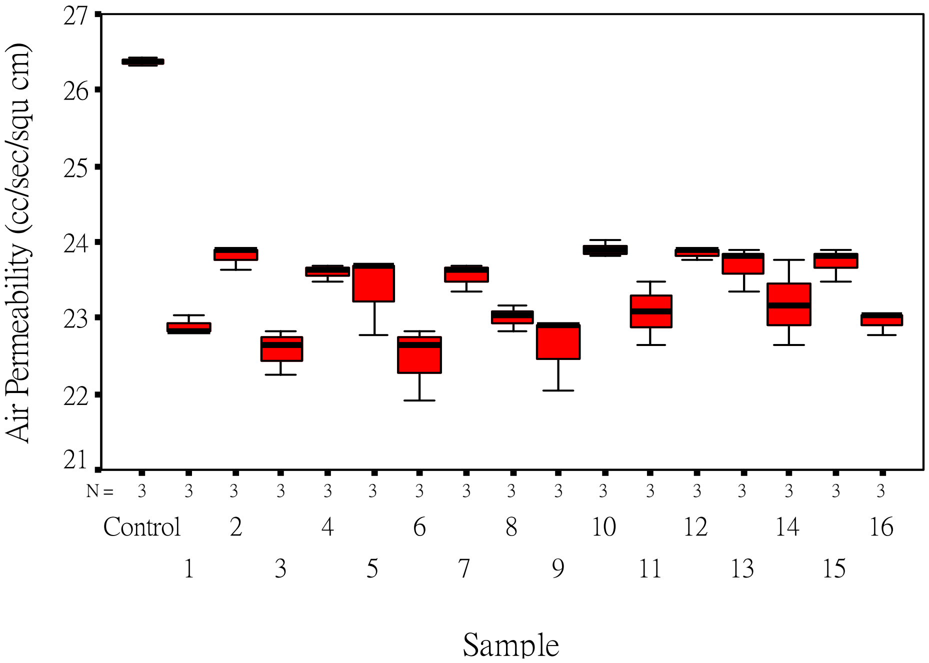

**Figure 5.3** Comparison of air permeability of the untreated polyester fabric and nano-wool treated polyester fabrics

The Pareto chart shown in Figure 5.4 illustrated that there were no significant factors that affected the air permeability of the nano-wool treated polyester fabrics. However, it was obvious that the curing time had greater effect on the air permeability of the polyester fabrics when compared with the other factors. The main effects plot shown in Figure 5.5 revealed that the air permeability of the nano-wool treated polyester fabric would increase with respect to greater amount of nano-wool particles added, longer dipping time and prolonged curing time, but would decrease with respect to the elevated curing temperature.

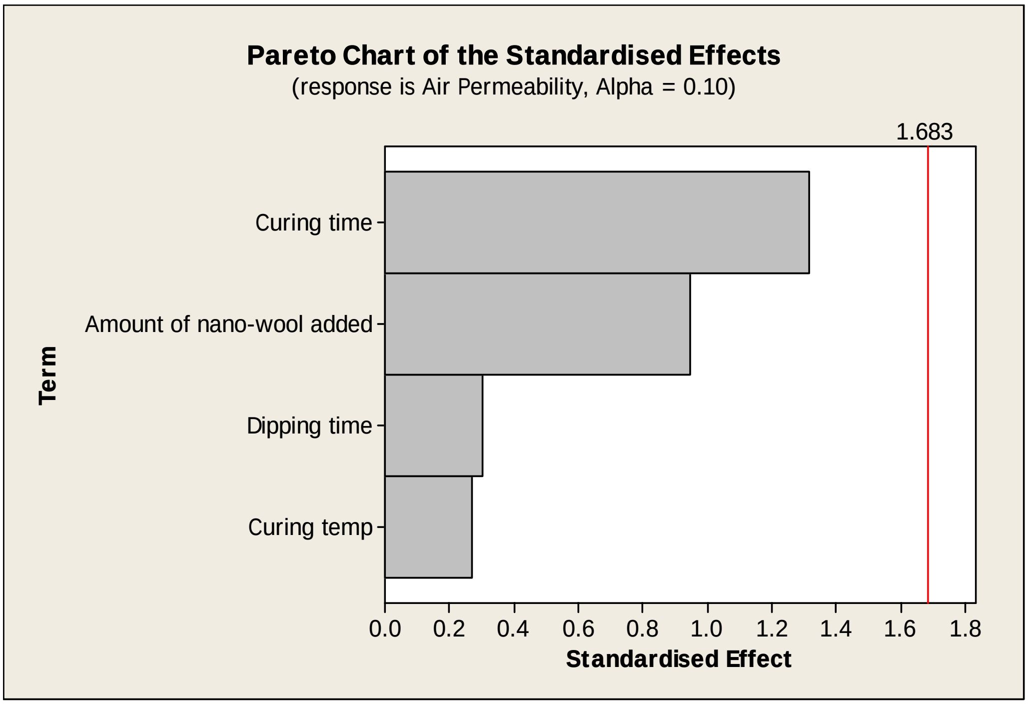

**Figure 5.4** Pareto chart of the air permeability of nano-wool treated polyester fabrics

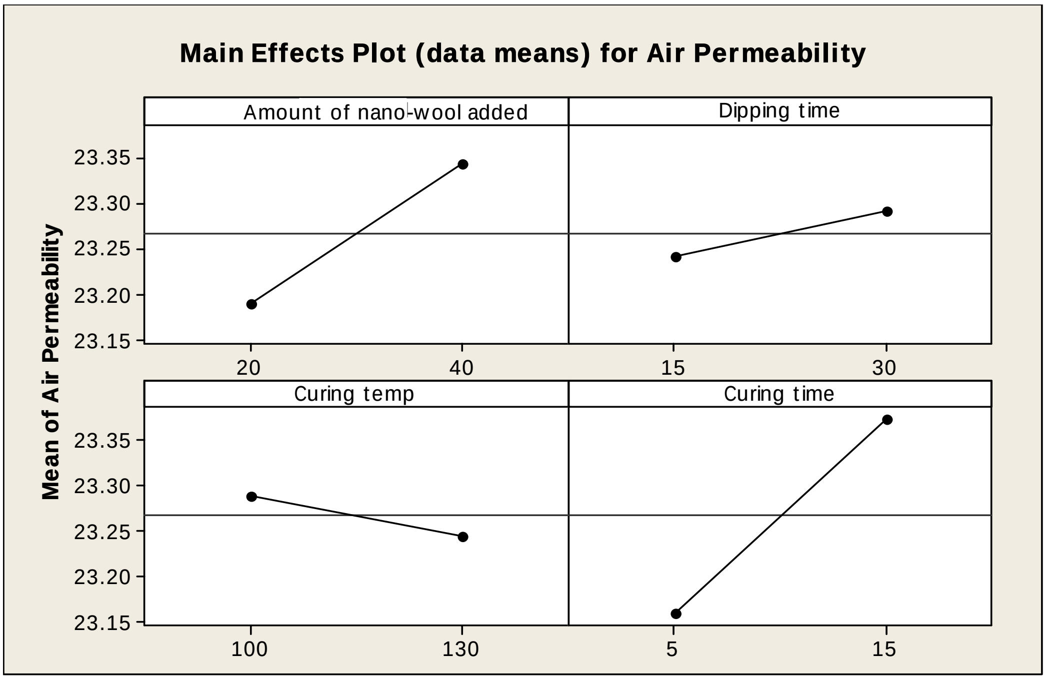

**Figure 5.5** Main effects plot for air permeability of nano-wool treated polyester fabrics

### 5.3.3. Thermal Properties

The thermal properties of the treated polyester fabrics were studied in the same ways as those of the treated cotton fabrics mentioned in Section 4.3.5 of Chapter 4. The properties were evaluated by using the KES-F7 Thermal Labo II (Precise and Prompt Thermal Prosperity Measurement Instrument). The evaluation covered precisely not only the warm/cool feeling (q-max value), but also the thermal conductivity and insulation value (keeping warmth ratio). Figures 5.6, 5.9 and 5.12 demonstrate the change in thermal properties of polyester fabrics after nano-wool treatment.

#### 5.3.3.1. Thermal Conductivity

The experimental results shown in Figure 5.6 revealed that the nano-wool treatment could reduce the thermal conductivity of polyester fabrics. This was probably due to the fact that the air permeability of polyester was reduced after the nano-wool treatment as mentioned in Section 5.3.2. The lower air permeability of the nano-wool treated polyester fabrics implied that less heat would be conducted by air (L. Schacher et. al., 2000). As a result, there was a reduction in the thermal conductivity of polyester fabrics after the nano-wool treatment.

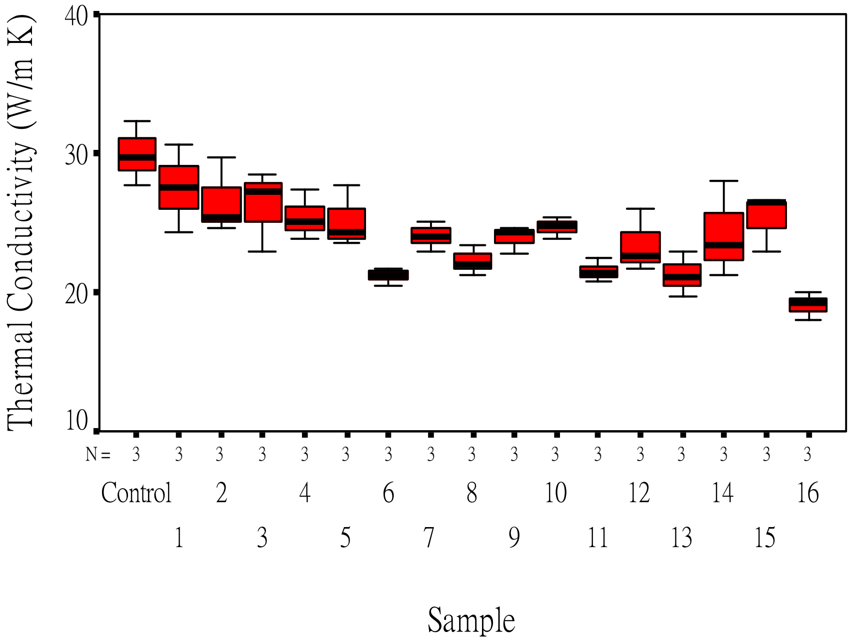

**Figure 5.6** Thermal conductivity of nano-wool treated polyester fabrics

Figure 5.7 demonstrates that the thermal conductivity of nano-wool treated polyester fabrics was affected significantly by the curing time and curing temperature. When compared, the curing temperature had greater effect on the thermal conductivity of the treated fabrics than the curing time.

Figure 5.8 reveals that the thermal conductivity of the nano-wool treated polyester fabrics would increase with respect to elevated curing temperature, prolonged dipping and curing times, but would decrease with respect to the greater amount of nano-wool added.

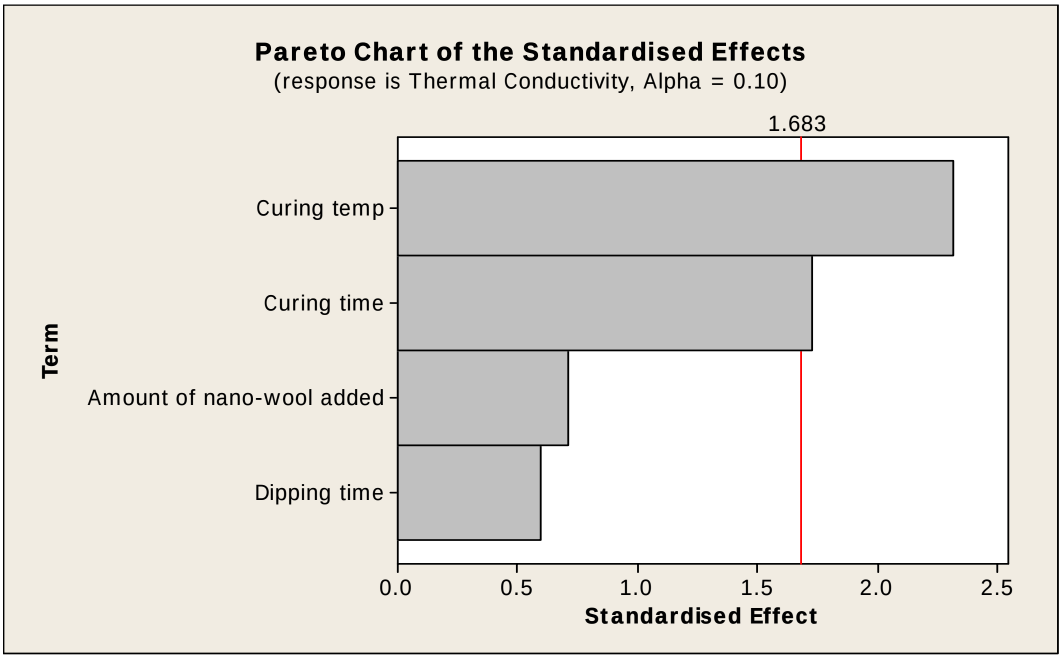

**Figure 5.7** Pareto chart of the thermal conductivity of nano-wool treated polyester fabrics

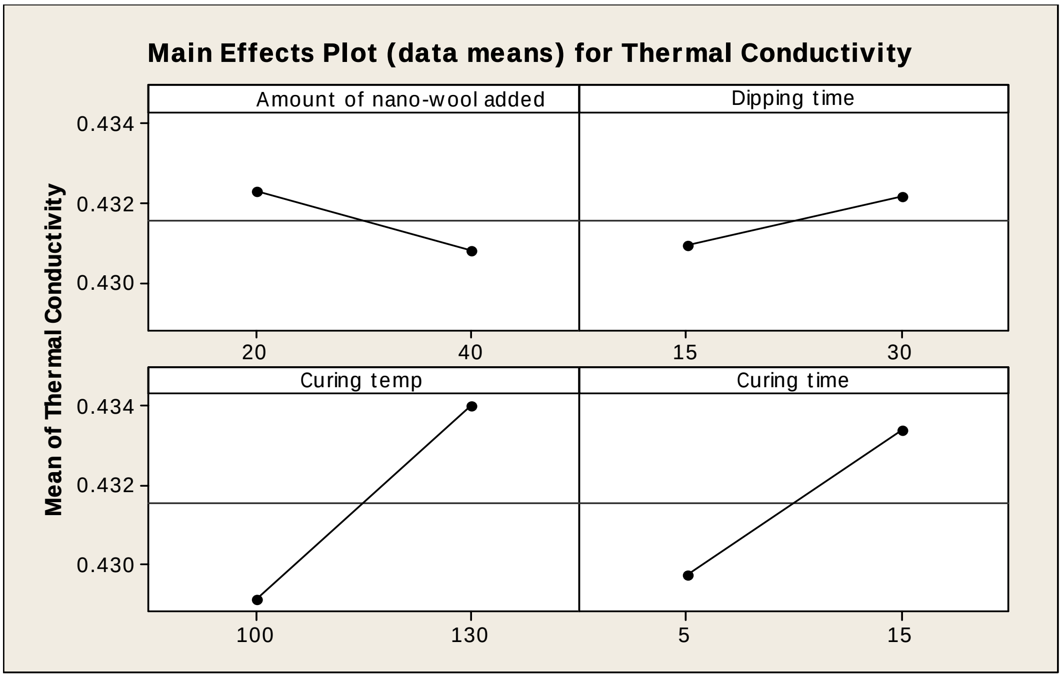

**Figure 5.8** Main effects plot for thermal conductivity of nano-wool treated polyester fabrics

### 5.3.3.2. Keeping Warmth Ratio

Figure 5.9 illustrates that the keeping warmth ratio of polyester fabrics was reduced by the nano-wool treatment similar to cotton fibres. Unlike cotton fibres that swelling occurred after the treatment, the presence of nano-wool particles on polyester fibre surface and the absorption of water molecules by nano-wool particles might also cause the reduction in inter-fibre spacing, resulting in smaller amount of air being trapped between the fibres. Owing to the good heat insulating properties of air, this small amount of trapper air might cause the reduction in keeping the warmth ability of nano-wool treated polyester fabrics.

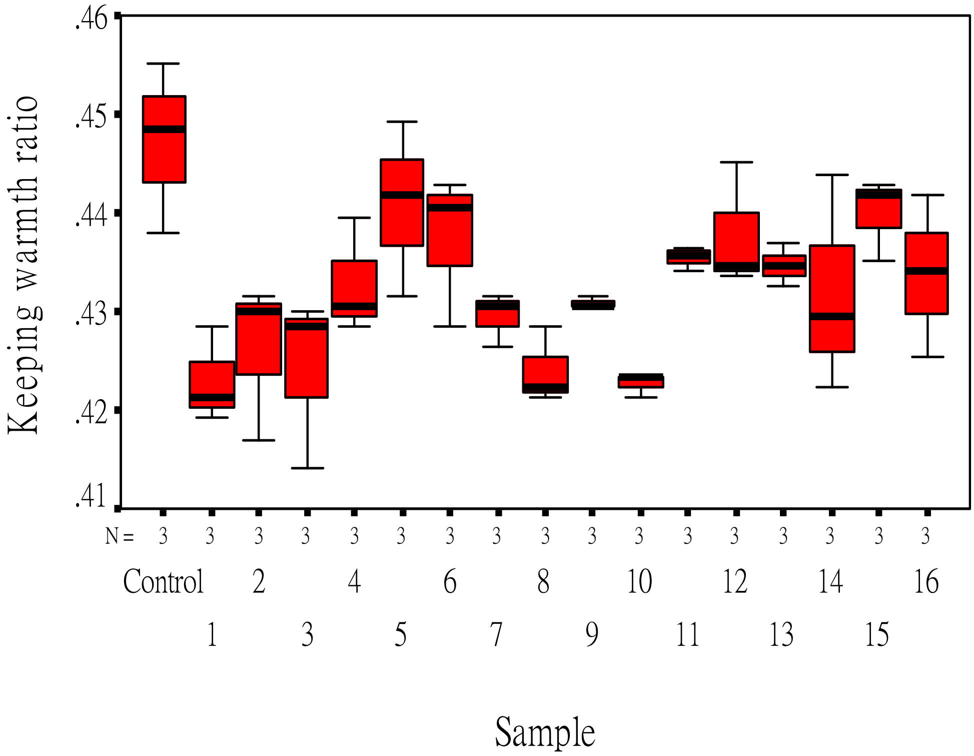

**Figure 5.9** Keeping warmth ratio of nano-wool treated polyester fabrics

The Pareto chart in response to the keeping warmth ratio as demonstrated in Figure 5.10 illustrated that the curing time and curing temperature had significant effect on the keeping warmth ratio of the nano-wool treated polyester fabric. When compared, the effect of curing temperature was more significant than curing time.

The main effects plot shown in Figure 5.11 revealed that the keeping warmth ratio of the nano-wool treated polyester fabrics was enhanced with respect to the increase in the amount of nano-wool added, elevated curing temperature, prolonged curing and dipping times.

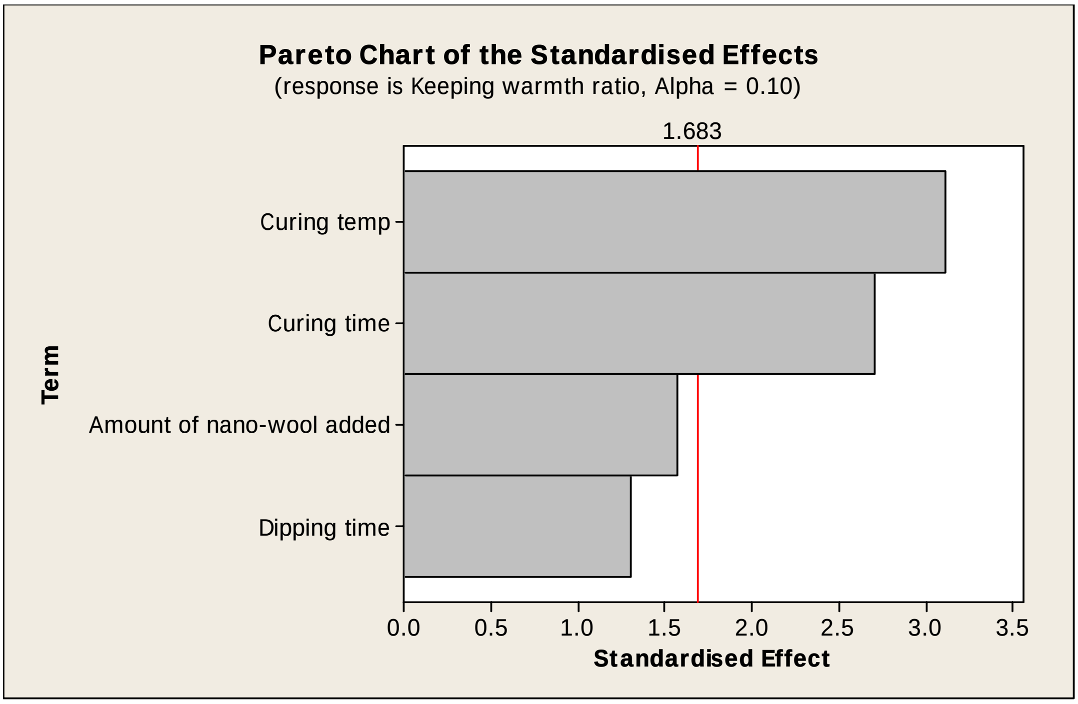

**Figure 5.10** Pareto chart of the keeping warmth ratio of nano-wool treated polyester fabrics

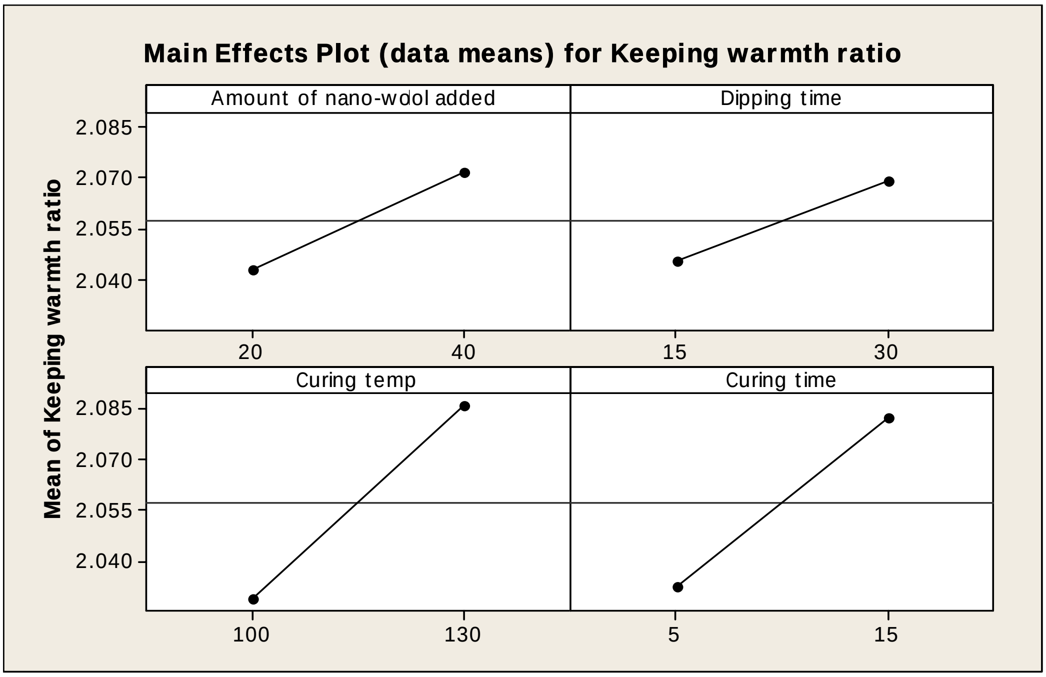

**Figure 5.11** Main effects plot for keeping warmth ratio of nano-wool treated polyester fabrics

### 5.3.3.3. Q-Max Value

Q-max value represents the ability of fabrics to provide cool feeling when touching the fabrics. The higher the q-max value, the cooler the feeling will be when touching the fabrics. Figure 5.12 reveals that the q-max value of the polyester fabrics was lowered by the nano-wool treatment, i.e. the feeling was not as cool as that of the untreated polyester fabrics when touching the treated polyester fabrics. This might be due to the fact that the thermal conductivity of the treated polyester fabrics was lower than that of the untreated polyester fabrics. Hence, the heat generated from our body could not be conducted so easily as that of the untreated one when touching the treated polyester fabrics. As a result, the heat trapped by the treated polyester fabrics provided us with the warmer feeling than that of the untreated one.

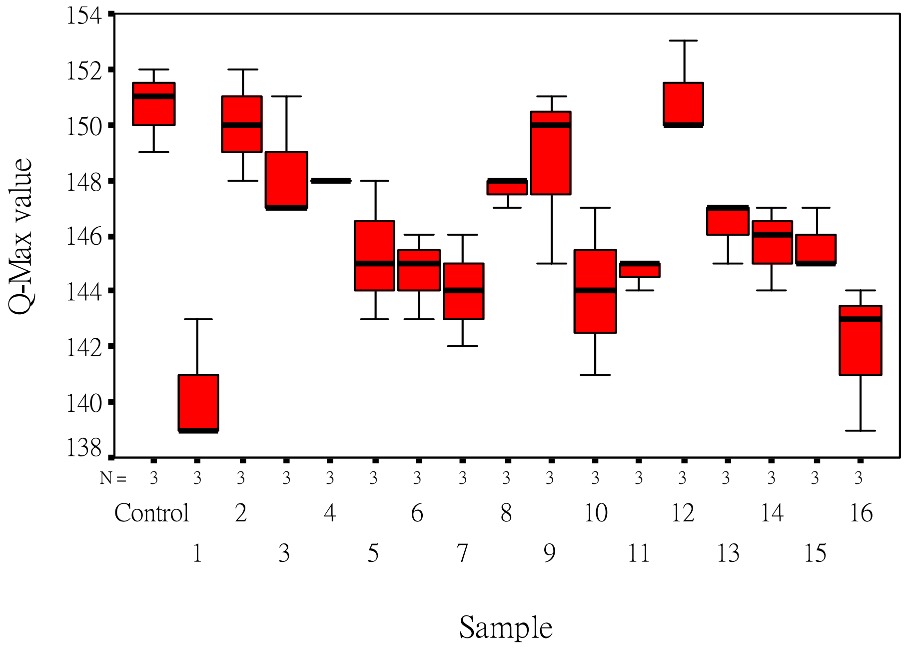

**Figure 5.12** Q-max value of nano-wool treated polyester fabrics

The Pareto chart demonstrated in Figure 5.13 illustrated that the curing temperature was the only factor affecting the q-max value of the nano-wool treated polyester fabrics significantly. The main effects plot shown in Figure 5.14 revealed that the q-max value of the nano-wool treated polyester fabrics was enhanced with respect to the increase in the amount of nano-wool added, prolonged dipping time and lower curing temperature. However, the q-max value of the treated polyester fabrics kept almost constant with respect to the prolonged curing time.

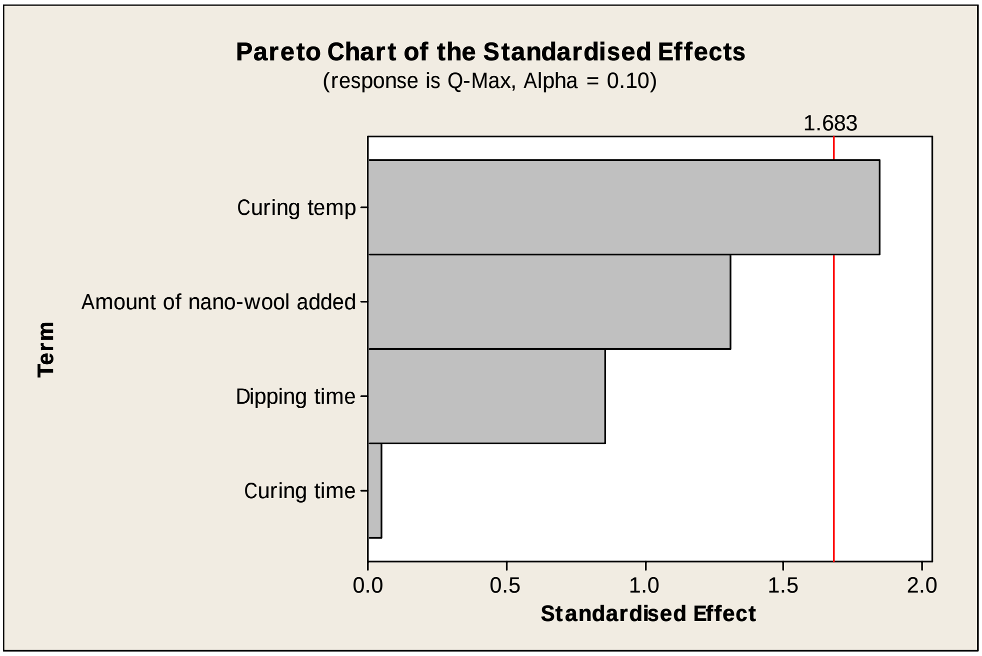

**Figure 5.13** Pareto chart of the q-max value of nano-wool treated polyester fabrics

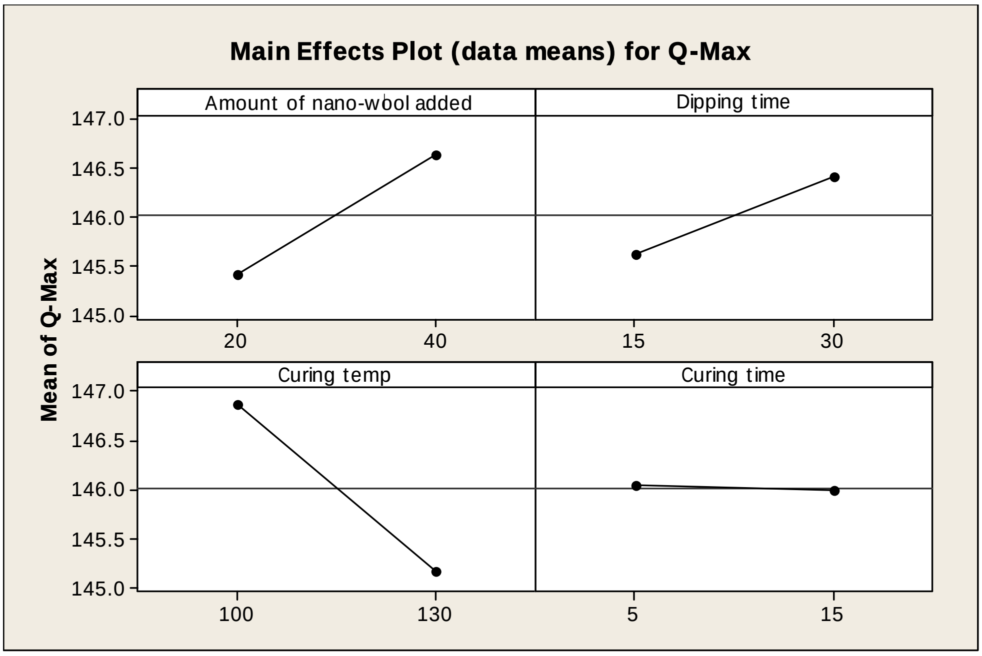

**Figure 5.14** Main effects plot for q-max value of nano-wool treated polyester fabrics

### 5.3.4. Liquid Water Transfer Properties

The Accumulative One-Way Transport Capacity (OWTC) and Overall Moisture Management Capacity (OMMC) of nano-wool treated polyester fabrics were tested by the moisture management tester. The larger the OMMC and OWTC values, the higher the overall moisture management capability and hydrophilicity of the fabric will be.

Figures 5.15 and 5.16 respectively show that the nano-wool treated polyester fabrics had higher OWTC and OMMC than those of the untreated polyester fabrics. This meant that polyester became more hydrophilic and had better moisture management capability after the treatment.

The experimental result illustrated in Chapter 3 showed that when wool was pulverised into nano-scale, the nano-wool particles exhibited greater amorphous region and hydrophilic hydroxyl (OH) group. There was the possibility that the enhancement in amorphous region and hydroxyl (OH) group of nano-wool particles might induce higher hydrophilic ability than wool fibres. Hence, when these kind of hydrophilic nano-wool particles were applied onto the polyester fabrics, they might act like the small capillary adhering to both the surface and internal part of polyester fibre (Adler and Walsh, 1984; Gu L, 1989). It was most likely that the nano-wool particles could move inside the polyester fibres to absorb more water molecules. By increasing the moisture content of polyester fibres, the capillary wicking would occur resulting in the enhancement of the hydrophilic properties of polyester fibres. Hence, the OWTC, OMMC and moisture management capability of nano-wool treated polyester fabrics were enhanced considerably.

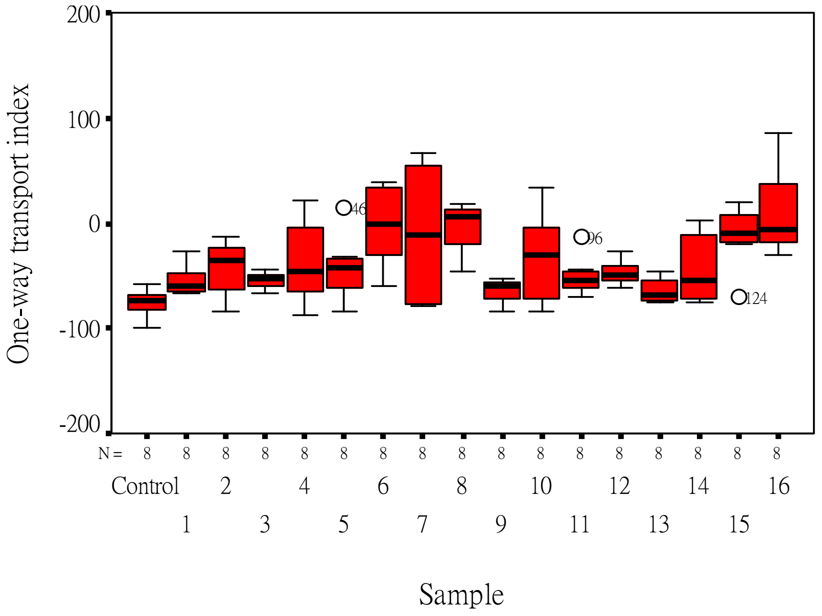

**Figure 5.15** OWTC of nano-wool treated polyester fabrics

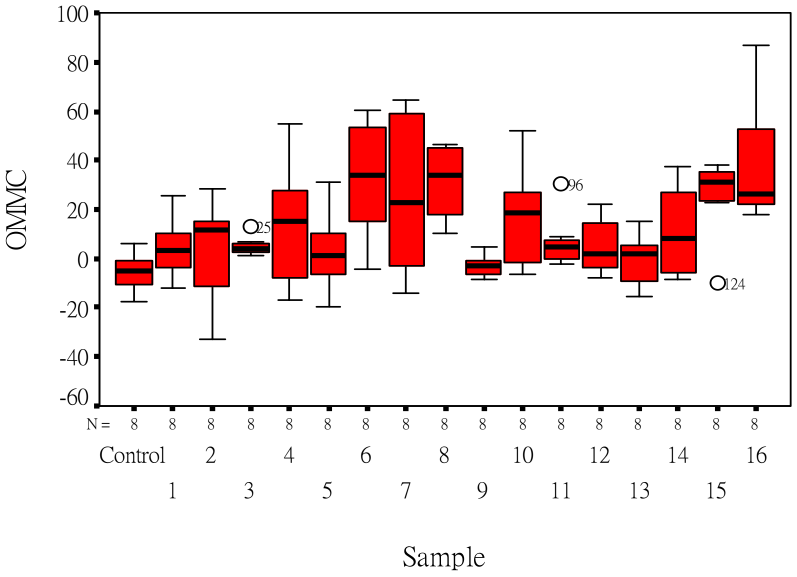

**Figure 5.16** OMMC of nano-wool treated polyester fabrics

The Pareto charts shown in Figures 5.17 and 5.18 illustrated that the OWTC and OMMC of the nano-wool treated polyester fabric were most significantly affected by the amount of nano-wool added followed by the curing temperature. The main effects plots shown in Figures 5.19 and 5.20 illustrated that the dipping time and curing time did not cause significant effect on both OWTC and OMMC of the nano-wool treated polyester fabric. The OWTC and OMMC of the nano-wool treated polyester fabric, however, were enhanced greatly with respect to the reducing amount of nano-wool added and elevated curing temperature.

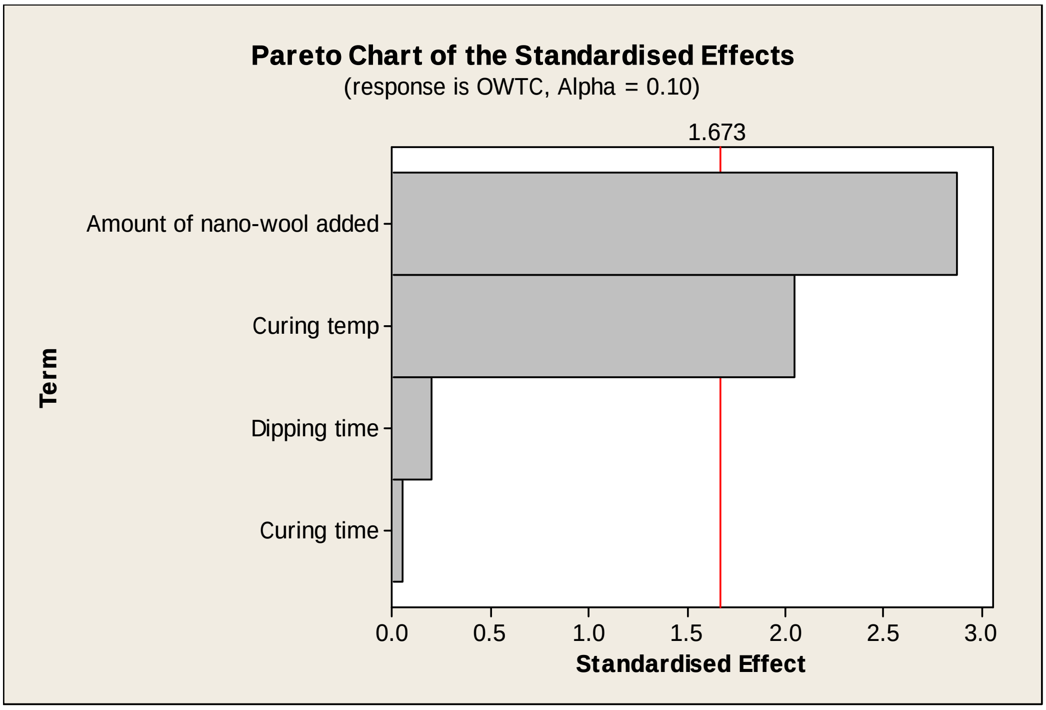

**Figure 5.17** Pareto chart of the OTWC of nano-wool treated polyester fabrics

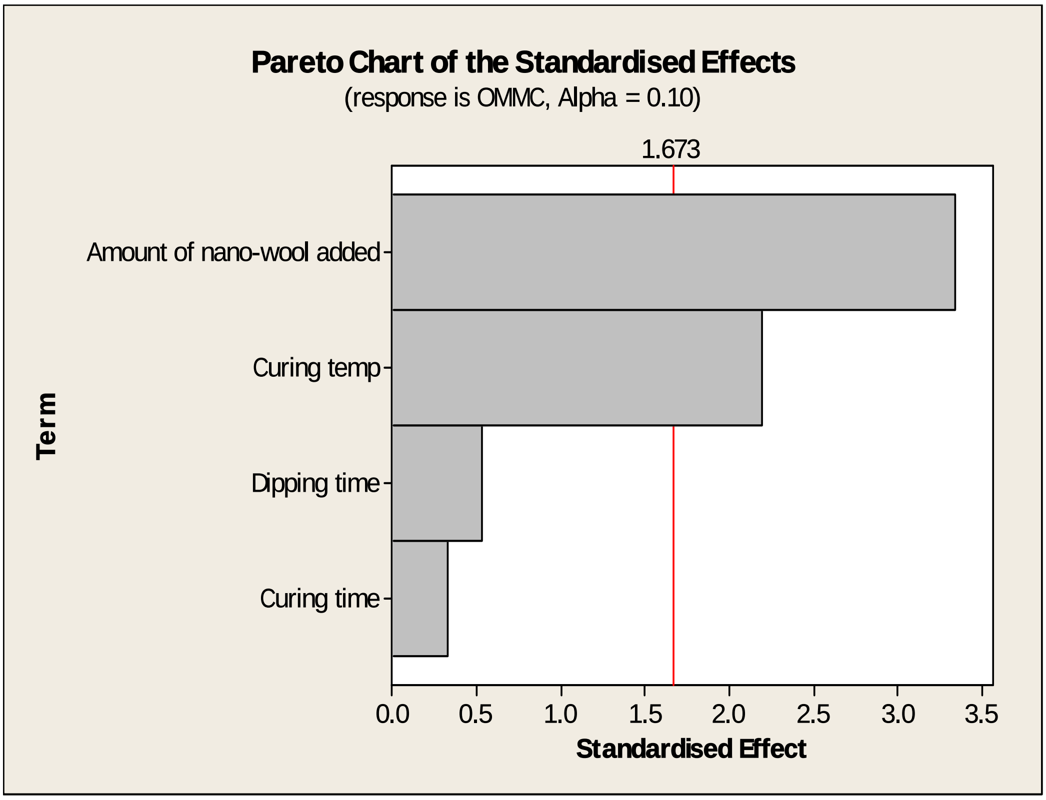

**Figure 5.18** Pareto chart of the OMMC of nano-wool treated polyester fabrics

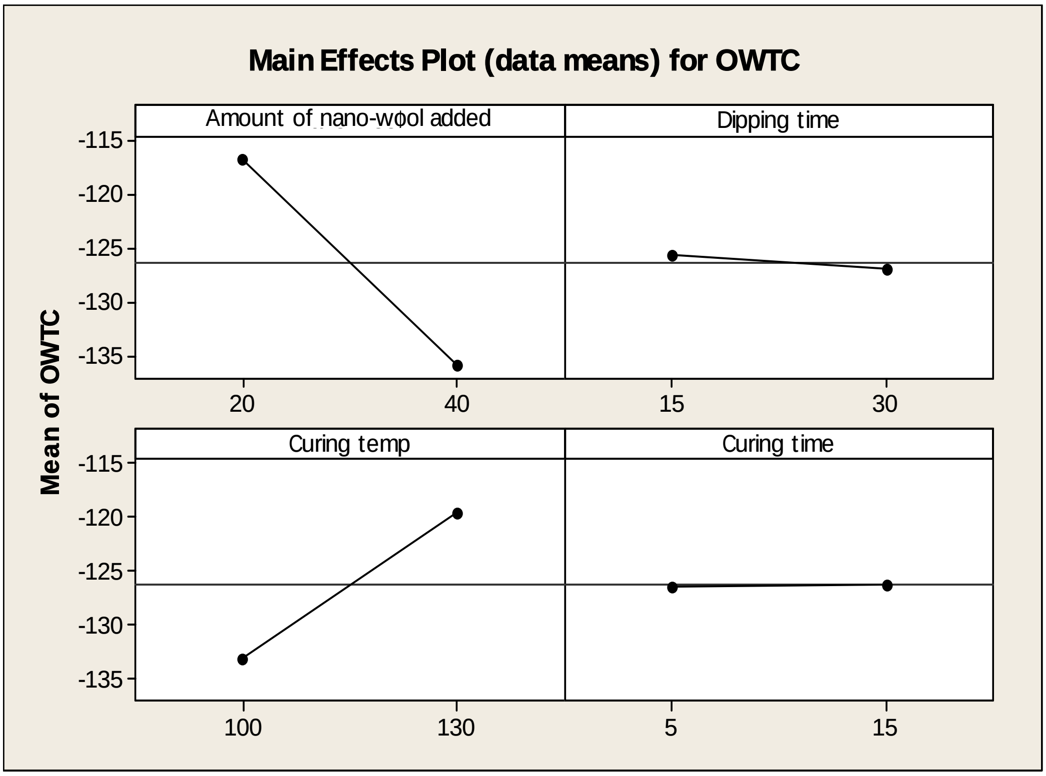

**Figure 5.19** Main effects plot for OWTC of nano-wool treated polyester fabrics

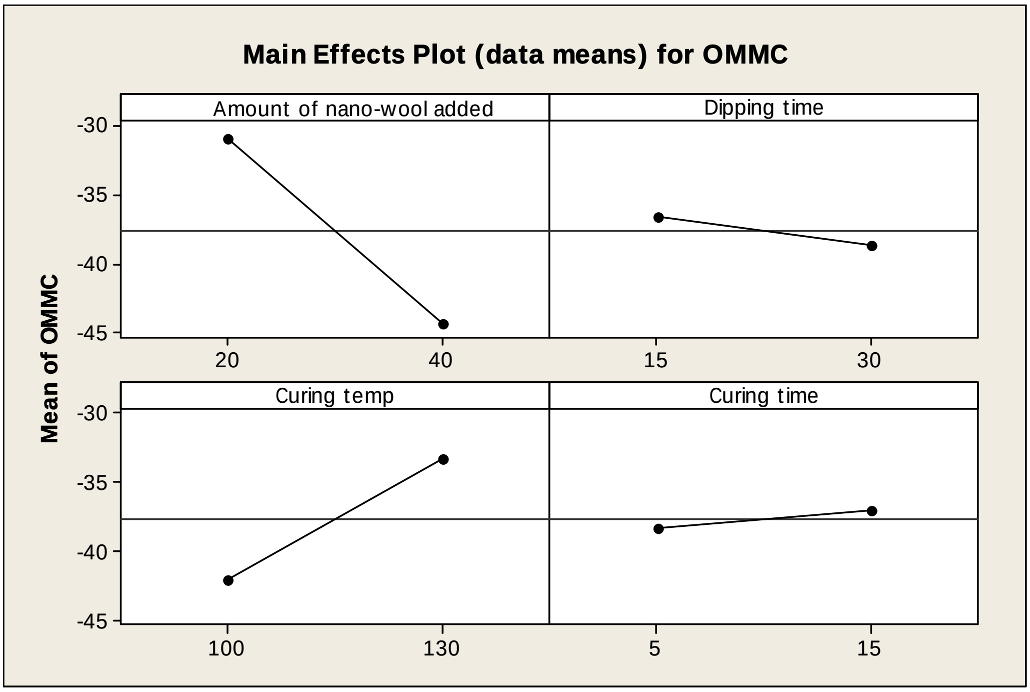

**Figure 5.20** Main effects plot for OMMC of nano-wool treated polyester fabrics

## 5.4. Summary

In this thesis, the pad-cry-cure method was adopted to apply nano-wool particles onto polyester fabrics. The morphology, thermal properties, liquid water transport properties and air permeability of the nano-wool treated polyester fabrics were thoroughly studied.

It was found that the nano-wool treatment could cause the reduction in air permeability, thermal conductivity, keeping warmth ability, q-max of polyesters fabrics. On the other hand, it could enhance the OWTC and OMMC values, moisture management capability, and hydrophility of polyester fabrics.

In this research, the fractional factorial design generated by means of the Minitab

Release 14 was used to identify some variables that affected the properties of the nano-wool treated polyester fabrics. These variables included (i) the amount of nano-wool added, (ii) dipping time, (iii) curing time and (iv) curing temperature.

Table 5.1 illustrates the summary of the effect of different parameters on the air permeability, hydrophobicity, thermal conductivity, keeping warmth ratio and q-max vale of nano-wool treated fabrics. The one with double arrows marking represents the most significant parameters that affect the properties of the polyester fabric.

Table 5.1 shows that the increase in the amount of nano-wool added would result in the enhancement of air permeability, hydrophobicity, keeping warmth ratio and q-max value of the treated fabrics. However, it would reduce the thermal conductivity of the nano-wool treated polyester fabrics.

Table 5.1 also demonstrates that the prolonged dipping time could cause the enhancement in all the properties of the nano-wool treated polyester fabrics. Moreover, the air permeability, thermal conductivity and keeping warmth ratio of the nano-wool treated polyester fabric were also enhanced by prolonging the curing time. Furthermore, the results showed that higher curing temperature would cause a decrease in air permeability, hydrophobicity and q-max of the nano-wool treated polyester fabrics, but an increase in thermal conductivity and keeping warmth ratio of the treated polyester fabrics.

In summary, a variety of properties as well as the performance of the nano-wool treated polyester fabrics were greatly affected by (i) the amount of nano-wool added, (ii) dipping time, (iii) curing temperature and (iv) curing time of the treatment in different ways.

**Table 5.1.** Comparison of the effect of different parameters on the properties of nano-wool treated polyester fabrics

| | Air permeability | Hydrophobicity | Thermal conductivity | Keeping warmth ratio | Q-max value |
|---|---|---|---|---|---|
| Increase in amount of nano-wool added | ↑ | ↑ ↑ | ↓ | ↑ | ↑ |
| Increase in dipping time | ↑ | ↑ | ↑ | ↑ | ↑ |
| Increase in curing time | ↑ | - | ↑ ↑ | ↑ ↑ | - |
| Increase in curing temperature | ↓ | ↓ ↓ | ↑ ↑ | ↑ ↑ | ↓ ↓ |
| Remark: The double marking of arrows represents the most significant factor that affects the properties of the nano-wool treated polyester fabrics. | | | | | |

# Chapter 6 Modification of Dyeing Properties of Fabric by Nano-Wool Particles

## 6.1 Introduction

In recent years, nanotechnology has been employed in textile finishing process for modifying the functions and properties of the fabrics such as wrinkle resistant and ultraviolet protection etc. However, the use of nanotechnology in dyeing process is rare and limited. In addition, no attempt has ever been made on the application of nano-scale fibre particles in the dyeing process. In fact, the use of nanotechnology in the dyeing process will benefit the textile industry greatly due to the unique properties of nano-materials. In this thesis, the application of nanotechnology in textile dyeing process is explored by means of nano-wool particles. It is found that the nano-wool particles did affect the dyeing properties of cotton and polyester fabrics tremendously.

## 6.2. Methodology

In order to study the dyeing performance of the nano-wool treated fabric, both the untreated and nano-wool treated cotton and polyester fabrics were dyed in red, blue and yellow colours using reactive dyes and disperse dyes respectively. Four main steps involved in the experiment are shown in the following:

Step 1: Pulverisation of wool fibre into nano-wool particles.

Step 2: Preparation of untreated and nano-wool treated cotton and polyester fabrics.

Step 3: Dyeing of the nano-wool treated and untreated cotton and polyester fabrics in red, yellow and blue colours using reactive dyes and disperse dyes respectively.

Step 4: Evaluation of the colour of the nano-wool treated and untreated dyed cotton and polyester fabrics with the aid of spectrophotometer.

### 6.2.1. Pulverisation of Wool Fibre into Nano-Wool Particles

The nano-wool particles were prepared according to the three pulverisation processes mentioned in Section 3.2.1 of Chapter 3. The rotate crusher, ultrasonic crusher, ultra-turrax® and nano-colliding machine were involved in the three pulverisation processes. 20g First Pulverised Wool Particles were pulverised in 800 ml deionised water for the formation of the nano-wool suspension. In the dyeing experiment, 3 pieces of cotton fabrics and 3 pieces of polyester fabrics were dyed respectively in red, yellow and blue colours. The pulverisation processes were repeated twice in order to prepare two separate 800ml nano-wool suspensions for treating the cotton and polyester fabrics separately.

### 6.2.2. Preparation of Nano-Wool Treated Fabric

The nano-wool suspension was prepared by adding the acrylic based Ciba Alcoprint PBA binder to the suspension according to Section 3.2.1 of Chapter 3 at the liquor ratio (volume/volume) of 1:100 correspondingly.

In the experiment, 3 pieces of 2g cotton fabrics and 3 pieces of 2g polyester fabrics were treated with nano-wool suspension separately. The cotton and polyester fabrics were placed separately into 800ml of the pulverised nano-wool suspension for 10 minutes and then padded 5 times with the suspension. Finally, the fabrics were dried at 100℃ for 10 minutes and then cured in an oven at 150℃ for 5 minutes.

### 6.2.3. Dyeing of Fabrics

All the untreated fabrics and nano-wool treated fabrics were dyed separately for colour evaluation. Reactive dyes and disperse dyes were used to dye the cotton and polyester fabrics respectively.

#### 6.2.3.1. Dyeing of Cotton Fabrics with Reactive Dye

Both the untreated cotton fabric and nano-wool treated cotton fabrics were dyed in red, blue and yellow colours by using the reactive dye of Procoin Red H-E3B, Procoin Yellow H-E4G and Procoin Navy H-ER 150 respectively. Totally, 3 pieces of 2g untreated cotton fabrics and 3 pieces of 2g nano-wool treated cotton fabrics were dyed separately. Each cotton fabric sample was dyed in 100 ml of dye solution containing 2% dyestuff and 1g/l leveling agent of Matexil PA-L at the liquor ratio of 50: 1. The temperature of dyebath was raised from room temperature to 60℃. During the dyeing process, 40g/l of Glauber's salt (sodium sulphate) and 10g/l soda ash (sodium carbonate) were added to the dye solution at 60℃ according to the dyeing curves shown in Figure 6.1.

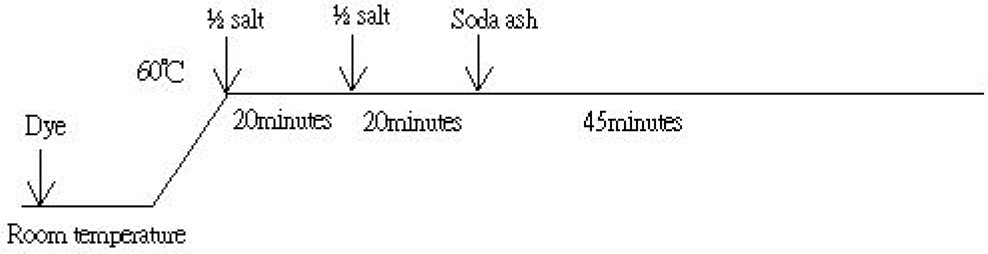

**Figure 6.1** Dyeing curve of reactive dye for cotton fabrics

After dyeing, the dyed cotton fabric sample was rinsed with deionised water and then placed in the soaping solution composing of 1g/l non-ionic detergent of Lissapol NP at 60℃ for 10 minutes. After soaping, the cotton fabric sample was rinsed with deionised water, centrifuged, dried and then conditioned before colour measurement.

#### 6.2.3.2. Dyeing of Polyester Fabrics with Disperse Dye

3 pieces of 2g untreated polyester fabrics and 3 pieces of 2g nano-wool treated polyester fabrics were dyed in red, blue and yellow colours by using the disperse dye of Polycron Red 3B, Polycron Yellow 5GC and Polycron Navy Blue GLP respectively.

The polyester fabric samples were first wetted with hot water at about 80℃ for 10 minutes and then centrifuged with hydroextractor for 1 minute before dyeing. Each polyester fabric sample was dyed in 100 ml of dye solution at the liquor ratio of 50: 1 inside the Ahiba, Roaches & Mathis IR dyeing machines according to the dyeing curve shown in Figure 6.2. In each dyebath, 3% dye, 1g/l dispersing agent of Sandozol KB and 0.5g/l acetic acid were added.

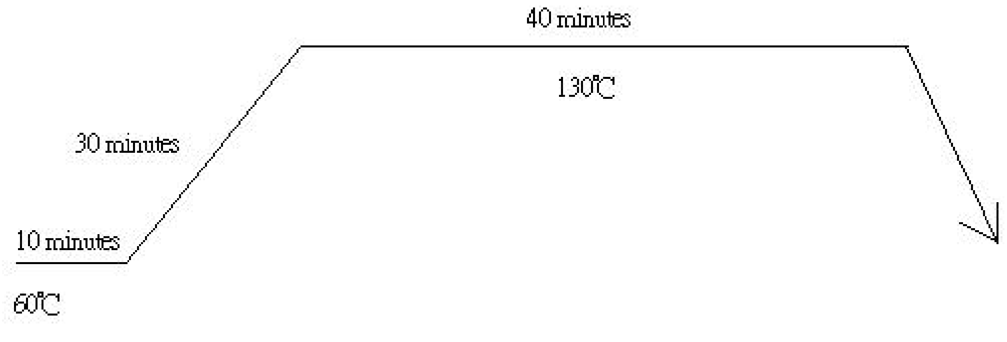

**Figure 6.2** Dyeing curve of disperse dye for polyester fabrics

When the dyeing process was completed, the temperature of the dyeing machine was cooled to 60℃ and the dyed polyester fabric samples were rinsed with deionised water. The dyed polyester fabric samples were then treated separately with 100ml of reduction clearing solution containing 2ml/l sodium hydroxide, 2g/l sodium hydrosulphite and 2g/l dispersing agent of Sandozol KB at 60℃ for 15 minutes. The polyester fabrics were finally rinsed with deionised water, centrifuged, dried and conditioned before colour measurement.

## 6.2.4. Colour Evaluation

After dyeing, the dyed fabric samples of cotton and polyester were conditioned according to the ASTM D1776-95 for 24 hours before colour measurement. The colour of the dyed fabric samples was evaluated with the aid of DataColour spectrophotometer version 450 according to the ASTM D3134 standard. The colour difference ($\Delta E$) between the untreated fabric sample and nano-wool treated fabric sample was elaborated using CIE Lab* formulation.

## 6.3 Result and Discussion

### 6.3.1. Comparison of the Colour of Untreated Cotton and Nano-Wool Treated Cotton Fabrics

The comparison of the reflectance values of the untreated dyed cotton and nano-wool treated dyed cotton fabric samples in yellow, red and blue colours is shown in Figures 6.3 to 6.5 respectively. These figures demonstrate that the reflectance values of the dyed cotton fabric samples were reduced after the nano-wool treatment, i.e. the reflection of light was lowered by the nano-wool particles.

In order to compare the absorption of dyestuffs by the untreated dyed cotton and nano-wool treated dyed cotton fabrics, the K/S curves of the dyed fabric sample in yellow, red and blue colours were plotted and illustrated in Figures 6.6 to 6.8 respectively. The K/S value was developed by Kubelka and Munk from the Kubelka-Munk theory in 1939. The Kubelka-Munk theory is generally used for the analysis of diffuse reflectance spectra obtained from the weakly absorbing samples. It provides a correlation between reflectance and concentration. The concentration of absorbed dyestuff can be determined using the Kubelka Munk formula (chm2web software, 2007):

$$F(R) = (1 - R)^2 / (2 \times R) = K / S = A \times C / S$$

where, R = reflectance; K = absorption coefficient; S = scattering coefficient; C = concentration of absorbing species; A = absorbance.

The equation shows that the K/S value increases with respect to the higher concentration of the absorbing species. When compared with the untreated cotton fabrics, the nano-wool treated cotton fabrics usually have higher K/S value, implying that the concentration of the dyestuff absorbed by the cotton fabrics was enhanced by the nano-wool particles.

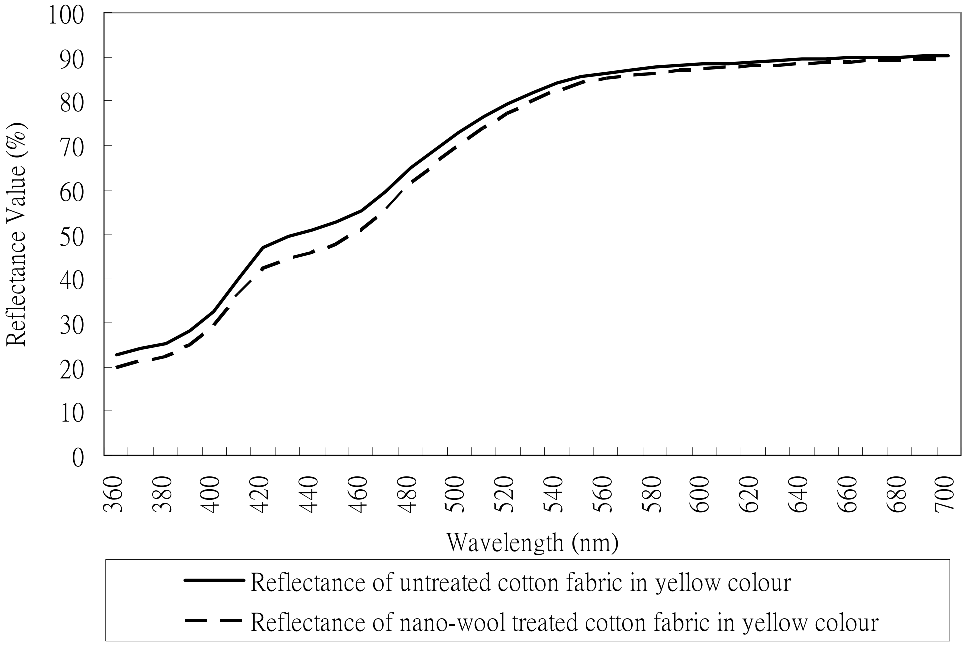

**Figure 6.3** Comparison of reflectance value of the untreated cotton and nano-wool treated cotton fabrics in yellow colour

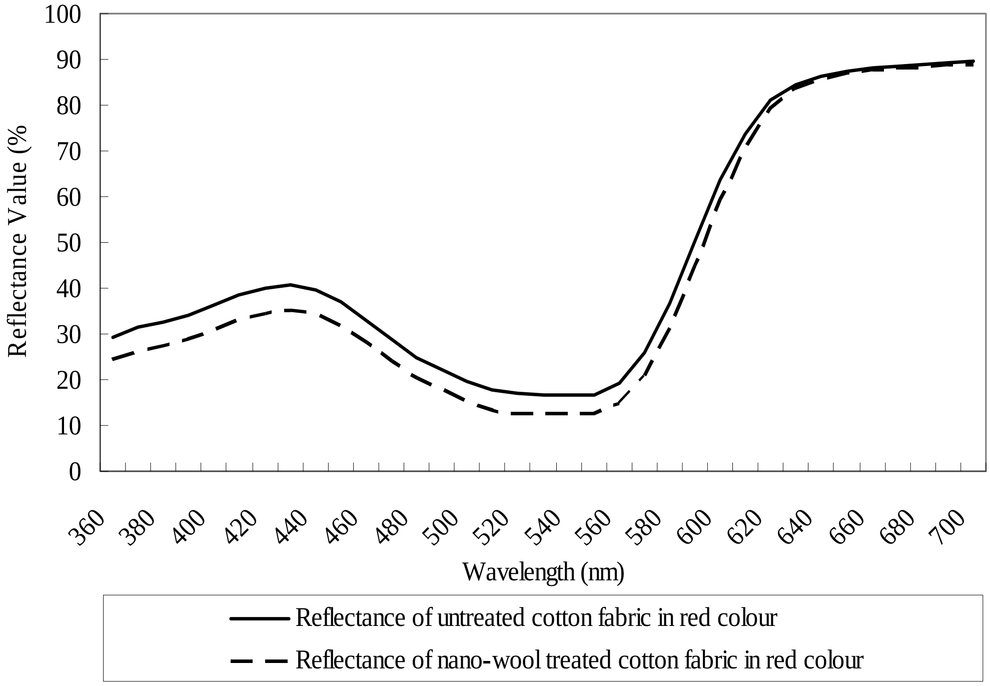

**Figure 6.4** Comparison of reflectance value of the untreated cotton and nano-wool treated cotton fabrics in red colour

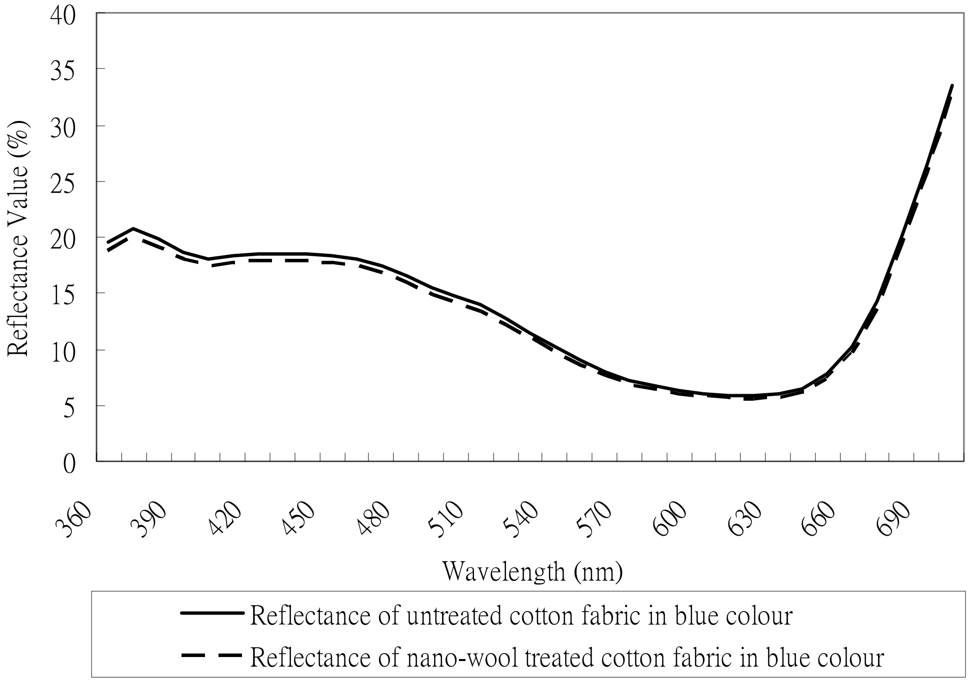

**Figure 6.5** Comparison of reflectance value of the untreated cotton and nano-wool treated cotton fabrics in blue colour

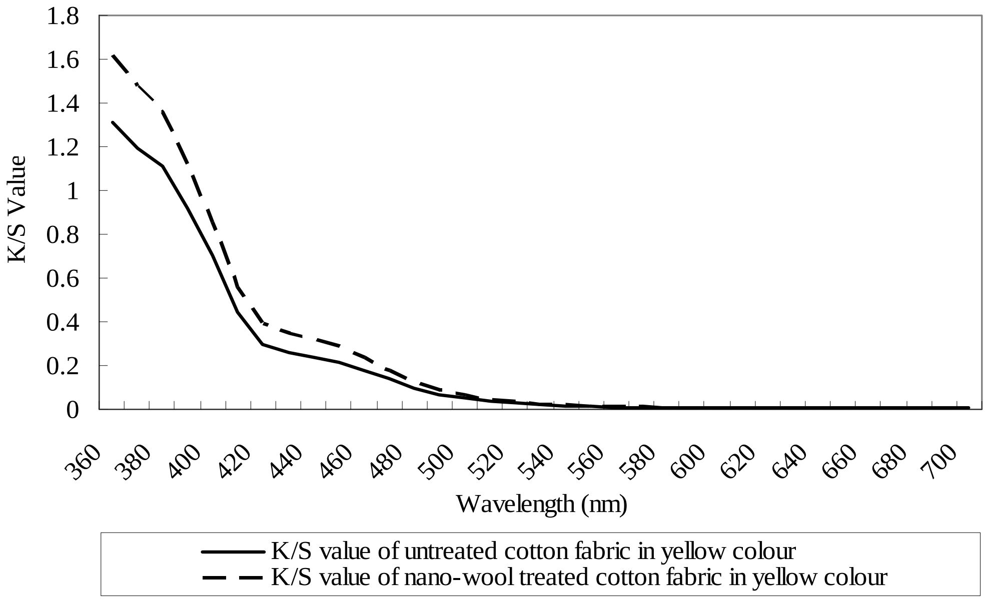

**Figure 6.6** Comparison of K/S value of the untreated cotton and nano-wool treated cotton fabrics in yellow colour

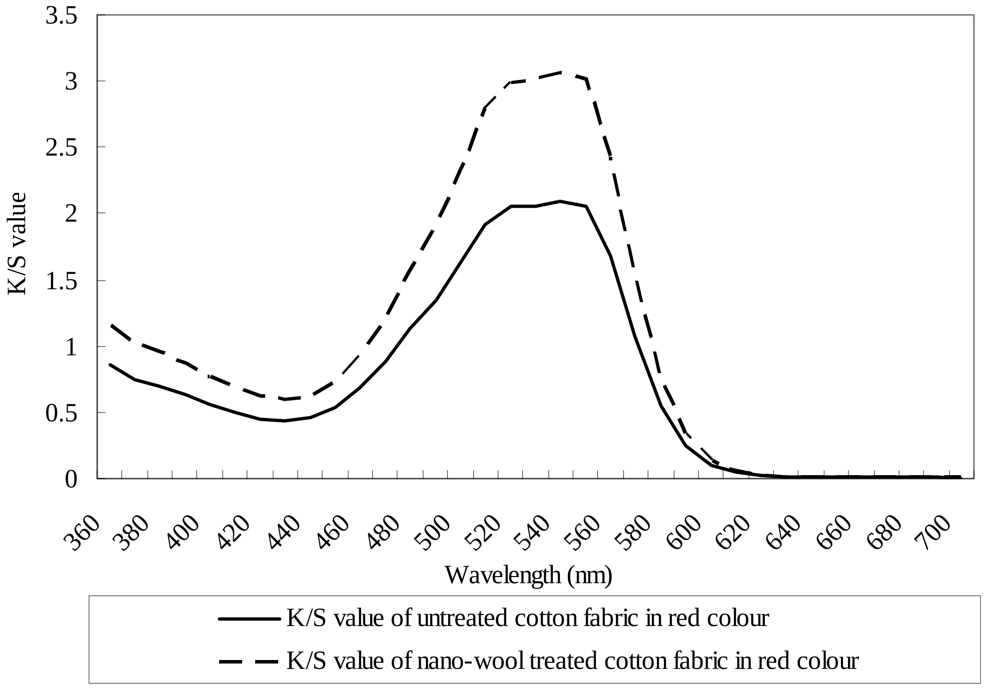

**Figure 6.7** Comparison of K/S value of the untreated cotton and nano-wool treated cotton fabrics in red colour

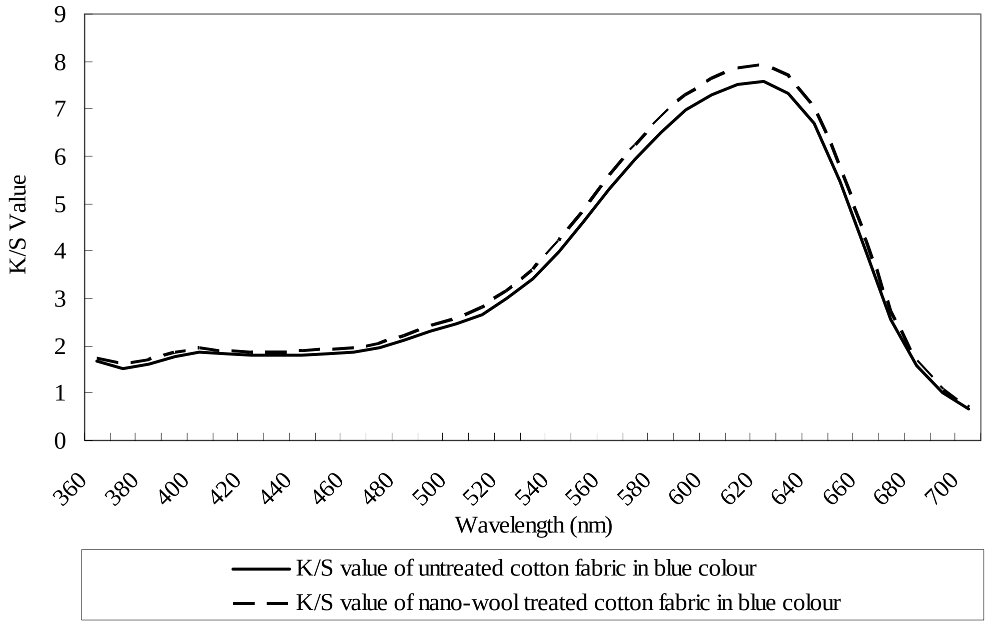

**Figure 6.8** Comparison of K/S value of the untreated cotton and nano-wool treated cotton fabrics in blue colour

Besides the reflectance value and K/S value, the colour difference between the untreated cotton and nano-wool treated cotton fabric in yellow, red and blue colours under D65 and A light source was compared by using the CIELab* colour system as shown in Table 6.1.

**Table 6.1** Colour difference of the untreated cotton and nano-wool treated cotton fabrics dyed with different reactive dyes

| **Colour** | **Light source** | **CIEΔE*** | **CIE ΔL*** | **CIE Δa*** | **CIE Δb*** | **CIE ΔC*** | **CIE ΔH*** | **Remarks** |
|---|---|---|---|---|---|---|---|---|
| Yellow | D65/10 Degree | 3.362 | -0.784 | -0.152 | 3.265 | 3.265 | -0.152 | Darker / Greener / Yellow |
| | A/10 Degree | 3.344 | -0.632 | 0.391 | 3.261 | 3.284 | -0.03 | Darker / Redder / Yellow |
| Red | D65/10 Degree | 5.788 | -3.615 | 4.398 | 1.045 | 4.354 | 1.214 | Darker/ Redder/ Less Blue |
| | A/10 Degree | 5.519 | -2.954 | 4.045 | 2.316 | 4.453 | 1.377 | Darker / Redder / Yellow |
| Blue | D65/10 Degree | 0.769 | -0.742 | 0.203 | -0.001 | -0.044 | 0.198 | Darker / Less Green |
| | A/10 Degree | 0.744 | -0.725 | 0.163 | 0.036 | -0.091 | 0.139 | Darker / Less Green |

Table 6.1 illustrates that there was colour difference between the untreated cotton and nano-wool treated cotton fabrics dyed with different colours. The colour difference between the untreated cotton and nano-wool treated fabric in blue colour was not visually detectable, i.e. CIE ΔE *<1, while the colour difference of yellow and red colours was obviously noticeable, i.e. CIE ΔE *>1. Table 6.1 also shows that the nano-wool treated cotton fabrics in red, yellow and blue colours were darker than those of the untreated cotton fabrics as reflected by the negative CIE ΔL* values. In the case of yellow colour, the nano-wool treated cotton fabrics looked more yellowier than that of the untreated cotton fabrics, i.e. CIE Δb* value was

positive. As for the red colour, the reddish shade of the cotton fabrics was also enhanced after the nano-wool treatment, i.e. CIE $\Delta a^*$ value was positive.

The colour difference between the untreated cotton and nano-wool treated cotton fabrics was mainly due to greater uptake of dyestuff by the cotton fabrics after the nano-wool treatment. As mentioned in Section 4.3.1 of Chapter 4, more water molecules were absorbed by the nano-wool particles present inside the cotton fibres after the nano-wool treatment. The water molecules so absorbed would position themselves between the cellulosic chains. It was suggested that both water molecules and nano-wool particles present between the cellulosic chains of cotton could reduce the bonding forces of the fibres, making the fibre more flexible and generally inducing swelling (Wallenberger 1978). Since the dissolved dyestuff could easily diffuse into the fibre through water medium, more water molecules present inside the fibre implied that more dissolved dyestuffs were also present inside the fibre. In addition, the swelling of cotton fibres could increase the size of amorphous region and inter-fibre spacing of the fibres for the absorption of dyestuff. As a result, the dye uptake of the cotton fibres was enhanced by the nano-wool treatment, leading to the colour difference between the untreated cotton and nano-wool treated cotton fabrics.

### 6.3.2. Comparison of the Colour of Untreated Polyester and Nano-Wool Treated Polyester Fabrics

The comparison results of the reflectance and K/S values of the untreated polyester and nano-wool treated polyester fabric in yellow, red and blue colours are shown in Figures 6.9 to 6.14. Figures 6.9 to 6.11 illustrate that the reflectance values of the untreated polyester fabrics after dyeing were higher than those of the nano-wool treated polyester fabrics, meaning that the nano-wool treatment could lower the light reflection of the dyed fabrics. Figures 6.12 to 6.14 demonstrate that the K/S values of nano-wool treated polyester fabrics after dyeing were higher than those of the untreated polyester fabrics. Since the K/S value increases with higher concentration of dyestuff, higher K/S values of nano-wool treated polyester fabrics implied that the concentration of the dyestuffs absorbed by the polyester fabrics was enhanced by the nano-wool particles when compared with those of the untreated polyester fabrics.

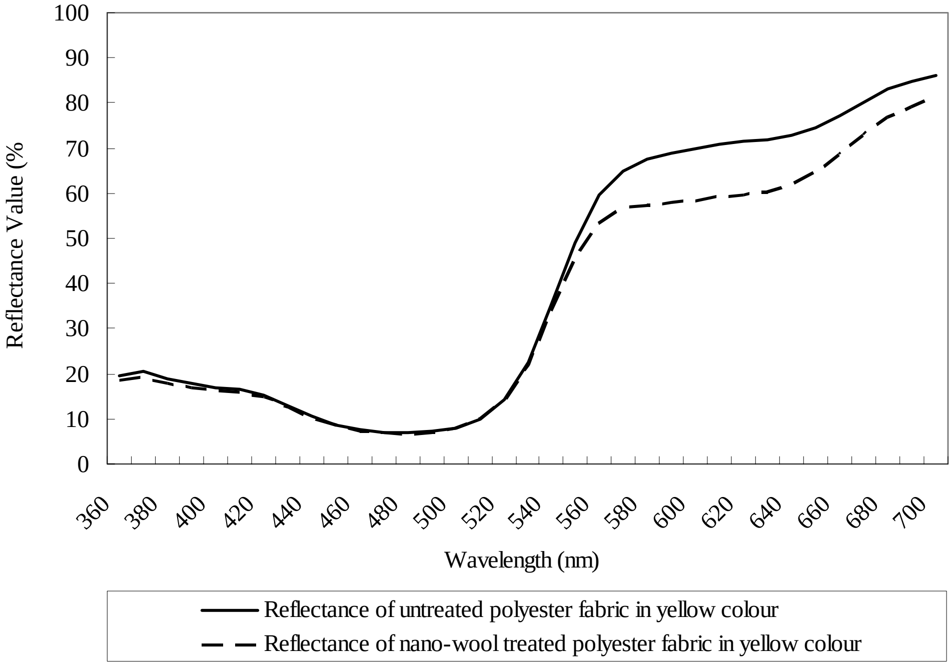

**Figure 6.9** Comparison of reflectance of the untreated polyester and nano-wool treated polyester fabrics in yellow colour

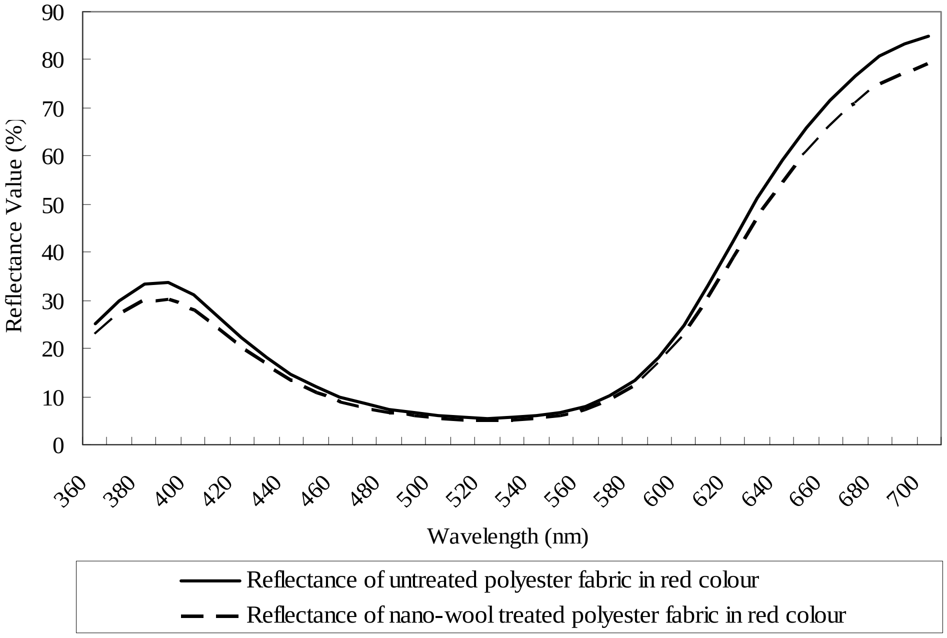

**Figure 6.10** Comparison of reflectance of the untreated polyester and nano-wool treated polyester fabrics in red colour

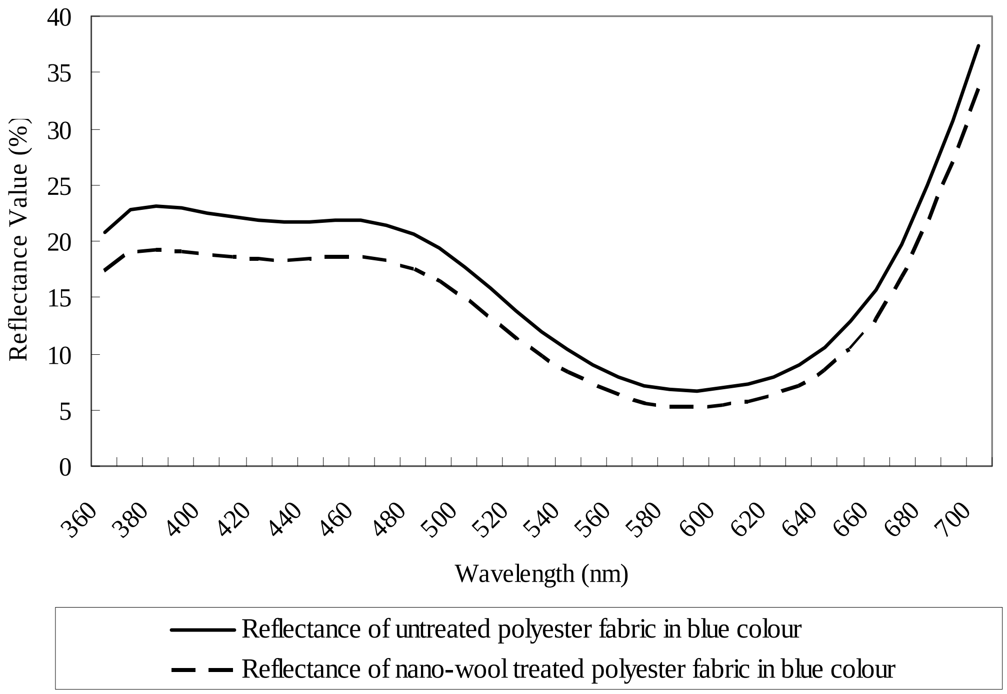

**Figure 6.11** Comparison of reflectance of the untreated polyester and nano-wool treated polyester fabrics in blue colour

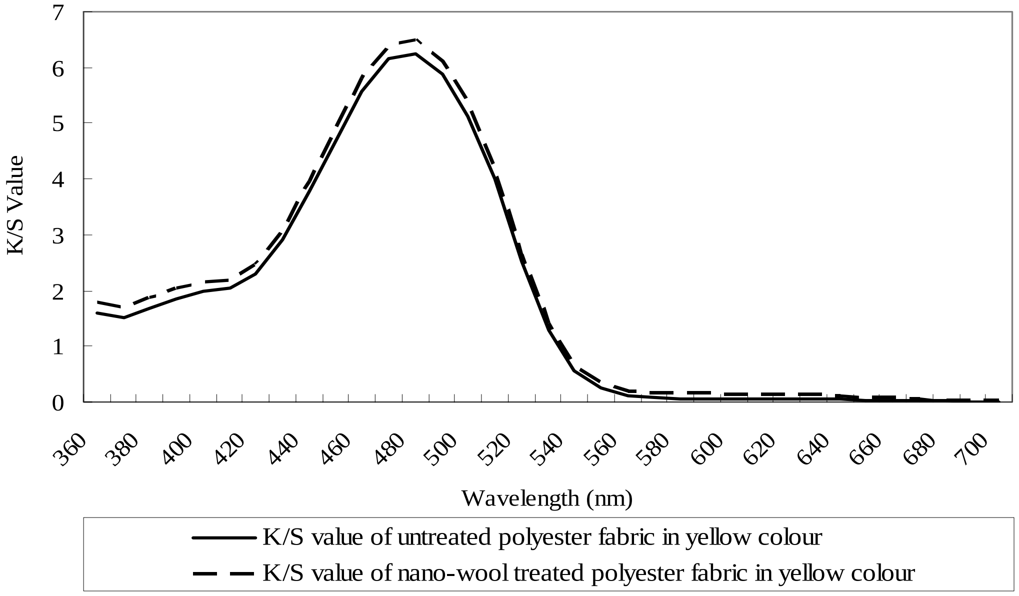

**Figure 6.12** Comparison of K/S value of the untreated polyester and nano-wool treated polyester fabrics in yellow colour

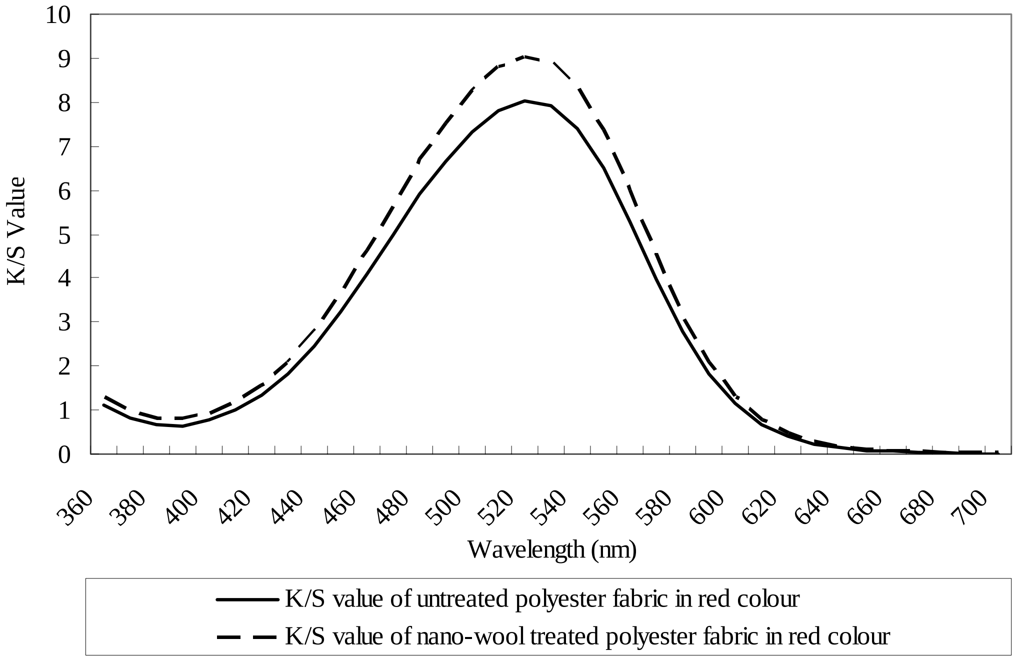

**Figure 6.13** Comparison of K/S value of the untreated polyester and nano-wool treated polyester fabrics in red colour

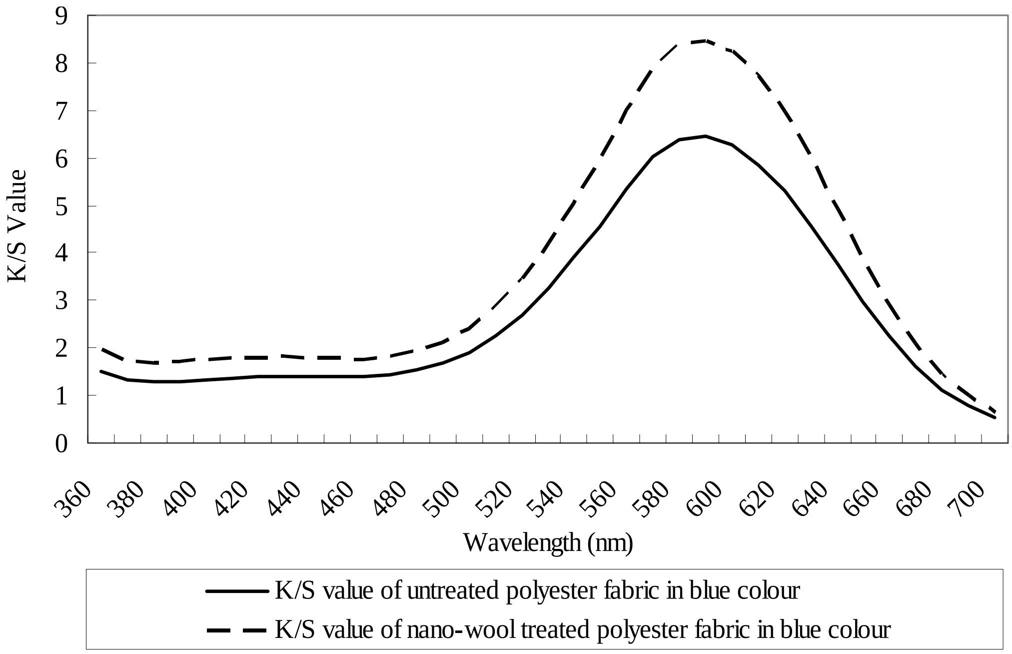

**Figure 6.14** Comparison of K/S value of the untreated polyester and nano-wool treated polyester fabrics in blue colour

Apart from the study of the comparison of reflectance value and K/S value of the untreated polyester and nano-wool treated polyester fabric after dyeing, their colour difference under D65 and A light sources using CIELab* colour system is shown in Table 6.2.

**Table 6.2** Colour difference of the untreated polyester and nano-wool treated polyester fabrics dyed with different disperse dyes

| Colour | Light source | CIE ΔE* | CIE ΔL* | CIE Δa* | CIE Δb* | CIE ΔC* | CIE ΔH* | Remarks |
|---|---|---|---|---|---|---|---|---|
| Yellow | D65/10 Degree | 8.91 | -4.451 | -4.4234 | -6.454 | -7.623 | 1.208 | Darker / Less Red / Less Yellow |
| | A/10 Degree | 9.915 | -5.137 | -3.611 | -7.674 | -8.449 | 0.731 | Darker / Less Red / Less Yellow |
| Red | D65/10 Degree | 5.411 | -0.888 | -4.92 | -2.068 | -4.983 | -1.912 | Darker / Less red / Less Yellow |
| | A/10 Degree | 6.515 | -1.687 | -5.14 | -3.63 | -6.014 | -1.854 | Darker / Less red / Less Yellow |
| Blue | D65/10 Degree | 3.607 | -3.605 | -0.013 | 0.123 | -0.122 | -0.018 | Darker / Less Blue |
| | A/10 Degree | 3.633 | -3.632 | -0.059 | 0.029 | -0.02 | -0.063 | Darker |

Table 6.2 illustrates that after dyeing, the nano-wool treated polyester fabrics had obviously noticeable colour difference with CIE ΔE* values much greater than 1 when compared with those of the untreated polyester fabrics. It was also demonstrated that the nano-wool treated polyester fabrics in red, yellow and blue colours were darker than those of the untreated polyester fabrics with negative CIE ΔL* values. As for the nano-wool treated yellow and red dyed polyester fabrics, they

were less redder and less yellowier than those of the untreated polyester fabrics under D65 and A light sources, i.e. CIE Δa* and CIE Δb* values were negative.

Similar to the explanation in Chapter 5, the nano-wool particles applied onto the polyester fabrics might behave like the small capillaries adhering on both the surface and internal part of polyester fibres. They did not only cause more water molecules to be adsorbed on the polyester fibre surface, but also induced more water molecules to move inside the fibres, resulting in enhancing the hydrophilicity of polyester fibres (Adler and Walsh, 1984; Gu L, 1989). Since water was used as the medium for dyeing, more water molecules would be absorbed by the polyester fibres, implying that more dyestuff molecules could enter inside the fibres during dyeing.

Moreover, the “free volume model” is applied to the study of the dyeing rate of disperse dye on polyester fibres. The free volume model is based on the concept that the fibre must have a certain size “hole” through which absorbing species can diffuse into the fibre. According to the free volume theory, the movement of a polymer segment requires the presence of space (free volume) into which the segment can move. In turn, the rotating segment vacates a space making a hole through which dye molecule can diffuse (Perkin Warren S., 1996).

Based on the concept of free volume model, it was believed that the extremely small size nano-wool particles could enter the free volume of the polyester fibres. When heat was applied during the dyeing process, the nano-wool particles collided intensively with each other as well as the polyester polymer chain. This caused the extent of segmental motion and free volume to increase continuously. The enhancement in free volume could improve the probability of creating a hole which was large enough for the dye molecules to diffuse through. This finally enhanced the dye uptake of the fibres.

## 6.4. Summary

In this chapter, the dyeing properties of the nano-wool treated cotton and polyester fabrics were studied by comparing their colour depths with those of the untreated and nano-wool treated fabrics after dyeing. The experimental results illustrated that the nano-wool treated fabrics had lower reflectance value and higher K/S value than those of the untreated fabrics. The uptake of dyestuff by the nano-wool treated fabrics was higher than those of the untreated fabrics. The results expressed in terms of CIELab* system showed that there was noticeable colour difference between the untreated fabrics and nano-wool treated fabrics. In general, the nano-wool treatment not only increased the colour depth of the fabrics, but also slightly affected the tone of the dyed fabrics.

# Chapter 7 Conclusions and Recommendations

## 7.1 Conclusions

Recently, nanotechnology has great impact the different areas. It is believed that the development of nanotechnology can create a tremendous market potential in the textile industry. On the other hand, many investigations have been conducted to explore the manufacturing and application of fibre particles. However, no attempt has been made to associate these two different areas together.

The concept of combining these two areas was proposed in this project. To achieve this, an innovative method was proposed to pulverise wool, cotton and silk fibres into nano-scale particles. The newly invented pulverisation method involved the use of the rotate crusher, ultrasonic crusher, ultra-turrax® and nano-colliding machine. It was found that most of the pulverisation procedures applied to various fibres were similar except the pulverisation duration. The particle size analysis and scanning electron microscopy confirmed that the particles obtained were in both micro-scale and nano-scale.

The chemical and crystallinity characteristics of the micro-scaled and nano-scaled fibre particles had been examined in order to study how the particles could alter the properties and function of fabrics. The Fourier transmittance infrared analysis illustrated that there was no great difference in the chemical structures between the micro-scaled and nano-scaled fibre particles of the original fibres. The X-ray diffraction analysis, however, demonstrated that there was a decline in the crystallinity of the pulverised wool, silk and cotton particles with respect to the decrease in particle size.

In order to explore the application of the nano-wool particles, attempt had been made to coat the nano-wool particles onto the cotton and polyester fabrics by means of pad-dry-cure method.

In the case of cotton fabric, there was a decrement in air permeability, thermal conductivity, keeping warmth ability, q-max after the nano-wool treatment. Moreover, the nano-wool treatment also reduced the OWTC and OMMC values, moisture management capability and hydrophility of the cotton fabrics. However, it could cause the swelling and enhance the wrinkle resistance and ultraviolet protection properties of the cotton fabric.

With regard to polyester fabric, the nano-wool treatment could reduce the air permeability, thermal conductivity, keeping warmth ability, q-max of the polyester fabrics. On the other hand, it could enhance the OWTC and OMMC values, moisture management capability and hydrophility of the polyester fabrics. The comparison of changes in properties of cotton and polyester fabrics caused by the nano-wool treatment was summarised in Table 7.1.

**Table7.1.** Comparison of changes in properties of cotton and polyester fabrics after the nano-wool particles treatment.

| | Cotton fabrics | Poleyster fabrics |
|---|---|---|
| Swelling of fibres | Noticeable | Not noticeable |
| Air permeability | Decrease after treatment | Decrease after treatment |
| Thermal conductivity | Decrease after treatment | Decrease after treatment |
| Keeping warmth ratio | Decrease after treatment | Decrease after treatment |
| Q-max value | Decrease after treatment | Decrease after treatment |
| OWTC value | Decrease after treatment | Increase after treatment |
| OMMC value | Decrease after treatment | Increase after treatment |
| Hydrophobic / Hydrophilic ability | Become more hydrophobic after nano-wool treatment | Become more hydrophilic after nano-wool treatment |

In the nano-wool treatment, some parameters such as (i) the amount of nano-wool added, (ii) dipping time, (iii) curing time and (iv) curing temperature did affect the properties and function of cotton and polyester fabrics in various ways. The effect of these parameters on the properties of cotton and polyester was compared as summarised in Table 7.2.

Finally, the effect of nano-wool treatment on the dyeing performance of the cotton and polyester fabrics was studied. It was achieved by dyeing the untreated and nano-wool treated cotton and polyester fabrics with reactive dyes and disperse dyes respectively and evaluating their colour difference. The experimental results illustrated that the nano-wool treatment could enhance the uptake of dyestuff by the polyester and cotton fabrics. It did not only increase the colour depth of the dyed fabrics, but also affected the tone of the dyed fabrics slightly.

**Table 7.2.** Comparison effect of different parameters on properties of nano-wool particles treated cotton and polyester fabrics

| | Air permeability | | Hydrophobicity | | Thermal conductivity | | Keeping warm ratio | | Q-max value | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Cot$^{\#}$ | PE $^{*}$ | Cot$^{\#}$ | PE $^{*}$ | Cot$^{\#}$ | PE $^{*}$ | Cot$^{\#}$ | PE $^{*}$ | Cot$^{\#}$ | PE $^{*}$ |
| Increase in amount of nano-wool added | ↓ ↓ | ↑ | ↓ | ↑ ↑ | ↑ | ↑ | ↑ | ↑ | ↑ ↑ | ↑ |
| Increase in dipping time | ↓ | ↑ | - | ↑ | ↑ ↑ | ↓ | ↓ | ↑ | ↑ | ↑ |
| Increase in curing time | ↓ | ↑ | - | - | - | ↓ ↓ | ↑ ↑ | ↑ ↑ | ↓ | - |
| Increase in curing temperature | ↑ | ↓ | ↑ ↑ | ↓ ↓ | ↑ | ↓ ↓ | ↓ | ↑ ↑ | ↑ | ↓ ↓ |

Remark: The double marking of arrows represents the most significant factors affecting the properties of the nano-wool treated fabrics.

Cot$^{\#}$ represents the nano-wool treated cotton fabric.

PE $^{*}$ represents the nano-wool treated cotton fabric.

## 7.2 Recommendations

The thesis provides a feasible means of pulverising fibres into nano-scaled fibre particles and applying these particles to modify the properties, function and dyeing performance of textile material in the textile industry. Due to the time constraint, some of the research study have not been thoroughly explored. Some future works related to this tropic are recommended and summarised as follows.

1. More research works should be conducted to pulverise other kinds of fibres into nano-scaled fibre particles using various means before applying these particles onto different kinds of fabrics.

2. The modification of properties and functions of fabrics treated by different kinds of nano-scaled particles should be studied and compared.

3. The mechanism of how the nano-scaled particles can alter the properties, function and dyeing performance of the treated fabrics is also worth to be investigated. The effect of heat and binder on the properties of treated fabrics should also be investigated.

4. In order to study the reality and possibility for applying nano-scaled fibre particles in the textile industry, the cost of the technology involved as well as the stability and washing durability of the nano-scaled fibre treated fabrics should also be examined.

5. Moreover, more research works should be conducted to study the chemical, biological, physical properties and safety of the nano-scaled fibre particles and their treated fabrics.

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