Application of Traditional Medicines on Textiles

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Design and Social Context

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Thesis presented
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**Declaration**

I, Ashish Swarup certify that:

a. Except where due acknowledgement has been made, the work is that of the candidate alone;

b. The work has not been submitted previously, in whole or in part, to qualify for any other academic award;

c. The content of the thesis is the result of work which has been carried out in the School of Fashion and Textiles, R.M.I.T. University between February 2005 and August 2007.

Ashish Swarup
August 2007
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Abstract

As science and technology has developed, the manner by which drugs can be delivered has grown. Medicines may be taken orally, applied as topical pastes, creams or oils, patches or administered by direct injection to the body. This research explores an alternative method for the delivery of therapeutic compounds to the body. The basis of the study involves the application of traditional medicinal compounds used to cure minor neck and back pains through transdermal topical application, using a textile substrate as the carrier.

Boswellia Serrata Extract (B.S.E.) is a common traditional medicine used in South Asia for curing body pains. The most common form of B.S.E. based products are creams. These are applied directly to the skin by users. Experiments show that these creams were not suitable as a basis for applying to textile materials, and then for transfer to the skin. This is due to such creams containing highly volatile compounds, which on drying the treated textile, post application, cause almost total loss of the B.S.E. The approach used was the application on textiles of a ‘commercial’ topical medicine applied as a cream for pain relief, where B.S.E. is a major constituent. Cotton woven fabric was padded with this cream and tested for washing and rubbing fastness. The presence of highly volatile substances in the topical cream resulted in a negligible amount of the medicine on the dried treated fabric.

Another approach was used for the application of B.S.E. onto the textile substrate. A commercially available B.S.E. powder was applied to woven fabric by a pad mangle. Tests were carried out to validate the “take-up” of the B.S.E. powder on the woven cotton fabric and to measure the wash fastness of the padded fabric. The aim was to assess the possibility of using the padded fabric for assembly into a garment, also assessing was whether the garment could withstand laundering and retain the B.S.E. for transdermal permeability in addition, a test was conducted to measure the amount of B.S.E. transferred from padded fabric to the skin over a specified span of time. However, due to ethics involvement, the tests were not conducted on humans or animals, but rather an alternative method was devised to evaluate the transfer of material from woven cotton fabric to the skin surface. Leather was used in the evaluation of dermal transfer of B.S.E. Rubbing fastness was also determined. Other tests completed were conducted on Perkin Elmer Spectrum 2000 FTIR Spectrum and ESEM on FEI Quanta 200.
Due to low wash and rubbing fastness values on the apparel fabrics and negligible transfer of B.S.E. powder from the treated fabric surface to the skin surface, an alternative approach was followed. The B.S.E. powder was coated on polyester viscose blended non-woven fabric. Using coated fabrics as patches that do not require washing fastness make them easy to use. The effectiveness of the dermal transfer was assessed using rubbing fastness, scanning electron microscopy and FTIR Spectroscopy.

This thesis has shown that the effective approach for using a textile fabric as a medium for applying B.S.E. to the skin is coating on non-woven material that can be used in patch form. The application of B.S.E. on woven fabric results in low solid add-on on the fabric, and increases the stiffness of fabric resulting in unsuitability of the finished woven fabric for garment construction.
CHAPTER 1

Introduction

In the past few decades there have been important developments in using one field of science to support another. Textiles, one of the oldest of technologies has advanced extensively, resulting in the improvement in the quality of life. Nowadays, textiles are being used to find solutions to medical problems by enhancing the properties of the textile materials, therefore improving the comfort of application. A specific aspect in the medical field is the delivery of drugs. Most commonly used methods of drug delivery include taking pills orally, applying ointments and administering injections. More recently is the development of transdermal drug delivery using “textile” patches. Such indirect method for the administration of drugs is more applicable and therefore practical with children, people having swallowing difficulties or people who are simply forgetful. These applications are also more suitable for prolonged drug treatment or where the therapeutic agent has no side effects in the case of an overdose [1].

The advantage of these alternative methods is the relatively low dosage to oral delivery systems. In oral delivery systems, the drug is absorbed by the intestinal tract [1, 2]. High dosage in oral delivery of drugs is required because of drug metabolism, resulting in the loss of efficacy before fulfilling their medical purpose and generating more liver toxic metabolities [1, 2]. This process is excluded in the delivery of drugs through the skin by-passing the liver passage.

Textiles provide basic human requirements for modesty, protection and comfort. The permeable breathing structure of textiles with absorptive capacity results in its use in the medical field. This study explores the application of Boswellic Acid in Boswellia Serrata Extract powder, a traditional South Asian medicine used for pain relief, onto the textile substrate. Other aspects which are usually important in textiles include fastness and washability properties. For this purpose, these topics have also been addressed in the study.
Chapter 1.1

Objectives of this thesis

This research investigates the use of textiles as an alternative approach for delivery of Boswellia Serrata Extract, a traditional South-Asian medicine. The work explores the use of woven and non-woven fabrics as carrier for transdermal drug delivery to relieve minor neck and back pains.

The introduction to this thesis is presented in two distinct sections:

One: An overview of the textile substrates and their properties.

Two: Discusses Boswellia Serrata Extract (B.S.E.), a traditional South Asian medicine known for its anti-inflammatory properties. Also discussed is the delivery of Boswellic Acids as anti-inflammatory agents, using textiles as a carrier.
Chapter 1.2

Textiles: An Overview

“Textiles” is a commonly used term that has a history going back many thousands of years. There has been evidence of fibre technology as old as 26,000 years back from the upper Palaeolithic period [3] and twisted fibres dated 19,000 years old [4]. Evidences of textile from Mesopotamia dates back to 3000 B.C. and from Denmark in the Bronze Age in the late 2nd Millenium B.C. [5]. The earliest known evidence of a vertical two beam loom can be seen in an Egyptian wall painting (dated late 15th century B.C.) at Thutnofer's tomb [5]. There has been evidence of cloth from the Catal Huyuk and Nahal Hemar fabrics from before 6000 B.C. [5].

A definition of textiles is “Any filament, fibre, or yarn that can be made into fabric or cloth, and the resulting material itself” [Britannica Encyclopedia]. Traditionally the term “textiles” was used for woven fabrics. Today it includes knitted, bonded, felted, tufted and other forms of construction. The basic raw material used in textile production is fibres. These fibres can be from natural sources or produced from chemical substances. Textiles have varied uses, ranging from apparel, household linens and bedding, upholstery, draperies and curtains, wall coverings, rugs and carpets and bookbinding. In addition, various textiles are used widely in many industries. Another area of textile usage is technical textiles. This now accounts for 25% of all fibre consumed and 40% of the total textile activity [6 & 7].

Characteristics of fabrics mainly depend on fibre or yarn properties. Important fibre characteristics include the fibre source, its chemistry and structure, length, fineness, denier, strength, flexibility, cohesiveness, elongation, modulus, elasticity, moisture absorption and flammability. Important yarn characteristics include the number of fibres per cross section, yarn structure, yarn linear density and the type of yarn manufactured. The basic properties of fabrics are determined by its construction (i.e. woven, knitted or non-woven), fabric thickness, density and any subsequent chemical or mechanical treatment finishes performed on the fabric.

The major source of raw material for fibres is vegetables, animals or oil. Fibres can be characterised under natural or man-made fibres (as shown in Figure 1). The type of fibre used
will determine the desired properties of yarn, which will in turn determine the properties of the desired end product and its use.

![Fibre categorization](image)

**Figure 1: Fibre categorization.** [64]

Whilst natural fibres have high moisture absorbing property, man-made fibres can be modified during the manufacturing process to improve the moisture absorbency of the fibre depending on the end usage.

Even though regenerated fibres are classified as man-made fibres, they are cellulose based fibres that have similar properties to vegetable fibres. In addition, they have high moisture regain.

Other man-made fibres are classed as synthetic fibres. Although these fibres have low moisture regain, they have the advantage of easy care property. The properties of man-made fibres can be modified during the manufacturing process.

The last of the man-made fibres are Inorganic fibres which are mainly used in technical textiles.

The same fibre can have different commercial names as their chemical and physical properties are modified during the manufacturing process. Each trade name defines a different set of properties for the fibre.
1.2.1

Fabric structures

Fabrics can broadly be classified into (i) woven fabrics (ii) knitted fabrics and (iii) non-woven fabrics. They are manufactured using yarns, fibres or combination of both. Textile fibres can be in filament or in staple form. All man-made fibres are manufactured as continuous filaments but can be cut into short staple fibres, whereas most natural fibres are naturally in staple form. During the spinning process, staple fibres are assembled together and then twisted to a desired level to form the yarn. These yarns are used as raw materials to manufacture woven and knitted fabrics.

Woven fabrics are manufactured using warp yarns (where parallel yarns lie side by side) and weft yarns (yarns lying perpendicular to the warp) are crossed as shown in Figure 2a. The configuration of the warp and weft crossing is called a weave. The most common weaves are plain weave, twill and satin. Woven fabrics tend to be more rigid when compared to knitted structures. Depending upon the stretch wovens require, an elasticated yarn or fibres (elastane) can be inserted in the warp or weft direction.

Knitted fabrics are manufactured using loop formations by a single yarn (as shown in Figure 2b). The two basic methods of knitting are warp and weft knitting. The characteristics of a weft knitted structure are that the fabric is elastic in both directions, whereas in warp knitted fabrics elasticity is only in the width direction. Elasticity of fabrics can be increased using elastane fibres in the type of yarn used. The type of raw materials used will almost always determine the desired properties and characteristics of the end product.

Non-woven fabrics are manufactured using fibrous webs that are mechanically, thermally or chemically bonded to each other (Figure 2c). Non-woven fabrics are low cost manufactured textiles as they do not use the costly process of spinning. Their consumption is high in medical textile products.
(a) Woven fabrics.

Source: http://ms.cc.sunysb.edu [66]
The production of textiles is an ancient technology, however mass production and the application of modern manufacturing techniques used in today’s society has forever altered the speed and scale of production, beyond recognition.
1.2.2
Dyeing and finishing

The manufacturing of textiles includes colouration and final finishing. Dyeing and finishing agents can be applied to fibre, yarn or fabric. The finishing treatments can improve the comfort, performance, durability, dimensional stability and handle of the final product. Common finishes commercially used are antistatic, water repellency, soil repellency, stain release, flame retardant and thermal treatments to change a fabric to the desired end requirements and dimensions. Most finishing treatments last for a certain number of washes. This depends on the finish applied to the fabric and the life span of the end product.

The finishing process may involve desizing, scouring, bleaching, mercerisation (for cotton fabrics), cropping or shearing, brushing and steaming, singeing and other treatments to increase the functional properties of the fabric. Wet finishes can be applied through exhaustion, padding or coating. The most commonly used being padding.

The padding process can be applied using a conventional pad-mangle system including modern low add-on systems that include foam technology.

Coating is another process commonly used for application of rubber, polyvinyl chloride, polyurethanes, acrylic polymers, adhesive treatments and radiation-cured coatings. A Coating is the layer of polymeric material applied on the textile to impart new characteristics to the basic fabric [7].

In resin coatings, both the general purpose resins and dispersion resins have different characteristics that define the coating. The most important characteristics are molecular weight, particle size, bulk density, dry flow or settling of resin [7].

Coating can be applied to the fabric by fluid coating and using dry compound (solid powder or film). For fluid coating, the coating material is in the form of paste, solution, or latices. The different types of fluid coatings are as follows:

- The post metering devices: Knife coaters, wire wound bars, round bars, etc
- The pre-metered application systems: Roll coaters, reverse roll coaters, kiss coaters, gravure coaters, dip coaters, etc
• Impregnators, where material is coated by dipping textile in the fluid and the excess fluid is removed by squeeze roll or doctor blades
• Spray coaters where the material is sprayed on the textile web or roll

On the other hand, dry compound coating can be done using the following processes:
• Melt coating for extrusion coating and powder coating
• Calendering, as used for thermoplastic polymers and rubber compounds, etc
• Lamination

The method used for coating depends on the following factors:
• Nature of substrate
• Form of resin and viscosity of coating fluid
• End product and accuracy of coating desired
• Economics of the process

Even though all processes from fibre to finished fabric have an important role in the characteristics of the textile, the end use of textile defines the finishing process followed to impart the required properties to the textile substrate.
1.2.3

Textile properties

An important aspect of textiles is the defining features of the fabric. These features are commonly referred to as “characteristics”. Textile materials can be characterised by the following [9]:

- Raw material used
- The fibre content
- Mass per unit area (gsm of the fabric)
- Thickness of the fabric
- Fabric construction (Woven, knitted & non-woven)
- Expected lifespan of the fabric or finish
- Care maintenance for the textile product
- Abrasion resistance (depending upon the use of the product)
- Tear resistance of the fabric
- Breaking strength of the textile product
- Seam strength
- Bursting strength
- Elastic properties
- Colour fastness
- Fabric finish (e.g.; crease resistance, stain repellancy, soil repellancy, water resistance & repellant, seam puckering, reactivity of finish with human skin and toxicity of the finish)
- Drapeability depending on the requirement for aesthetics
- Static charge generation in the fabric (also depends on the end requirement)
- Pilling of the textile
- Thermal insulation
- Water vapour transmission and breathability of the textile
- Air permeability, mainly for active wear and outdoor wear
- Handle properties (feel of the fabric)
- Shrinkage of the textile after washing and steaming
Use of medical textile products has expanded from hospitals, hygiene and the healthcare sector, to hotels and other environments where hygiene is required. Another use for medical textiles that has been explored by researchers is Nanotechnology. Studies completed by David Rigby Associates in 2003 and 2005 (world market forecasts to 2010) demonstrate that there is a noticeable growth expected for this sector. Textiles can impart antibacterial, antifungal and mildew resistance properties in order to combat potential bacteria. In order to develop such textile materials, different compounds are coated, padded or exhausted on the textile substrate. These additional properties are given to the textile substrates during the finishing process. Finishing can be administered in the fibre, yarn and fabric form.
Chapter 1.3

Boswellia Serrata Extract (B.S.E.)

1.3.1

Introduction:

Throughout the ages, man has been dependent on his environment for food, shelter and clothing. The primary source that has satisfied these needs have comes from plants and plant materials. These “materials” are used nowadays for food, shelter, clothing, transport, spices, fragrances, cosmetics, colour and medicinal ingredients.

One of the most important plant extracts that has been extensively used by mankind is resins. Resins have been used as adhesives, ingredients in cosmetic preparations, for fragrances, during religious ceremonies and for coating for a range of medicinal purposes.

In ancient times, Hindus, Babylonians, Assyrians, Persians, Romans, Chinese and Greeks (as well as the people of old American civilizations like Incas, Mayas and Aztecs) used natural resins for embalming and for incense in cultural ceremonies [10]. These people believed that when these materials were used in contact with fire, the smoke and the fragrance produced would not only soothe their souls but also please their Gods. Burning of these natural resins had become an important part of their cultural life [10]. It was thought that burning these resins during scarification ceremonies or in their daily rituals would prevent the influence of bad spirits on one’s soul (or alternatively used to honour the dead or living people) [10].

For centuries these resins have also been used for medicinal purposes [11 & 12]. The use of such material in wound healing and during operations has been described in ancient scripts. In the first known pharmacopoeia [13, 14 & 15], there were approximately 600 remedies advised. These remedies were prepared from mixtures of natural resins and balsams as well as some herbal preparations.
1.3.2
The History of Characterisation of Resins

Alexander Tschirch (1856-1939), a pharmacologist, was the first to study natural resins in detail [10]. His studies involved the identification of the physical and chemical characteristics of resins, the classification into different types, and their chemical compositions. In 1906, Tschirch published the results in his book [16]. He then developed a methodology that mainly depended on the cold extraction and fractionation of the resin material with different solvents [10] (Figure 3) to characterize the chemical composition of the natural resins.

Further characterization, achieved through modifications such as acetylation, benzylolation or esterification, helped him to comment on the functionality and elemental composition of the resin constituents [10].
As time has passed, technology has developed in extraction and fractionation to allow improved chemical characterisation of natural resins. The result from the improved method of extraction and characterization of natural resins explores many unfound mysteries of these natural resins, answering the reason behind the medicinal use of these resins in ancient civilizations [10].
1.3.3

What is Olibanum?

Olibanum is an important class of natural resins. They are also referred to as “oleo-resins” or “oleogum” resins. These oleo-resins exude from incisions in the bark of the Boswellia family of plants. This resin has been important to civilizations for over 5000 years as it was considered an important trade material [17, 18 & 19]. It has been of interest to Kings and Queens, including the Queen of Sheba 700 B.C. [20 & 21].

Frankincense is another name for the oleo-resin Boswellia. Frequent references were made to frankincense in the Bible. For example, the Gospel of Matthew (2:1-2:12)

... where the baby Jesus was born in a manger and how the three Magis went to pay homage to him ... “The sight of the star filled them with delight and going on their knees they did him homage. Then opening their treasures, they offered him gifts of gold and frankincense and myrrh.”

 solop of Matthew (2:1-2:12)

Pharmaceutical companies have recently shown interest in oleo-resins. Even though these resins have been used by ancient civilizations for medicinal purposes, the development in technology has answered many unsolved mysteries about these resins. This created an interest of pharmaceutical companies into these oleo-resins. The most important feature that draws on the attention of these companies is the anti-septic, anti-inflammatory, anti-arthritic, anti-tumour and hypolipidaemic properties, including their similarity with synthesized medicinal compounds.

In the ancient traditional Indian medical texts, the gummy exudate from Boswellia is grouped with other gum resins and referred to collectively as “gugguls” [22].
1.3.4

Boswellia Serrata: An Overview

The anti-inflammatory property of the Boswellia family is derived from the presence of Boswellic Acids in the terpenoids of the oleo-resin exuded from the plant. The most prominent species of the Boswellia family are Boswellia Serrata, Boswellia Carterii, Boswellia Frereana, Boswellia Neglecta and Boswellia Rivae.

When the tree trunk is tapped, a gummy oleoresin percolates out. Oleo-gum-resin is fragrant, transparent, and brownish-yellow in colour. The active component in the gum oleoresin consists of essential oils, gum and terpenoids. The terpenoid portion contains the Boswellic Acids. Boswellic Acid has been identified as the active constituent in Boswellia Serrata.

Isoprenoids, terpenes and terpenoids are universal metabolites present in all living organisms. Studies have shown that the presence of Boswellic Acid, which is responsible for anti-inflammatory property, is highest in Boswellia Serrata.

The Boswellia Serrata (more commonly known as Salai guggul or guggul) plant is a medium to large sized deciduous balsamiferous tree primarily found in the dry forests of northern, eastern and peninsular part of India [23]. The aromatic oleo-gum-resin exuded from the plant has powerful anti-inflammatory qualities that have now been proved through clinical research [24, 25]. It has also been shown to possess cholesterol and triglyceride lowering activity [24, 26 & 27]. Clinical trials on rheumatic patients have also shown promising results [22]. The gummy extract from the Boswellia Serrata has been used traditionally for pains and stiffness in joints. Now it is also used as an expectorant and diuretic. Its other pharmacological uses include treatment of diarrhoea, dysentery, skin disorders and pulmonary infections.

The gum is credited with astringent, stimulant, expectorant, diuretic, diaphoretic, antipuretic, stomachic emmenagogue, ecbolic and antiseptic properties. It is reported to be useful in treatment of ulcers, tumours, goitre, cystic breast, diarrhea, dysentery, piles, asthma, bronchitis, chronic laryngitis, syphilitic, jaundice and skin diseases [28]. In addition, it is used in the preparation of an ointment for sores [29] and is used to cure syphilis [30, 31]. Chronic toxicity studies in healthy monkeys revealed that the drug do not have bio-chemical, hematological and other toxicities [22].
Boswellic Acids are effective anti-inflammatory and anti-arthritic agents. They can be used for osteoarthritis and rheumatoid arthritis, soft tissue rheumatism, and lower back pain. They also help control excessively high blood lipids and atherosclerosis [32], and protect the liver against bacterial galactosamine-endotoxins [33].
1.3.5

Boswellia Serrata: Technical Description

Commercially available Boswellia Serrata extracts are “standardized” to contain 37.5-70% Boswellic Acids. Typical marketable extracts of Boswellia plants are available in solids or powder form. They have a wider melting range [34].

Although the Boswellia Serrata Extract can be dissolved in solvents such as methanol, ethyl acetate, acetone and the like; these solvents are not suitable for topical or internal applications because of their aggressive action on human tissue and hair. This can be attributed to their high volatility and strong odour [34].

Other common solvents such as glycerine, sorbitol, mineral oil, cyclomethicone, dimethicone and petrolatum are not suitable for Boswellia extracts, Boswellic Acids or their derivatives as they are either too polar or not sufficiently polar [34].

Alkalies can produce aqueous solutions of Boswellic Acids by forming the respective salts. However, prolonged skin or hair contact by alkaline products (e.g. with a pH greater than 8.5) is not recommended, especially for sensitive skin or scalps [34].

However, a group of fatty alcohols or acids (or derivatives and their mixtures) are suitable carriers for dissolving and dispersing at least one Boswellic Acid or their derivative. Furthermore, these carriers aid the incorporation of the extract or acid, into compositions suitable for use on the skin or hair. They also improve the stability of compositions suitable for human usage [34].

Saturated fatty alcohols generally have good stability against oxidation, a key requirement for personal creams, pastes or ointments. Saturated and branched fatty alcohols and acids that have 8 to 20 carbon atoms are preferred. Particularly useful bases for preparing B.S.E. preparations are isostearyl alcohol, isostearic acid, isocetyl alcohol, isopalmitic acid, octyldodecanol, octyldecanol, hexyldecanol, butyloctanol and ethylhexanol [34].

Unsaturated fatty alcohols and unsaturated fatty acids (containing 12 to 24 carbon atoms which are liquid at room temperature) are also good carriers for Boswellia extracts. Particularly useful compounds include oleyl alcohol, oleic acid, linoleic acid and linolenic
acid. However, their presence may be disadvantageous in compositions requiring long term storage. Because they are unsaturated, they have the potential to become oxidised and rancid over time [34].

A typical extract of a Boswellia plant comprises a mixture of Boswellic Acids comprising at least one of 3a-hydroxyurs-12-ene-23-oic acid, 3a-acetoxyurs-12-ene-23-oic acid, 3a-hydroxyurs-12-ene-11-keto-23-oic acid and 3a-hydroxyurs-9, 12-dien-23-oic acid.

All Boswellic Acids have a Pentacyclic structure based on 12-ursen-23-oic acid with differing substituents. Many individual Boswellic Acid compounds have been isolated from the Boswellia extract including Alpha- and Beta-Boswellic Acids and their derivatives. Of these, Beta-Boswellic Acid and their derivatives are thought to be the active components.

Specifications of the Beta-Boswellic Acid, which is of high interest for this research, are provided in Table 1 – 4 on page 23 & 24. This specification has been taken from literature provided (on the website) by Sabinsa Corporation, a major supplier of Boswellia Serrata Extract, Piscataway, NJ, USA.
The standard HPLC chromatograms [35] for the four beta-Boswellic Acids are shown in Figures 4 and 5:

**Figure 4: HPLC CHROMATOGRAM OF b-BOSWELLIC ACIDS AT 210 nm**

Retention time (RT) is 17.213 minutes for Beta-Boswellic Acid

Retention Time is 25.278 minutes for Acetyl-Beta-Boswellic Acid

**Figure 5: HPLC CHROMATOGRAM OF b-BOSWELLIC ACIDS AT 254 nm**

Retention Time is 7.393 minutes for 11-Keto-Beta-Boswellic Acid

Retention Time is 10.835 minutes for Acetyl-11-Keto-Beta-Boswellic Acid
Specifications of **BOSWELLIC ACIDS** (Source: Sabinsa Corporation)

### Table 1: β-Boswellic Acid (BA)

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Chemical structure:</td>
<td><img src="image" alt="Chemical Structure" /></td>
</tr>
<tr>
<td>2. Chemical name</td>
<td>3a-Hydroxy-urs-12-en-23-oic acid</td>
</tr>
<tr>
<td>3. Molecular formula</td>
<td>C_{26}H_{40}O_{3}</td>
</tr>
<tr>
<td>4. Molecular weight</td>
<td>456.7</td>
</tr>
<tr>
<td>5. Melting point</td>
<td>226 - 228°C (Lit. mp 228 - 230°C)</td>
</tr>
<tr>
<td>6. Specific rotation</td>
<td>$-106.8^\circ$ (Lit. $-108^\circ$)</td>
</tr>
<tr>
<td>7. FTIR (in KBr)</td>
<td>3500 cm$^{-1}$, (OH) 1699.5 cm$^{-1}$, (COOH)</td>
</tr>
<tr>
<td>8. UV (methanol)</td>
<td>Maxima at 208 nm (Lit. 208 nm)</td>
</tr>
<tr>
<td>9. NMR (in CDCl$_3$)</td>
<td>5.15 (CH=C, vinylic proton) 4.08 (CH-OH) 2.3-1.1 (Methylene and methine 23 protons) 1.1-0.7 (Methyls, 21 protons)</td>
</tr>
<tr>
<td>10. GC-MS</td>
<td>394 (M+68[44 due to CO$_2$ &amp; 18 due to -H$_2$O]) 218 (base peak, due to retro-Diels-Alder fragmentation).</td>
</tr>
</tbody>
</table>

### Table 2: Acetyl-Beta-Boswellic Acid

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Chemical structure</td>
<td><img src="image" alt="Chemical Structure" /></td>
</tr>
<tr>
<td>2. Chemical name</td>
<td>3a-Acetoxy-urs-12-en-23-oic acid</td>
</tr>
<tr>
<td>3. Molecular formula</td>
<td>C_{26}H_{40}O_{3} Molecular weight 498.74</td>
</tr>
<tr>
<td>4. Melting point</td>
<td>252 - 255°C (Lit. mp 253°C)</td>
</tr>
<tr>
<td>5. Specific rotation</td>
<td>$+138^\circ$ (Lit. $+141.3^\circ$)</td>
</tr>
<tr>
<td>6. FTIR (in KBr)</td>
<td>1732 cm$^{-1}$ (OAc), 1701 cm$^{-1}$ (COOH)</td>
</tr>
<tr>
<td>7. UV (methanol)</td>
<td>Maxima at 208 nm (Lit. 208 nm)</td>
</tr>
<tr>
<td>8. NMR (in CDCl$_3$)</td>
<td>5.31 (CH=C, vinylic proton) 5.2 (CH-OAc) 2.1 (COCH$_3$) 1.9-1.25 (Methylene and methine, 23 protons) 1.2-0.7 (Methyls, 21 protons)</td>
</tr>
<tr>
<td>9. GC-MS</td>
<td>394 (M+104[44 due to CO$_2$ &amp; 60 due to -HOAc]) 218 (base peak, due to retro-Diels-Alder fragmentation).</td>
</tr>
</tbody>
</table>

*1: Sabinsa Corporation, a major supplier of Boswellia serrata Extract (commercial name Boswellin®) Piscataway, NJ, USA.*
### Table 3: 11-Keto-Beta-Boswellic Acid (KBA)

<table>
<thead>
<tr>
<th>1. Chemical Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image1" alt="Chemical Structure" /></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>2. Chemical name</th>
<th>3α-Hydroxy-urs-12-en-11-keto-23-oic acid</th>
</tr>
</thead>
<tbody>
<tr>
<td>3. Molecular formula</td>
<td>C_{32}H_{44}O_{2} Molecular weight: 470.69</td>
</tr>
<tr>
<td>4. Melting point</td>
<td>195 - 197°C (Lit. mp 195°C)</td>
</tr>
<tr>
<td>5. Specific rotation</td>
<td>+78.5° (Lit. +79.5°)</td>
</tr>
<tr>
<td>6. FTIR (in KBr)</td>
<td>3460 cm(^{-1}) (OH), 1693 cm(^{-1}) (COOH) 1647 cm(^{-1}) (a,b unsaturated carbonyl)</td>
</tr>
<tr>
<td>7. UV (in Methanol)</td>
<td>Maxima at 250 nm (Lit. 250 nm)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>8. NMR (in CDCl(_3))</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.55 (CH=C, vinyl proton)</td>
</tr>
<tr>
<td>4.06 (CH-OH)</td>
</tr>
<tr>
<td>2.6-1.4 (Methylene and methine 21 protons)</td>
</tr>
<tr>
<td>1.25-0.75 (Methyls, 21 protons)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>9. GC-MS</th>
</tr>
</thead>
<tbody>
<tr>
<td>408 (M-68 due to CO(_2) &amp; 18 due to H(_2)O) 252 (base peak, due to retro-Diels-Alder fragmentation).</td>
</tr>
</tbody>
</table>

### Table 4: Acetyl-11-Keto-Beta-Boswellic Acid (AKBA)

<table>
<thead>
<tr>
<th>1. Chemical Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image2" alt="Chemical Structure" /></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>2. Chemical name</th>
<th>3α-Acetoxy-urs-12-en-11-keto-23-oic acid</th>
</tr>
</thead>
<tbody>
<tr>
<td>3. Molecular formula</td>
<td>C_{32}H_{44}O_{2} Molecular weight: 512.73</td>
</tr>
<tr>
<td>4. Melting point</td>
<td>271 - 274°C (Lit. mp 271°C)</td>
</tr>
<tr>
<td>5. Specific rotation</td>
<td>-88.5° (Lit. -87.0°)</td>
</tr>
<tr>
<td>6. FTIR (in KBr)</td>
<td>1740 cm(^{-1}) (AO), 1701 cm(^{-1}) (COOH) 1647 cm(^{-1}) (a,b unsaturated carbonyl)</td>
</tr>
<tr>
<td>7. UV (in Methanol)</td>
<td>Maxima at 250 nm (Lit. 250 nm)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>8. NMR (in CDCl(_3))</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.55 (CH=C, vinyl proton)</td>
</tr>
<tr>
<td>5.2 (CH-OAc)</td>
</tr>
<tr>
<td>2.6-1.4 (Methylene and methine 21 protons)</td>
</tr>
<tr>
<td>1.25-0.75 (Methyls, 21 protons)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>9. GC-MS</th>
</tr>
</thead>
<tbody>
<tr>
<td>408 (M-68 due to CO(_2) &amp; 18 due to H(_2)OAc) 252 (base peak, due to retro-Diels-Alder fragmentation).</td>
</tr>
</tbody>
</table>
1.3.6

Acid Components of Boswellia Serrata

A typical analytical result for a commercial Boswellin (Sabinsa Corporation, Piscataway, NJ, USA) is shown in Table 5.

The concentration of β-Boswellic Acids by HPLC in Boswellia Serrata Extract was:

<table>
<thead>
<tr>
<th>Component</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>β-Boswellic Acid (β BA)</td>
<td>10.1</td>
</tr>
<tr>
<td>Acetyl-β-Boswellic Acid (A β BA)</td>
<td>6.8</td>
</tr>
<tr>
<td>11-keto-β-Boswellic Acid (KBA)</td>
<td>5.1</td>
</tr>
<tr>
<td>Acetyl-11-keto-β-Boswellic Acid (AKBA)</td>
<td>3.8</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>25.8</strong></td>
</tr>
</tbody>
</table>

A random sampling was done by the Sabinsa Corporation and it was concluded from the average result from 7 random samples of Boswellin® that the total organic acid by titration is 70.9% [35].
1.3.7

The Anti-inflammatory properties of Boswellia Serrata Extract

Although the anti-inflammatory properties of Boswellia Serrata Extract (B.S.E.) have been known for centuries, it was not until the early 1990’s that effective constituents were identified as Boswellic Acids [36]. The most important active components identified were Acetyl 11-Keto β-Boswellic Acid (AKBA) and 11-keto β-Boswellic Acid (KBA).

Boswellic Acids share the same basic pentacyclic triterpene (steroid-like) structure and only differ in their side groupings.

In a recent study, it was shown that among the Boswellic Acids, Acetyl-11-keto- β -Boswellic Acid was observed to have the most pronounced inhibition of 5-lipoxygenase product formation [37].

It has been shown to be the inhibitor of 5-lipoxygenase and human leukocyte elastase [38, 39]. It has been proposed in the treatment of various inflammatory conditions [40].
The Human Body & Boswellia Serrata: The Mechanism of Anti-inflammatory Action

The human body undergoes many complex activities at the same time. Even though our perception of inflammation is that it is a simple process of the human body, it is in fact a complex process that involves a series of actions and reactions. Inflammation involves a range of biologically active substances (e.g. bradykinins, histamines, prostaglandins, thromboxanes, hydroxy-fatty acids, leukotrienes, lysosomal enzymes, and lymphokines) that are triggered by the body’s immunological response to the tissue damage [41].

Of all these biologically active substances stimulated, the most important mediator in the inflammatory and allergic processes are the leukotrienes. The leukotrienes are derived from arachidonic acid. The arachidonic acid is an essential fatty acid that is synthesized in the body via the enzyme 5-lipoxygenase [41, 42, 43].

The acetyl-11-keto-β-Boswellic Acid (AKBA) restraints the 5-lipoxygenase (5-LOX) by a selective, enzyme-directed, non-redox and non-competitive mechanism [44]. β - Boswellic Acid (β-BA), that lacks 11-keto-function, also has a property of restraining 5-LOX (5-lipoxygenase) activity, but not as much as AKBA. The alcohol from β-BA and amyrin, acetyl-11-keto-amyrin and 11-keto-β-Boswellic Acid methyl ester (the other extracts from the resin) do not have a 5-LOX inhibiting property [45]. There is a common binding site for pentacyclic triterpenes with and without intrinsic 5-LOX inhibitory activity. The pentacyclic ring system is crucial for binding to this effector site, as functional groups (especially the 11-keto-function and a hydrophilic group on C4) are essential for 5-LOX inhibitory activity [45]. The anti-inflammatory compounds most commonly used around the world are Naproxen, Nimesulide and Refecoxib. These are antinociceptive agents that act via cyclooxygenase (COX) [46, 47].

Preliminary studies [48] reason that Boswellic Acids (BAs), as specific, non-redox inhibitors of 5-lipoxygenase. Therefore BAs inhibited the production of inflammatory leukotrienes. Based on IC₅₀ (effective inhibitory concentration of tested substance) values, acetyl-11-keto-beta-Boswellic Acid alone provided the most potent inhibitory action due to its optimal structure [48, 49].
Studies [48] suggest that Boswellic Acids inhibit 5-LOX either by directly interacting with 5-LOX or by interacting with five-lipoxygenase-activating protein (also called as FLAP) [48]. The two pro-inflammatory enzymes, namely 5-LOX and human leukocyte elastase (HLE) restrict Boswellic acids. Recent research gives additional information to elucidate the mechanism of this unique inhibiting property of BAs [39, 50 & 51].
1.3.9

Why Boswellia Serrata?

The process of inhibition of leukotriene synthesis is important in the understanding of the effect of Boswellic Acids on the human body. Photoaffinity labelling was used to identify the presence of the specific KABA-binding site on 5-LOX, a distinct feature from the arachidonate substrate-binding site. The labelling of human 5-lipoxygenase was done by using azido$^{125}$I-KBA (4-azido-5$^{125}$ido-salicyloyl-b-alanyl-11-keto-beta-Boswellic Acid), a radioiodinated photosensitive analog of AKBA [50]. There is characteristic similarity in inhibition of 5-lipoxygenase between Azido$^{125}$I-KBA and AKBA, but the structural difference is that it has a 4-azido-5$^{125}$ido-salicyloyl-b-alanyl moiety at the C3 position instead of an acetoxy group in AKBA [50].

In the class of leukotriene synthesis inhibitors, Boswellic Acids are preferred because of their non-redox inhibiting property. This is because Boswellic acids do not interact with other biological redox systems (unlike the redox type inhibitors), resulting in the reduction of the probability of side-effects like methamoglobin formation [51, 52]. Studies show that AKBA is found to be the only non-competitive, non-redox leukotriene synthesis inhibitor, inhibiting 5-LOX activity [50]. Synthetic non-redox type inhibitors, like L-739,010 and ZM-230,487, are prepared due to the success of Boswellic Acid for anti-inflammatory effect, but these synthetic non-redox inhibitors are still competitive type inhibitors [51].

There is less to no 5-LOX inhibitory activity of ZM-230,487 and L-739,010 in chronic inflammatory processes. It was experimentally proved that for efficient 5-LOX inhibition by ZM-230,487, a low hydroperoxide concentration was necessary [51]. Inflammation is a physiological condition and is characterized by oxidative stress and increased peroxide levels lowering the performance of synthetic non-redox type inhibitors.

One of the exclusive properties of Boswellia Serrata is that Boswellic Acid has a dual inhibitory action. Besides the 5-LOX inhibitory property described above, Boswellic Acid also inhibits human leukocyte elastase (HLE). This is a serine protease produced and released by polymorphonuclear leukocytes (PMNLs). Due to its aggressive and destructive property HLE has a role in several diseases (pulmonary emphysema, cystic fibrosis, chronic bronchitis, acute respiratory distress syndrome, glomerulonephritis and rheumatic arthritis).
Many of other leukotriene biosynthesis inhibitors do not show HLE inhibitory activity. This is shown in the figure below where beta-Boswellic Acid, AKBA, ursolic acid and amyrin significantly inhibit HLE [39].

![HLE Activity Graph](image)

**Table 6: HLE activity in the presence of leukotriene synthesis inhibitors and cyclic and noncyclic hydrophobic compounds**

*Source: http://www.boswellin.com/Mechanism-2.htm [68]*

Boswellia Serrata has vast pharmacological usage. One area of the usage is in preclinical and controlled experimental environment (rather than within a living organism or natural setting). This area comprises of apoptosis induction by AKBA, anti-inflammatory effect and anti-tumor activity. Another area is in the clinical studies are malignant glioma, ulcerative colitis and bronchial asthma.

Research has shown that unlike non-steroidal anti-inflammatory drugs, long-term use of Boswellia does not lead to irritation or ulceration of the stomach [39, 53]. It was also found
that Boswellia improves blood supply to the joints and restores integrity of vessels weakened by convulsion [54].

Boswellia Serrata has been used as a cure for bursitis, osteoarthritis and rheumatoid arthritis. Studies have shown that Boswellia has been more effective, more potent and less toxic than Ketoprofen [55]. However, Ketoprofen (also known as Benzoyl hydrotropic acid) is preferred over other anti-inflammatories such as indomethacin, phenylbutazone and acetylsalicylic acid.

Boswellic Acids have the property to suppress the proliferating tissue found in the inflamed areas. It also prevents the breakdown of connective tissue. This process is similar to the action of non-steroidal groups of anti-arthritic drugs with no side effects, gastric irritation and ulcerogenic activity.
1.3.10

Dosage of Boswellia Serrata

There have been different recommendations for the intake of B.S.E. for an effective medicinal effect on the human body in respect to anti-inflammatory processes. One study has suggested the dosage of 286 mg, 3 times daily (equals 858 mg) of Boswellia Serrata Extract (70% total Boswellic Acid Extract) for anti-inflammatory purposes [56].

It has been suggested by Emory Healthcare, Atlanta, Georgia; USA that a typical dose of Boswellia Serrata can be 300 mg to 400 mg, three times daily for an extract containing 37.5% Boswellic Acids. This information was sourced by Emory Healthcare’s website, which also states that some other studies use a high dosage of 1200 mg 3 times daily [57]. The other recommended dosage is 150 mg of Boswellic Acid three times a day [58].
CHAPTER 2
EXPERIMENTAL, RESULTS & DISCUSSION

2.1
Raw materials:

1. Boswellia Serrata Extract; the commercially available product from Science Lab, Texas, USA, was used in all experimentation.

The characteristics and properties of this B.S.E are shown in the table below:

<table>
<thead>
<tr>
<th>Product Name</th>
<th>Boswellia Serrata Powder Extract</th>
</tr>
</thead>
<tbody>
<tr>
<td>GMO Status</td>
<td>GMO Free</td>
</tr>
<tr>
<td>Preservative Status</td>
<td>Free</td>
</tr>
<tr>
<td>Extract Method:</td>
<td>Water &amp; Ethanol Extraction</td>
</tr>
<tr>
<td>Appearance</td>
<td>Brown Fine Powder</td>
</tr>
<tr>
<td>Taste and Odour:</td>
<td>Characteristic</td>
</tr>
<tr>
<td>Mesh Size</td>
<td>100% through 80 Mesh</td>
</tr>
<tr>
<td>Extract Ratio/Active Ingredients</td>
<td>10:1 TLC</td>
</tr>
<tr>
<td>Loss on Drying</td>
<td>3.98% Max (5g/105°C/2hrs)</td>
</tr>
<tr>
<td>Ash</td>
<td>3.33% Max (2g/525°C/3hrs)</td>
</tr>
<tr>
<td>Heavy Metals</td>
<td>10ppm Max (Atomic Absorption)</td>
</tr>
<tr>
<td>Arsenic</td>
<td>2ppm Max (Atomic Absorption)</td>
</tr>
<tr>
<td>Lead</td>
<td>2ppm Max (Atomic Absorption)</td>
</tr>
<tr>
<td>Sterilization method</td>
<td>High Temperature &amp; Pressure</td>
</tr>
<tr>
<td>Total Plate Count</td>
<td>251 cfu/g (AOAC)</td>
</tr>
<tr>
<td>Total Yeast &amp; Mold</td>
<td>19 cfu/g (AOAC)</td>
</tr>
<tr>
<td>E.Coli</td>
<td>Negative (AOAC)</td>
</tr>
<tr>
<td>Salmonella</td>
<td>Negative (AOAC)</td>
</tr>
<tr>
<td>Staphylococcus</td>
<td>Negative (AOAC)</td>
</tr>
</tbody>
</table>

Table 7: Characteristics of B.S.E.
**GMO:** Genetically Modified Organism  
**TLC:** Thin Layer Chromatography  
**ppm:** Parts Per Million  
**cfu/g:** Colony forming unit per gram (for microbiological culture)  
**AOAC:** Association of Analytical Communities

2. Water: De-ionized

3. 100% cotton plain weave bleached apparel fabric.  
   Weight: 189 grams per sq. meter (Appendix I)  
   Ends per inch: 62.5  
   Picks per inch: 48.3
2.2 Application of B.S.E. to 100% cotton fabric

The following series of experiments examine the solubilization or dispersion of B.S.E. and its subsequent application to the 100% cotton bleached fabric.

Cotton was used as the textile substrate for the application of B.S.E. on textiles because:

i. A natural fibre is preferred for fabrics that are in contact with the human skin because of its moisture regain and comfort properties. Cotton, an easily available, most preferred and widely used fibre for garments was chosen over wool, silk and other natural fibres. Other natural fibres were not considered for this specific application due to limited availability, cost and less suitability than cotton for the desired end usage.

ii. Synthetic fibres and blends also have less applicability to this study because they have low “pick-up” capability, hence a reduced capacity to act as a source of B.S.E. when the garment is worn adjacent to the skin [59, 60].

The raw materials used in this series of experiments include:

- 100% Cotton Woven Fabric (bleached) (Specification given in 2.1)
- B.S.E. (Specification given in 2.1)
- Water: De-ionized
- Tubiprint Binder AS 30
2.2.1

**Dissolution of B.S.E.:**

The most common form of application of a substance to a textile material is to pass the fabric in continuous open-width form through a solution of the substance (at an appropriate concentration), after which the textile material is squeezed in a mangle and then dried. This process is commonly referred to as “padding” or “pad drying”.

As this is the first known attempt to apply B.S.E. to textile materials, it was important to use padding as this is the simplest and most widely used process for the application of substances to textile materials.

The basic requirement of padding is a solution of substance that has to be applied on to the fabric using a padding mangle. The raw material used in the experiment was B.S.E. in powder form. Therefore, suitable solvents were explored to make a B.S.E. solution.

The main constituent of B.S.E. is Boswellic Acid. Eyre, Hills and Watkins (2003) suggests that long carbon chain aliphatic compounds are suitable solvent for Boswellic Acid. Therefore, 2-ethyl hexanol was examined as a solvent. However, 2-ethyl hexanol did not form a clear solution because of its apparent inability to dissolve all the compounds present in the B.S.E. used.

Eyre (2003) & Pandey (2005) [69] also indicated that B.S.E. is partially soluble in water and alcohol. Thus the other solvents explored were ethanol and water. It was observed that there was little difference between solutions formed by alcohol and water. In both the solvents, a vigorous stirring was required to form a “solution”. Water formed a homogeneous dispersion, but B.S.E. settled when the solution was left for few hours. The time required for sedimentation depended on the concentration of B.S.E. in the solution. The solution referred here was a dispersion of B.S.E. in water. Also, the term “B.S.E. solution” used in the experiments in Chapter 2.2 refers to the dispersion of B.S.E. in water.

Whilst this instability was not desirable, it was decided to proceed with dispersion in water, with the condition that the materials be padded within 30 minutes of forming the dispersion.
This was done because water is used almost exclusively as the solvent in textile processing and finishing. Furthermore the main objective was to assess the suitability of B.S.E. treated textiles as a means of delivering B.S.E. to human skin.

Two approaches were used for padding. In the first approach the padding was done immediately after forming the dispersion. In the second approach, the padding process was applied after 30 minutes of dispersion formation. This allowed examination of any possible impact of the suitability of the dispersion under industrial application conditions (Note: Typical industrial production rates lie between 30 to 80 m/min. Hence the 0 minute and 30 minutes padding represent the start and end of production runs of 900 to 2400 meters).
2.2.2

Padding of cotton woven fabric with B.S.E. dispersion in water:

2.2.2.1

Standardization of cotton fabric:

To calculate the actual weight gain of the fabric due to padding, the fabric weight and moisture regain was standardized. The method used is explained below.

Each fabric sample was cut into a 10 cm X 10 cm specimen. The fabric sample was then weighed after drying it to zero moisture content in the oven for 1 hour at 104 degree Celsius and dessicating for 30 minutes. The same process was repeated after padding the fabric to calculate actual weight gain. The reason for this process was to minimize the error percentage due to variation of moisture regain in the cotton fabric, which varies directly with the relative humidity of the ambient atmosphere.

The woven cotton fabric was padded with 5%, 10%, 15%, 20%, 25% and 35% B.S.E. dispersion in water.

Each specimen was dipped in the B.S.E. dispersion (for nearly a minute) to completely wet-out the fabric with the B.S.E. dispersion in water. Fabric was then padded using a conventional pad-mangle system. For each fabric sample of 10cm X 10cm, 20 ml of dispersion was prepared.

The amount of B.S.E. added on the fabric was calculated by two methods.

1) **Theoretical Weight Gain:** The theoretical weight gain was calculated by the formula given below:

\[
P = \frac{T \times A}{D}
\]  

(1)
Where:

- \( P \) is the percentage weight gain on the fabric
- \( T \) is the wet pick-up
- \( A \) is the percentage of solution
- \( D \) is the density of the solution (the density of solution was calculated and a correlation is shown in Appendix II between B.S.E. percentage in the solution and density of the solution)

The wet pick-up percentage is defined as the amount of liquor that remains on the fabric after padding. The wet pick-up is dependent on the nip pressure and the area of the nip, modulus and hardness of the roller cover, roller diameter and the speed of the fabric passed through the padding mangle [61, 62] and is calculated from:

\[
\text{Wet pick-up} \% = \frac{(\text{weight of wet sample} - \text{weight of dry sample})}{\text{Weight of dry sample}} \times 100\%
\]  
(2)

In the industry the weight gain percentage of the fabric is calculated by the equation (1) for theoretical weight gain percentage. Ideally the theoretical weight gain equals to actual weight gain (calculating using equation (3)) for finishing process. The basis of using theoretical weight gain in our experiments is to show that when B.S.E. is applied on the textile substrate the theoretical weight gain is not equal to actual weight gain. The explanation of the difference in theoretical and actual percentage weight gain is given in section 2.2.4. This concludes that if a fixed amount of B.S.E. (% weight gain) is required on the textile substrate then the equation given in section 2.2.4 for actual weight gain should be used for bulk production.

2) **Actual Weight Gain**: The other method of calculating the amount of B.S.E. added to the fabric is to determine the actual weight gain. This can be calculated by the formula below:

\[
\text{Actual weight gain} = \frac{\text{final weight of the fabric (dried to zero moisture content) after padding} - \text{weight of the fabric (dried to zero moisture content) before padding}}{\text{Weight of dry sample}} \times 100\%
\]  
(3)
2.2.2.2

Distribution of B.S.E. within Fabric:

B.S.E. was applied to cotton fabric by impregnation with an aqueous solution, squeezed in a mangle followed by removal of the water by drying.

It is known that a low wet pick-up percentage will give a more efficient and homogenous distribution of the finish, but too low wet pick-up gives a non-uniform chemical distribution on the fabric [62]. However, achievement of a uniform distribution of the chemical within the fabric is unattainable by the normal application method of using a dip and nip pad mangle, which is immediately followed by thermal drying [63]. This is because a conventional padding mangle is not able to reduce the pick-up of cotton fabric to the point (approximately 35%) where migration does not occur [63].

A high wet pick-up was chosen for the experimentation for the following reasons:

A high wet pick-up causes migration of chemical to the fabric surface [62]. This migration of liquor increases with increase in wet pick-up [63]. The concentration of the applied chemical on the fabric surface is caused by migration of the chemical towards the heat source (usually double sided) [63].

A high percentage solid add-on of B.S.E. is desired, and a high wet pick-up will result in high percentage solid add-on of B.S.E. on the cotton fabric. A 100 % – 120 % wet pick-up value is not desired because of the high liquor migration during the drying resulting in chemical migration leading to a highly non-uniform finish distribution [59]. Therefore a wet pick-up percentage of 90% was targeted to achieve optimization of the process.

A high wet pick-up value ensures adequate distribution of the B.S.E. in the fabric as the squeezing removes large amount of the surface liquor and also the capillary liquor in the interstitial spaces between the yarns [62].

More importantly, the outcome that is desired from this experimentation requires the B.S.E. to be predominantly on the surface of the fabric so that there is higher availability of the chemical compound to be transferred from the fabric surface and therefore onto human skin.
Fabric Treatment with B.S.E.:

The following tables and charts show the initial dried weight (weight before padding) and final dried weight (weight after padding) of the cotton fabric specimen. Wet take-up percentage is also included. The chart calculates the amount of material present on the fabric.

**Padding of cotton woven fabric with 5% B.S.E. powder dispersion in water:**

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>(W_{\text{initial}})</th>
<th>(W_{\text{after padding}})</th>
<th>Wet take-up %</th>
<th>(W_{\text{final}})</th>
<th>Actual weight gain percentage</th>
<th>Theoretical weight gain percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>01</td>
<td>1.80</td>
<td>3.41</td>
<td>89.4%</td>
<td>1.86</td>
<td>3.3%</td>
<td>4.7%</td>
</tr>
<tr>
<td>02</td>
<td>1.79</td>
<td>3.40</td>
<td>89.9%</td>
<td>1.87</td>
<td>4.5%</td>
<td>4.7%</td>
</tr>
<tr>
<td>03</td>
<td>1.81</td>
<td>3.44</td>
<td>90.1%</td>
<td>1.88</td>
<td>3.9%</td>
<td>4.7%</td>
</tr>
<tr>
<td>04</td>
<td>1.81</td>
<td>3.45</td>
<td>90.6%</td>
<td>1.87</td>
<td>3.3%</td>
<td>4.7%</td>
</tr>
<tr>
<td>05</td>
<td>1.82</td>
<td>3.44</td>
<td>89.0%</td>
<td>1.88</td>
<td>3.3%</td>
<td>4.6%</td>
</tr>
</tbody>
</table>

Dry weight of sample before padding (in grams) = \(W_{\text{initial}}\)

Weight of sample after padding (in grams) = \(W_{\text{after padding}}\)

Weight of dried sample after padding (in grams) = \(W_{\text{final}}\)
Comparison of theoretical weight gain to actual weight gain on Woven fabric after padding

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>weight gain percentage</th>
<th>theoretical weight gain percentage</th>
<th>ratio actual to theoretical</th>
</tr>
</thead>
<tbody>
<tr>
<td>01</td>
<td>3.3%</td>
<td>4.7%</td>
<td>0.70</td>
</tr>
<tr>
<td>02</td>
<td>4.5%</td>
<td>4.7%</td>
<td>0.96</td>
</tr>
<tr>
<td>03</td>
<td>3.9%</td>
<td>4.7%</td>
<td>0.83</td>
</tr>
<tr>
<td>04</td>
<td>3.3%</td>
<td>4.7%</td>
<td>0.70</td>
</tr>
<tr>
<td>05</td>
<td>3.3%</td>
<td>4.6%</td>
<td>0.72</td>
</tr>
</tbody>
</table>

Samples 1, 2 were soaked in B.S.E. 5% dispersion for 30 minutes before padding the fabric on the conventional padding mangle. Samples 3, 4 & 5 were padded with zero idle time for the fabric in the B.S.E. solution.

The reason for the variation in weight gain percentage is the procedure followed for padding of B.S.E. on woven cotton fabric. The two different procedures followed include the pre-soak method (pre-soaking for 30 minutes) and non-presock method. These methods are discussed later in Section 2.2.2.3 of the report. Also, Section 2.2.2.4 discusses the reasons for differences in theoretical and actual weight gain.
Padding of Cotton woven fabric with 10% B.S.E. powder dispersion in water:

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>W\text{ initial}</th>
<th>W\text{ after padding}</th>
<th>wet take-up %</th>
<th>W\text{ final}</th>
<th>Actual weight gain percentage</th>
<th>Theoretical weight gain percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>06</td>
<td>1.81</td>
<td>3.44</td>
<td>90.1%</td>
<td>1.96</td>
<td>8.3%</td>
<td>9.2%</td>
</tr>
<tr>
<td>07</td>
<td>1.82</td>
<td>3.39</td>
<td>86.3%</td>
<td>1.93</td>
<td>6.0%</td>
<td>8.8%</td>
</tr>
<tr>
<td>08</td>
<td>1.85</td>
<td>3.47</td>
<td>87.6%</td>
<td>1.97</td>
<td>6.5%</td>
<td>9.0%</td>
</tr>
<tr>
<td>09</td>
<td>1.84</td>
<td>3.49</td>
<td>89.7%</td>
<td>1.98</td>
<td>7.6%</td>
<td>9.2%</td>
</tr>
<tr>
<td>10</td>
<td>1.83</td>
<td>3.49</td>
<td>90.7%</td>
<td>1.97</td>
<td>7.7%</td>
<td>9.3%</td>
</tr>
<tr>
<td>11</td>
<td>1.84</td>
<td>3.51</td>
<td>90.8%</td>
<td>1.99</td>
<td>8.2%</td>
<td>9.3%</td>
</tr>
<tr>
<td>12</td>
<td>1.81</td>
<td>3.45</td>
<td>90.6%</td>
<td>1.94</td>
<td>7.2%</td>
<td>9.3%</td>
</tr>
<tr>
<td>13</td>
<td>1.8</td>
<td>3.43</td>
<td>90.6%</td>
<td>1.92</td>
<td>6.7%</td>
<td>9.3%</td>
</tr>
<tr>
<td>14</td>
<td>1.79</td>
<td>3.41</td>
<td>90.5%</td>
<td>1.93</td>
<td>7.8%</td>
<td>9.2%</td>
</tr>
</tbody>
</table>

Dry weight of sample before padding (in grams) = W\text{ initial}

Weight of sample after padding (in grams) = W\text{ after padding}

Weight of dried sample after padding (in grams) = W\text{ final}

Comparison of Theoretical Weight Gain to Actual Weight Gain on Woven Fabric after Padding
<table>
<thead>
<tr>
<th>Sample No.</th>
<th>weight gain percentage</th>
<th>theoretical weight gain percentage</th>
<th>ratio actual to theoretical</th>
</tr>
</thead>
<tbody>
<tr>
<td>06</td>
<td>8.3%</td>
<td>9.2%</td>
<td>0.90</td>
</tr>
<tr>
<td>07</td>
<td>6.0%</td>
<td>8.8%</td>
<td>0.68</td>
</tr>
<tr>
<td>08</td>
<td>6.5%</td>
<td>9.0%</td>
<td>0.72</td>
</tr>
<tr>
<td>09</td>
<td>7.6%</td>
<td>9.2%</td>
<td>0.82</td>
</tr>
<tr>
<td>10</td>
<td>7.7%</td>
<td>9.3%</td>
<td>0.83</td>
</tr>
<tr>
<td>11</td>
<td>8.2%</td>
<td>9.3%</td>
<td>0.88</td>
</tr>
<tr>
<td>12</td>
<td>7.2%</td>
<td>9.3%</td>
<td>0.77</td>
</tr>
<tr>
<td>13</td>
<td>6.7%</td>
<td>9.3%</td>
<td>0.72</td>
</tr>
<tr>
<td>14</td>
<td>7.8%</td>
<td>9.2%</td>
<td>0.85</td>
</tr>
</tbody>
</table>

It can be seen here that the variation in the first three readings (Samples 6, 7 & 8) is more than 9, 10 & 11. The first three fabric samples (6, 7 & 8) were soaked in the B.S.E. solution for 30 minutes, whereas samples 09, 10, 11, 12, 13 & 14 were instantly padded with B.S.E. giving no time for soaking of the fabric samples in B.S.E. solution. In case of fabric sample 12, 13 & 14, an acrylic binder (Tubiprint Binder AS 30 – 4 g/l) was added. This was in attempt to improve the washing fastness of the B.S.E. applied. The results are discussed in Section 2.2.2.6.
Padding of Cotton woven fabric with 15% B.S.E. powder dispersion in water:

Dry weight of sample before padding (in grams) = \( W_{\text{initial}} \)
Weight of sample after padding (in grams) = \( W_{\text{after padding}} \)
Weight of dried sample after padding (in grams) = \( W_{\text{final}} \)

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>( W_{\text{initial}} )</th>
<th>( W_{\text{after padding}} )</th>
<th>wet take-up %</th>
<th>( W_{\text{final}} )</th>
<th>Weight gain percentage</th>
<th>Theoretical weight gain percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>1.77</td>
<td>3.38</td>
<td>91.0%</td>
<td>1.96</td>
<td>10.7%</td>
<td>13.6%</td>
</tr>
<tr>
<td>16</td>
<td>1.80</td>
<td>3.41</td>
<td>89.0%</td>
<td>1.98</td>
<td>10.0%</td>
<td>13.3%</td>
</tr>
<tr>
<td>17</td>
<td>1.81</td>
<td>3.44</td>
<td>90.1%</td>
<td>2.01</td>
<td>11.0%</td>
<td>13.5%</td>
</tr>
<tr>
<td>18</td>
<td>1.82</td>
<td>3.45</td>
<td>89.6%</td>
<td>2.01</td>
<td>10.4%</td>
<td>13.4%</td>
</tr>
<tr>
<td>19</td>
<td>1.85</td>
<td>3.52</td>
<td>90.3%</td>
<td>2.05</td>
<td>10.8%</td>
<td>13.5%</td>
</tr>
<tr>
<td>20</td>
<td>1.82</td>
<td>3.47</td>
<td>90.7%</td>
<td>2.02</td>
<td>11.0%</td>
<td>13.6%</td>
</tr>
<tr>
<td>21</td>
<td>1.85</td>
<td>3.49</td>
<td>88.7%</td>
<td>2.04</td>
<td>10.3%</td>
<td>13.3%</td>
</tr>
<tr>
<td>22</td>
<td>1.86</td>
<td>3.48</td>
<td>87.1%</td>
<td>2.05</td>
<td>10.2%</td>
<td>13.1%</td>
</tr>
<tr>
<td>23</td>
<td>1.87</td>
<td>3.57</td>
<td>90.9%</td>
<td>2.06</td>
<td>10.2%</td>
<td>13.6%</td>
</tr>
</tbody>
</table>

Comparison of Actual to Theoretical Weight Gain in 15% BSE Padding on Woven Cotton Fabric
The padding process used was same for the above fabric samples, but a variation in drying temperature was used in sample 18, 19 & 20. Fabric sample 18 was kept at a temperature of 104°C in the oven whereas sample 19 was kept at 80°C, and sample 20 at 50°C in the oven for drying. All the fabric samples were transferred from the oven to the desiccator where they were kept for 30 minutes and then weighed. This process was followed to evaluate the effect of the drying temperature on actual weight gain on the fabric. It can be deduced from the graphs and readings in the table given above, that there is variation in actual weight gain between the specimens (fabric) dried at a lower temperature after padding. Fabric samples padded and dried by the standard process followed. The average weight gain for fabric samples dried at 104°C is 10.4%, whereas the fabric sample dried at 80°C has a weight gain of 10.8%. The fabric sample dried at 50°C had a weight gain of 11.0%. Therefore it can be concluded that the drying temperature has a direct effect on weight gain of the fabric after padding with B.S.E. dispersion when the fabric was kept in the desiccator for 30 minutes. The difference in weight gain due to temperature is attributed to volatile substances present in B.S.E. powder (refer to Section 2.2.2.4). Fabric samples 15, 16 & 17 were soaked in the B.S.E. dispersion for 30 minutes.
Padding of Cotton woven fabric with 25% B.S.E. powder dispersion in water:

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>$W_{\text{initial}}$</th>
<th>$W_{\text{after padding}}$</th>
<th>Wet take-up %</th>
<th>$W_{\text{final}}$</th>
<th>Actual weight gain percentage</th>
<th>Theoretical weight gain percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>24</td>
<td>1.84</td>
<td>3.51</td>
<td>90.8%</td>
<td>2.12</td>
<td>15.2%</td>
<td>21.5%</td>
</tr>
<tr>
<td>25</td>
<td>1.85</td>
<td>3.51</td>
<td>89.7%</td>
<td>2.14</td>
<td>15.7%</td>
<td>21.3%</td>
</tr>
<tr>
<td>26</td>
<td>1.83</td>
<td>3.47</td>
<td>89.6%</td>
<td>2.15</td>
<td>17.5%</td>
<td>21.2%</td>
</tr>
<tr>
<td>27</td>
<td>1.81</td>
<td>3.47</td>
<td>91.7%</td>
<td>2.12</td>
<td>17.1%</td>
<td>21.7%</td>
</tr>
<tr>
<td>28</td>
<td>1.86</td>
<td>3.53</td>
<td>89.8%</td>
<td>2.14</td>
<td>15.1%</td>
<td>21.2%</td>
</tr>
<tr>
<td>29</td>
<td>1.81</td>
<td>3.45</td>
<td>90.6%</td>
<td>2.12</td>
<td>17.1%</td>
<td>21.4%</td>
</tr>
</tbody>
</table>

Dry weight of sample before padding (in grams) = $W_{\text{initial}}$

Weight of sample after padding (in grams) = $W_{\text{after padding}}$

Weight of dried sample after padding (in grams) = $W_{\text{final}}$

![Comparison of Actual to theoretical Weight Gain in 25% BSE Padding on Cotton Woven Fabric](chart.png)
<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Actual weight gain percentage</th>
<th>theoretical weight gain percentage</th>
<th>ratio actual to theoretical</th>
</tr>
</thead>
<tbody>
<tr>
<td>24</td>
<td>15.2%</td>
<td>21.5%</td>
<td>0.71</td>
</tr>
<tr>
<td>25</td>
<td>15.7%</td>
<td>21.3%</td>
<td>0.74</td>
</tr>
<tr>
<td>26</td>
<td>17.5%</td>
<td>21.2%</td>
<td>0.83</td>
</tr>
<tr>
<td>27</td>
<td>17.1%</td>
<td>21.7%</td>
<td>0.79</td>
</tr>
<tr>
<td>28</td>
<td>15.1%</td>
<td>21.2%</td>
<td>0.71</td>
</tr>
<tr>
<td>29</td>
<td>17.1%</td>
<td>21.4%</td>
<td>0.80</td>
</tr>
</tbody>
</table>

Fabric samples 24, 25 & 26 were soaked in B.S.E. 25% dispersion for 30 minutes before padding the fabric samples on the conventional padding mangle. The ratio of theoretical to actual weight gain in these fabric samples ranged from 0.71 to 0.83. Fabric samples 27, 28 & 29 had zero idle time in the B.S.E. dispersion in water before padding. The ratio of actual to theoretical weight gain in the fabric samples ranged from 0.71 to 0.80.
Padding of Cotton woven fabric with 35% B.S.E. powder dispersion in water:

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>W&lt;sub&gt;initial&lt;/sub&gt;</th>
<th>W&lt;sub&gt;after padding&lt;/sub&gt;</th>
<th>wet take-up %</th>
<th>W&lt;sub&gt;final&lt;/sub&gt;</th>
<th>Actual weight gain percentage</th>
<th>Theoretical weight gain percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>1.83</td>
<td>3.58</td>
<td>95.6%</td>
<td>2.22</td>
<td>21.3%</td>
<td>29.9%</td>
</tr>
<tr>
<td>31</td>
<td>1.80</td>
<td>3.51</td>
<td>95.0%</td>
<td>2.16</td>
<td>20.0%</td>
<td>29.7%</td>
</tr>
<tr>
<td>32</td>
<td>1.84</td>
<td>3.56</td>
<td>93.5%</td>
<td>2.20</td>
<td>19.6%</td>
<td>29.2%</td>
</tr>
<tr>
<td>33</td>
<td>1.83</td>
<td>3.51</td>
<td>91.8%</td>
<td>2.22</td>
<td>21.3%</td>
<td>28.7%</td>
</tr>
<tr>
<td>34</td>
<td>1.86</td>
<td>3.54</td>
<td>90.3%</td>
<td>2.23</td>
<td>19.9%</td>
<td>28.2%</td>
</tr>
<tr>
<td>35</td>
<td>1.88</td>
<td>3.59</td>
<td>91.0%</td>
<td>2.24</td>
<td>19.1%</td>
<td>28.4%</td>
</tr>
</tbody>
</table>

Dry weight of sample before padding (in grams) = W<sub>initial</sub>

Weight of sample after padding (in grams) = W<sub>after padding</sub>

Weight of dried sample after padding (in grams) = W<sub>final</sub>

Comparison of Actual to Theoretical Weight Gain in 35% BSE Padding on Woven Cotton Fabric

![Comparison of Actual to Theoretical Weight Gain](image-url)
<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Actual weight gain percentage</th>
<th>theoretical weight gain percentage</th>
<th>ratio actual to theoretical</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>21.3%</td>
<td>29.9%</td>
<td>0.71</td>
</tr>
<tr>
<td>31</td>
<td>20.0%</td>
<td>29.7%</td>
<td>0.67</td>
</tr>
<tr>
<td>32</td>
<td>19.6%</td>
<td>29.2%</td>
<td>0.67</td>
</tr>
<tr>
<td>33</td>
<td>21.3%</td>
<td>28.7%</td>
<td>0.74</td>
</tr>
<tr>
<td>34</td>
<td>19.9%</td>
<td>28.2%</td>
<td>0.71</td>
</tr>
<tr>
<td>35</td>
<td>19.1%</td>
<td>28.4%</td>
<td>0.67</td>
</tr>
</tbody>
</table>

Fabric samples 33, 34 & 35 were soaked in B.S.E. at 35% dispersion in water for 30 minutes before padding. Fabric samples 30, 31 & 32 had zero idle time in the B.S.E. dispersion before padding. The padding process is difficult for 35% B.S.E. dispersion as there is a high viscosity of B.S.E. powder in the dispersion that restrains the free flow. It can be deduced from the low actual weight gain to theoretical weight gain ratio, that any higher percentage solution than this (35%), will result in low actual to theoretical weight gain. The high value of viscosity (appendix II) also confirms that any higher percentage dispersion will cause problems in padding process. Therefore it can be recommended that for a higher deposit of B.S.E. on the fabric, alternative processes like coating should be followed.
Experimental Analysis - I

An analysis was carried out to examine the reasons for the difference in results when the fabric was padded immediately after wetting or soaked in B.S.E. solution for 30 minutes. It is suggested that the difference in weight gain percentage could be due to:

- Sedimentation of B.S.E. in 30 minutes which was not visually observed,
- Density and concentration variation within the B.S.E. solution,
- Change from homogeneous (0 minutes) to heterogeneous (30 minutes) dispersed solution of B.S.E.,
- Affinity of compounds present in B.S.E. for cotton fabric when cotton fabric is soaked in B.S.E. solution for 30 minutes

A series of experiments were carried out in order to verify if one or all of the above given reasons were responsible for the difference in weight gain percentage between the two processes followed.

The following graphs shows the effect of 0 minutes dwell time and 30 minutes dwell time, taking the average of readings for each percentage of B.S.E. dispersion in water.

The first graph given below shows the relationship between percentage weight-gain to the percentage of B.S.E. in the solution. This is for the process when fabric is soaked for zero minutes in the solution before the fabric is padded with B.S.E.
The second graph (on the following page) shows the relationship between percentage weight-gain to the percentage of B.S.E. in the solution for the process where the fabric was soaked for 30 minutes in the solution before fabric sample was padded with B.S.E.
It can be seen that both mentioned graphs follow a polynomial equation.

The stability of the dispersed B.S.E. solution was examined by the following experiment:

**Methodology:**

A black dyed cotton woven fabric was dipped into a freshly prepared 15% B.S.E. solution. It was then immediately oven dried at 104°C.

**Analysis & Conclusion:**

The described methodology was followed to determine whether there was a visual presence of dry B.S.E. powder on the surface of oven dried fabrics. After visual examination of the dried fabric, it was concluded that there was no B.S.E. present on the surface of the cotton fabric in the form of powder, suggesting the relative stability of the freshly dispersed B.S.E. solution.
To examine if there was any deposition or affinity of compounds present in B.S.E. for cotton when the fabric is soaked in B.S.E. solution, another set of experiments were carried out as follows:

**Methodology:**

Cotton fabric specimen were padded with 15% B.S.E. solution after soaking the fabric in B.S.E. solution for 0 min, 30 min, 60 min, 2.5 hrs and 3 hrs.

<table>
<thead>
<tr>
<th>Fabric Sample No.</th>
<th>Time for soaking in B.S.E. solution</th>
<th>W_{initial}</th>
<th>W_{after padding}</th>
<th>wet take-up %</th>
<th>W_{final}</th>
<th>Actual weight gain percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0.0 hrs</td>
<td>1.87</td>
<td>3.57</td>
<td>90.9</td>
<td>2.06</td>
<td>10.2</td>
</tr>
<tr>
<td>B</td>
<td>0.5 hrs</td>
<td>1.80</td>
<td>3.41</td>
<td>89.4</td>
<td>1.98</td>
<td>10.0</td>
</tr>
<tr>
<td>C</td>
<td>1.0 hrs</td>
<td>1.83</td>
<td>3.45</td>
<td>88.5</td>
<td>2.01</td>
<td>9.8</td>
</tr>
<tr>
<td>D</td>
<td>2.5 hrs</td>
<td>1.82</td>
<td>3.48</td>
<td>91.2</td>
<td>1.99</td>
<td>9.4</td>
</tr>
<tr>
<td>E</td>
<td>3.0 hrs</td>
<td>1.83</td>
<td>3.47</td>
<td>89.6</td>
<td>2.00</td>
<td>9.3</td>
</tr>
</tbody>
</table>

The graph above shows that there is a negative affinity of B.S.E. that increases with the time of fabric soaked in B.S.E. dispersion. Therefore the affinity is such that the repulsion is linear with time.
2.2.4

Experimental Analysis - II

The following graphs show the difference in the theoretical and actual weight gain in the fabric when treated with B.S.E. solution.

The first graph takes into consideration all the readings taken for different percentage of B.S.E. by both methods applied (0 minutes soaking time and 30 minutes soaking time):

![Comparison of Theoretical to actual weight gain of Fabric](chart.png)

**Figure 8**

The graph above shows that the difference between theoretical and actual weight gain of fabric increases with the increase in percentage of B.S.E. in the solution.

The lower values of actual weight gain, relative to the theoretical calculation, indicate that the following reasons may cause the difference between theoretical and actual values of weight gain.

- A component or components in the B.S.E. which are volatile under the drying conditions used (1 hour drying at 104°C), and
• Negative affinity (repulsion) from the cellulosic fabric to B.S.E.

An experiment was performed to evaluate the effect of temperature on any weight loss due to volatile substances present in B.S.E. Samples of B.S.E. powder (5 grams) were oven dried at 104°C for one, two and three hour time period. The B.S.E. powder was weighed at one hour intervals. During the three hour oven dry heating of B.S.E., establishing the variation in weight loss when B.S.E. powder is exposed at higher temperature (104°C). The table below shows the weight loss after every hour for three hours.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Weight Loss (%) 0 hours</th>
<th>Weight Loss (%) 1 hour</th>
<th>Weight Loss (%) 2 hours</th>
<th>Weight Loss (%) 3 hours</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>0</td>
<td>8.4</td>
<td>8.8</td>
<td>8.8</td>
</tr>
<tr>
<td>Sample 2</td>
<td>0</td>
<td>8.8</td>
<td>9.0</td>
<td>9.0</td>
</tr>
<tr>
<td>Sample 3</td>
<td>0</td>
<td>8.4</td>
<td>8.4</td>
<td>8.6</td>
</tr>
<tr>
<td>Average</td>
<td>0</td>
<td><strong>8.5</strong></td>
<td><strong>8.7</strong></td>
<td><strong>8.8</strong></td>
</tr>
</tbody>
</table>

It can be concluded from the above table (table 8) that because of the presence of volatile substances in the B.S.E. powder, there is a loss in weight when B.S.E. powder is exposed at high temperature. The average weight loss when B.S.E. powder is oven dry heated at 104°C for one hour is 8.5%, whereas for two hours, it is 8.7% and when heated for three hours it is 8.8%. Therefore it can be inferred from the above data that the presence of volatile substances in B.S.E. powder is part of the reason for the difference in actual to theoretical weight gain in fabric sample padded with B.S.E. The therapeutic property of the finished fabric is not affected by the drying temperature used in this research, as the melting point of boswellic acids present in Boswellia Serrata Extract ranges from 195°C to 274°C.

The same procedure was followed for oven dry heating temperature of 50°C & 80°C.
The following tables (table 9 & 10) show the percentage of weight loss and weight when the B.S.E. powder was oven dry heated at 50°C and 80°C for one, two and three hour intervals:

**Table 9**

<table>
<thead>
<tr>
<th>Sample 1</th>
<th>Weight Loss (% 0 hours)</th>
<th>Weight Loss (% 1 hours)</th>
<th>Weight Loss (% 2 hours)</th>
<th>Weight Loss (% 3 hours)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>0</td>
<td>3.4</td>
<td>3.6</td>
<td>3.8</td>
</tr>
<tr>
<td>Sample 2</td>
<td>0</td>
<td>3.0</td>
<td>3.8</td>
<td>3.8</td>
</tr>
<tr>
<td>Average</td>
<td>0</td>
<td>3.2</td>
<td>3.7</td>
<td>3.8</td>
</tr>
</tbody>
</table>

**Table 10**

<table>
<thead>
<tr>
<th>Sample 1</th>
<th>Weight Loss (% 0 hours)</th>
<th>Weight Loss (% 1 hours)</th>
<th>Weight Loss (% 2 hours)</th>
<th>Weight Loss (% 3 hours)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>0</td>
<td>6.6</td>
<td>7.2</td>
<td>7.4</td>
</tr>
<tr>
<td>Sample 2</td>
<td>0</td>
<td>7.2</td>
<td>7.6</td>
<td>7.6</td>
</tr>
<tr>
<td>Average</td>
<td>0</td>
<td>6.9</td>
<td>7.4</td>
<td>7.5</td>
</tr>
</tbody>
</table>

The average weight loss when B.S.E. powder is oven dry heated at 50°C for one hour is 3.2%, whereas for two hours, it is 3.7% and when heated for three hours it is 3.8%. Whereas the average weight loss when B.S.E. powder is oven dry heated at 80°C for one hour is 6.9%, whereas for two hours, it is 7.4%, and when heated for three hours it is 7.5%.

It can be inferred from this that the different volatile substances present in B.S.E. powder are evaporated at different temperatures and therefore the drying temperature is important for the process followed.

The negative affinity (repulsion) of the cellulosic fabric is confirmed in experiment given in 2.2.2.3.

The following graph shows a correlation between weight gain in fabric to loss of weight in B.S.E. powder at 50°C, 80°C and 110°C.
It can be concluded from the above graph that:

Temperature $\alpha$ weight loss of B.S.E. powder, and

Temperature $\alpha$ $1/\text{weight gain of cotton cellulosic fabric when padded with B.S.E.}$

This correlation is valid when the drying temperature ranges between 50°C and 110°C.
Rubbing Fastness:

Samples of fabric previously treated with B.S.E. to a range of levels were subjected to rub fastness testing using a crock-meter (A.A.T.C.C. Crock-meter, Atlas Electronic Device Co., USA). The tests were carried out by rubbing under wet and dry conditions. The transferrance of B.S.E. was measured by the change in colour of adjacent cotton fabric after rubbing and was relatively graded. The lower the value rated, the higher the presence of B.S.E. on the adjacent cotton fabric. (Note: B.S.E. treated fabrics are a muddy yellow colour).

This was done to provide an indication of the presence of B.S.E. on the surface of the padded fabric and its relative ability to be transferred to an opposing fabric. The possibility of B.S.E. transferring from the fabric to the surface of the skin (B.S.E. on the surface of the skin is for the availability for the absorption of B.S.E. by the skin) is high when the rubbing fastness values are low.

<table>
<thead>
<tr>
<th>% B.S.E. applied*</th>
<th>Dry Rubbing</th>
<th>Wet Rubbing</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 %</td>
<td>4 – 5</td>
<td>3 – 4</td>
</tr>
<tr>
<td>10 %</td>
<td>4 – 5</td>
<td>3 – 4</td>
</tr>
<tr>
<td>10 % + 4 g/l acrylic binder</td>
<td>4 – 5</td>
<td>3 – 4</td>
</tr>
<tr>
<td>15 %</td>
<td>4 – 5</td>
<td>3</td>
</tr>
<tr>
<td>25 %</td>
<td>4 – 5</td>
<td>2 – 3</td>
</tr>
<tr>
<td>35 %</td>
<td>4 – 5</td>
<td>2 – 3</td>
</tr>
</tbody>
</table>

B.S.E. applied* as a dispersion of B.S.E. powder in water

The above table of results shows that under dry conditions (20°C, 65% RH) there is only a minimum transfer of B.S.E. from the treated fabric to the adjacent cotton fabric. The transfer however is increased if the adjacent fabric is moist; therefore transfer under these conditions increased at B.S.E. concentrations levels from 15% on weight of the fabric.
2.2.6

Washing Fastness:

The padded fabric samples were assessed for wash fastness of B.S.E. A test that closely approximates one domestic laundering was carried out for the fabric samples. ECE Reference Detergent was used in the test. The procedure followed for the test was ISO 105-C06:1994(E) (A1S) with a modification in test specimen preparation, liquor volume and absence of steel balls in the process. The padded sample of size 100mm X 100 mm was added to 100 ml of detergent solution (4 g/l). The samples were washed at 40°C for 30 minutes. After washing, specimens were rinsed twice at 40°C in warm water. The samples were then dried at 50 - 60°C.

The washed fabric samples were then oven dried at 104°C for 60 minutes and kept in desiccators for 30 minutes. The samples were then weighed. This was done to measure the percentage of B.S.E. retained in the fabric sample after washing, and can be seen in the table below:

<table>
<thead>
<tr>
<th>Percentage B.S.E. Dispersion in water</th>
<th>Percentage weight gain on the fabric after padding</th>
<th>Percentage weight gain on the padded fabric afterwashing</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 %</td>
<td>3.3%</td>
<td>1.7%</td>
</tr>
<tr>
<td>10 %</td>
<td>6.1%</td>
<td>2.0%</td>
</tr>
<tr>
<td>10 % + 4 g/l acrylic binder</td>
<td>7.8%</td>
<td>2.2%</td>
</tr>
<tr>
<td>15 %</td>
<td>10.8%</td>
<td>2.3%</td>
</tr>
<tr>
<td>25 %</td>
<td>21.7%</td>
<td>2.7%</td>
</tr>
<tr>
<td>35 %</td>
<td>20.0%</td>
<td>2.2%</td>
</tr>
</tbody>
</table>

These results show that B.S.E. on fabric has a relatively low fastness to washing. The table also shows that the presence of acrylic binder to the B.S.E. did not improve the wash fastness. It can also be seen in the table that the retention of B.S.E. on fabric ranges from 1.7% - 2.7%, irrespective of the percentage of B.S.E. in the dispersion.
2.2.7

Assessment of the transfer of B.S.E. from treated fabric to simulated skin:

A Martindale abrasion testing apparatus was used to assess whether B.S.E. could be transferred from the treated fabric surface to a “simulated skin” (Appendix III).

The Martindale abrasion machine causes a controlled amount of multidirectional abrasion between two fabric surfaces at comparatively low loads. In this experiment the conventional standard abradant fabric was replaced with a natural leather to simulate human skin.

Methodology:

In this test a circular specimen of B.S.E. treated fabric was subjected to a defined load of nearly 200 grams (the weight of the specimen holder in the Martindale abrasion machine) and rubbed against the natural leather in a translational movement tracing a Lissajous figure. The specimen holder is freely rotatable around its axis perpendicular to the horizontal plane. The B.S.E. treated fabric was subjected to abrasive wear for 10,000 rub cycles.

The initial weight of the specimen (woven fabric treated with B.S.E.) and the abradant (natural leather) was recorded to three decimal points. After 10,000 rubbing cycles the fabric and leather were weighed again. Unlike in the abrasion test, no extra weights were added to the specimen as it was assumed that the fabric rubs with the human body every 4 seconds and each rubbing cycle signifies the fabric rubbing with human body. Therefore, it was assumed that when the fabric is worn for a period of 12 hours, it rubs 10,000 times with the human body.

There were two different approaches followed for this method. In the first approach, the moisture regain of the fabric specimen was not altered. Whereas in the second procedure, the B.S.E. treated fabric specimen had high moisture content (water was sprayed on the fabric specimen).
Conclusion:

When the sample was dry, there was no evidence that the B.S.E. transferred from the fabric to the leather surface. Wetting the B.S.E. treated fabric did not improve the transfer of B.S.E. There was a minimal amount of weight loss observed in the fabric specimen.

Notes to Abrasion Test:

*Abrasion rub:* one revolution of the two outer drives of the Martindale abrasion tester.

*Abrasion cycle:* completion of all the translational abrasion movements tracing a Lissajous figure comprising 16 rubs.

*Inspection interval:* number of continuously performed rubs.

*Lissajous figure:* figure created by the movement which changes from a circle to gradually narrowing ellipses, until it becomes a straight line, from which progressively widening ellipses develop, in a diagonally opposite direction before the pattern is repeated.
2.2.8

Electron Microscopy:

Electron Microscopy was used to confirm the presence of

- B.S.E. on Surface of fabric
- B.S.E. on the fibre surface

![Figure 10: Electron Micrographs at 100X at 0, 5, 15 and 25% B.S.E.](image)

Figure 10: Electron Micrographs at 100X at 0, 5, 15 and 25% B.S.E.

The above micrographs show the scanning electron microscopic view at 100X with increasing of B.S.E. in the dispersed solution used for padding (0, 5, 15 and 25% B.S.E.). The
figures show the presence of B.S.E. on the fabric surface as well as trapped between the fibres within the yarn.

**Figure 11: Electron Micrographs at 400X at 0, 5, 15 and 25% B.S.E.**

The above micrographs (now at 400X) confirm the presence of B.S.E. on the surface of the fabric as well as lying between the fibres in the yarns. The B.S.E. is most visible in cotton sample padded with 15% B.S.E. dispersed solution and also in 25% B.S.E. dispersed solution.
Increasing the magnification to 1000X clearly demonstrates the presence of B.S.E. on and within the fabric. This is further confirmed in the next two figures with a magnification of 2000 times and 5000 times.

**Figure 12: Electron Micrographs at 1000X at 0, 5, 15 and 25% B.S.E.**
Figure 13: Electron Micrographs at 2000X at 0, 5, 15 and 25% B.S.E.
Figure 14: Electron Micrographs at 5000X at 0, 5, 15 and 25% B.S.E.
2.2.9

**Fourier Transform Infrared Spectrophotometer (FTIR)**

Perkin Elmer Spectrum 2000 Fourier Transform spectrophotometer was used to characterize the B.S.E. powder and the woven cotton fabric treated with different concentrations of B.S.E.

B.S.E. powder sample was ground up (made into powder) then analysed with potassium bromide as micropellet (microgram quantities of sample with a 20-fold excess of anhydrous KBr). The untreated and finished cotton fabric (padded with 5, 10, 15 and 25% B.S.E. solution) was analysed by Horizontal ATR technology using Zn Se crystals.

![Figure 15: Commercial B.S.E. powder from Science Lab, Texas, USA](image)
Figure 16: 100% Cotton untreated woven fabric
Figure 17: 100% Cotton fabric padded with 5% B.S.E. Solution
Figure 18: 100% Cotton fabric padded with 15% B.S.E. Solution
Figure 19: 100% Cotton fabric padded with 25% B.S.E. Solution
Figure 20: Normalized IR of 100% Cotton fabric padded with 0%, 5%, 15% & 25% B.S.E. Solution
The IR Spectra of cotton fabric padded with concentrations of B.S.E. are shown in figures 16, 17, 18, 19 & 20.

The IR Spectrum for 25% B.S.E. padded cotton fabric is shown in figures 19 & 20. Presence of alkanes is confirmed with absorption between 2850 and 960 cm\(^{-1}\) corresponds to CH stretching. The strong multiple peaks between 675 and 1000 cm\(^{-1}\) confirms the presence of alkenes corresponding to bending. The CH absorption between 2960 and 2850 cm\(^{-1}\) is due to aliphatic hydrogens.

The presence of CH bond in phenyl ring substitution overtones is evident by the weak peaks (in fingerprint region) 1616, 1686, 1706, 1718 & 1738 cm\(^{-1}\). The peaks 1350, 1372 & 1422 cm\(^{-1}\) also confirms scissoring and bending of CH bond in alkenes.

The carboxylic acid group is confirmed by the peaks 2850 cm\(^{-1}\) and 2918 cm\(^{-1}\) showing a \(-\text{OH}\) bond; 1706 cm\(^{-1}\) and 1718 cm\(^{-1}\) showing a C=O bond and 1022, 1052, 1110 & 1158 cm\(^{-1}\) showing a presence of C–O bond.

The presence of amines is shown by 1616 cm\(^{-1}\) for a N–H bond and 1022, 1052, 1110, 1158, 1278 and 1340 cm\(^{-1}\) showing a C–N bond.

In figure 20, it can be seen that the intensity of peak increases between 2810 cm\(^{-1}\) to 2960 cm\(^{-1}\) and also between 1200cm\(^{-1}\) and 1650 cm\(^{-1}\) due to application of B.S.E. on the cotton fabric.
2.3

**Application of B.S.E. to non-woven fabric**

In the previous sections it has been shown that even though B.S.E. can be applied to woven apparel fabrics, there was no evidence for transfer to a simulated skin when there was minimum load applied during rubbing of adjacent fabrics.

Non-wovens are the most widely used textile material for medical textiles. Non-wovens are low cost textile materials and therefore find widespread use in disposable products. The purpose of using a non-woven was to apply higher quantity of B.S.E. on the surface of the fabric that can be used as disposable patches. In order to apply as much B.S.E. to the fabric surface as possible the fabrics were ‘coated’, on one side rather than using padding.
2.3.1

Fibre identification & standardization of non-woven fabric:

A commercial non-woven fabric similar to fabrics used for food filtering or garment interlining was used. The weight was 66.3 gm per sq. mt. and the nominal fibre blend was polyester / viscose (65% / 35%). The microscopic view of the fabric proves that the non-woven fabric was thermally bonded.

A burning test, chemical test and microscopic test were performed to confirm the presence of polyester and viscose in the non-woven fabric.

The non-woven fabric was standardized by the same process that was followed for woven cotton fabric (refer to 2.2.2.1). The only difference made was the size of the sample changed from 10 cm X 10 cm to 20 cm X 10 cm.

A laboratory coating machine (K Bar Hand Coater) was used to apply a nominally 40 micrometer coating film to the non-woven fabric. In this way the B.S.E. could be applied to one side of the fabric, hence providing greater opportunity for transfer of B.S.E. to the skin of a wearer.
2.3.2

Coating of Non-Woven fabric with B.S.E. dispersion / paste in water:

The non-woven fabric was coated with 30%, 60%, 90%, 120%, 150% and 180% B.S.E.
dispersion / paste in water. All the dispersed solutions and pastes used in the process were
freshly prepared.

Each non-woven fabric sample of size 20 cm X 10 cm was coated with the freshly prepared
B.S.E. dispersed solution or paste. B.S.E. powder was weighed and added to 5 ml of water to
make the respective dispersion or paste (for example 3 gm of B.S.E. powder was added to 5
ml of water to form 60% B.S.E. dispersion, whereas 7.5 gm of B.S.E. powder was added to 5
ml of water to form 150% B.S.E. paste).

The following charts show the oven dry weight of the fabric before coating, the weight of the
fabric after coating and the oven dry weight of the fabric after coating. It also shows the
percentage weight gain by the fabric after coating process.

**Percentage Weight Gain**

\[
\text{Percentage Weight Gain} = \frac{\text{(fabric oven dry weight after coating} - \text{fabric oven dry weight before coating} \times 100)}{\text{weight of oven dry fabric before coating}}
\]

The 30%, 60% and 90% B.S.E. in water is stated as dispersion of B.S.E. in water whereas
120%, 150% and 180% are paste of B.S.E. in water. This is because in the latter case, the
viscosity of the dispersion of B.S.E. is high and the ‘mixture’ approaches a viscous paste.
2.3.2.1

Coating of Non-woven fabric with 30% B.S.E. dispersion in water:

<table>
<thead>
<tr>
<th>Sample</th>
<th>weight of dry fabric before coating</th>
<th>weight after coating</th>
<th>weight of dry fabric after coating</th>
<th>% weight gain</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>1.22</td>
<td>4.52</td>
<td>2.35</td>
<td>93</td>
</tr>
<tr>
<td>b</td>
<td>1.55</td>
<td>6.68</td>
<td>2.62</td>
<td>69</td>
</tr>
<tr>
<td>c</td>
<td>1.27</td>
<td>5.78</td>
<td>2.33</td>
<td>83</td>
</tr>
<tr>
<td>d</td>
<td>1.44</td>
<td>6.27</td>
<td>2.51</td>
<td>74</td>
</tr>
</tbody>
</table>

The table and graph above show that when the non-woven fabric is coated (nominal 40 micron thickness) with 30 % B.S.E. dispersion, the weight gain of the fabric ranged from 69% - 93%. The average weight gain was 80%. The reason for the large variation in weight gain is, when the fabric is coated, variable wicking occurs as the paste is drawn along the fabric and under the coating blade. The movement of the paste into the interstices of the non-woven also depends on the rate at which the paste or dispersion is drawn over the fabric. As this coating process was manual (Black colour coded K hand coater) some variability must be expected. The experiments confirmed this. However, large concentrations of B.S.E. could be
applied relatively simply, to one side of an inexpensive non-woven textile material. In this way, the treated non-woven could rest against the human body under conventional clothing, and be supported by a range of means to maintain close contact with the skin.
Coating of Non-woven fabric with 60% B.S.E. dispersion in water:

<table>
<thead>
<tr>
<th>Sample</th>
<th>weight of dry fabric before coating</th>
<th>weight after coating</th>
<th>weight of dry fabric after coating</th>
<th>% weight gain</th>
</tr>
</thead>
<tbody>
<tr>
<td>e</td>
<td>1.32</td>
<td>5.18</td>
<td>2.88</td>
<td>118</td>
</tr>
<tr>
<td>f</td>
<td>1.33</td>
<td>4.56</td>
<td>2.81</td>
<td>111</td>
</tr>
<tr>
<td>g</td>
<td>1.36</td>
<td>5.06</td>
<td>2.99</td>
<td>120</td>
</tr>
</tbody>
</table>

The same process as in 30% B.S.E. coating was followed for 60% B.S.E. coating on the non-woven fabric. The weight gain of the fabric ranged from 111% - 120% with an average of 116%. We can see that the difference in weight gain percentage between 30% and 60% B.S.E. coating was 36% with an increase of 30% material in the dispersed solution.
Coating of Non-woven fabric with 90% B.S.E. dispersion in water:

<table>
<thead>
<tr>
<th>Sample</th>
<th>weight of dry fabric before coating</th>
<th>weight after coating</th>
<th>weight of dry fabric after coating</th>
<th>% weight gain</th>
</tr>
</thead>
<tbody>
<tr>
<td>h</td>
<td>1.29</td>
<td>5.20</td>
<td>3.30</td>
<td>126</td>
</tr>
<tr>
<td>i</td>
<td>1.41</td>
<td>5.51</td>
<td>3.10</td>
<td>130</td>
</tr>
<tr>
<td>j</td>
<td>1.36</td>
<td>5.45</td>
<td>3.29</td>
<td>136</td>
</tr>
</tbody>
</table>

Here also we can see that the weight gain in the fabric ranges from 126% - 136%. The average weight gain is 131%. The difference between average value for weight gain for 60% and 90% B.S.E. solution is 15%.
Coating of Non-woven fabric with 120% B.S.E. paste in water:

<table>
<thead>
<tr>
<th>Sample</th>
<th>weight of dry fabric before coating</th>
<th>weight after coating</th>
<th>weight of dry fabric after coating</th>
<th>% weight gain</th>
</tr>
</thead>
<tbody>
<tr>
<td>k</td>
<td>1.38</td>
<td>5.11</td>
<td>3.42</td>
<td>148</td>
</tr>
<tr>
<td>l</td>
<td>1.37</td>
<td>5.46</td>
<td>3.07</td>
<td>146</td>
</tr>
<tr>
<td>m</td>
<td>1.27</td>
<td>4.64</td>
<td>3.1</td>
<td>144</td>
</tr>
<tr>
<td>n</td>
<td>1.34</td>
<td>5.07</td>
<td>3.43</td>
<td>156</td>
</tr>
</tbody>
</table>

The above graph and table shows the weight gain of the fabric ranges from 144% -156%. The average weight gain in 120% B.S.E. paste is 149%.
Coating of Non-woven fabric with 150% B.S.E. paste in water:

<table>
<thead>
<tr>
<th>Sample</th>
<th>weight of dry fabric before coating</th>
<th>weight after coating</th>
<th>weight of dry fabric after coating</th>
<th>% weight gain</th>
</tr>
</thead>
<tbody>
<tr>
<td>o</td>
<td>1.22</td>
<td>4.02</td>
<td>2.84</td>
<td>133</td>
</tr>
<tr>
<td>p</td>
<td>1.57</td>
<td>5.21</td>
<td>3.72</td>
<td>137</td>
</tr>
<tr>
<td>q</td>
<td>1.56</td>
<td>4.94</td>
<td>3.62</td>
<td>132</td>
</tr>
</tbody>
</table>

Even though we can see a more uniform weight gain in the fabric for 150% B.S.E. paste coating on non-woven fabric (weight gain range from 132% - 137%), the average weight gain is 134% which is less than average weight gain for 120% B.S.E. coating.
Coating of Non-woven fabric with 180% B.S.E. paste in water:

<table>
<thead>
<tr>
<th>Sample</th>
<th>weight of dry fabric before coating</th>
<th>weight after coating</th>
<th>weight of dry fabric after coating</th>
<th>% weight gain</th>
</tr>
</thead>
<tbody>
<tr>
<td>r</td>
<td>1.45</td>
<td>5.17</td>
<td>3.85</td>
<td>166</td>
</tr>
<tr>
<td>s</td>
<td>1.26</td>
<td>4.86</td>
<td>3.58</td>
<td>184</td>
</tr>
<tr>
<td>t</td>
<td>1.37</td>
<td>4.47</td>
<td>3.39</td>
<td>147</td>
</tr>
<tr>
<td>u</td>
<td>1.46</td>
<td>5.24</td>
<td>3.85</td>
<td>164</td>
</tr>
</tbody>
</table>

The weight gain range for 180% B.S.E. paste coating was 147% - 184%, with an average of 165%. But due to very high viscosity, the coating was not even and became patchy. This is demonstrated by the variation in weight per unit area within the fabric.
2.3.3.

**Experimental Analysis:**

The graph below shows the weight gain (%) of the fabric (after coating with B.S.E.) against B.S.E. % of the coating paste. These experiments suggest there is a maximum weight gain on the non-woven fabric of about 150%. However at this level, there should be sufficient B.S.E. on a disposable patch for transfer of B.S.E. from the surface of the fabric to the surface of the skin (for absorption into the body).

\[ y = -0.0075x^2 + 2.189x \]
\[ R^2 = 0.65 \]

The above graph may be used for designing the amount of B.S.E. on the fabric surface, hence varying the potential delivery rate to the body. The data suggests that there is a decrease efficiency of application of B.S.E. at higher concentrations in the paste.
Using the equation given on the previous page, we find the maxima value for the equation using differential equations.

\[ y = -0.0075 \, x^2 + 2.189 \, x \]

\[ \frac{dy}{dx} = -0.0075 \times 2 \, x + 2.189 \]

To find the maxima,

\[ \frac{dy}{dx} = 0 \]

Therefore,

\[ 0 = -0.0075 \times 2 \, x + 2.189 \]

\[ 0.0150 \, x = 2.2901 \]

\[ x = 146 \]

The calculations show that the percentage of B.S.E. powder in B.S.E. paste for maximum weight gain is nearly 145%.

However, as the B.S.E. dispersed solution at 30% is non-suitable for coating because of its low viscosity and also 180% B.S.E. paste because of its high viscosity, these two readings were excluded to find the maximum value of B.S.E. in water to get the maximum weight gain. In both the 30% B.S.E. solution and 180% B.S.E. paste, the coating is uneven.

The graph on the following page shows the equation that is used to find the maximum value of B.S.E. powder required to get maximum weight gain on the non-woven fabric (not taking into consideration the values for 30% B.S.E. solution for coating and 180% B.S.E. paste for coating on non-woven fabric):
Using the equation given above, we find the maxima value for the equation using differential equations.

\[
y = -0.0106x^2 + 2.4927x
\]

\[
dy / dx = -0.0106 \times 2x + 2.4927
\]

To find the maxima,

dy / dx = 0

Therefore,

\[
0 = -0.0106 \times 2x + 2.4927
\]

\[
0.0212x = 2.4927
\]

\[
x = 118
\]

These results suggest that the most suitable percentage of B.S.E. powder in B.S.E. paste for coating on non-woven fabric for maximum weight gain is 118\%.
2.3.4.

Rubbing Fastness

Coated fabric samples were subjected to rub fastness testing using an electric crock-meter (A.A.T.C.C. Crock-meter by Atlas Electronic Device Co). The tests were carried out by rubbing with both wet and dry rubbing cloths.

This test was carried out in order to evaluate the presence of B.S.E. on the surface of the coated fabric. The lower is the values of rubbing fastness, the higher the possibility of B.S.E. transferring from the fabric to the surface of the skin. B.S.E. on the surface of the skin is for the availability for the absorption of B.S.E. by the skin.

<table>
<thead>
<tr>
<th>Percentage B.S.E. solution*or paste coated on non-woven fabric</th>
<th>Dry Rubbing</th>
<th>Wet Rubbing</th>
</tr>
</thead>
<tbody>
<tr>
<td>30 %</td>
<td>4 – 5</td>
<td>2</td>
</tr>
<tr>
<td>60 %</td>
<td>4 – 5</td>
<td>2</td>
</tr>
<tr>
<td>90 %</td>
<td>4 – 5</td>
<td>1 – 2</td>
</tr>
<tr>
<td>120 %</td>
<td>4 – 5</td>
<td>1 – 2</td>
</tr>
<tr>
<td>150 %</td>
<td>4</td>
<td>1 – 2</td>
</tr>
<tr>
<td>180 %</td>
<td>4</td>
<td>1</td>
</tr>
</tbody>
</table>

**B.S.E. Solution* or paste is dispersion of B.S.E. powder in water**

Under dry rubbing conditions there was minimal transfer of B.S.E. from the treated fabric to the adjacent cotton fabric. The transfer however increased significantly when the adjacent fabric was moist.
2.3.5.

Assessment of the transfer of B.S.E. from treated fabric to simulated skin:

A Martindale abrasion testing apparatus was used to assess whether B.S.E. could be transferred from the treated fabric surface to a simulated skin. The same procedure that was followed for treated woven fabric was applied here for coated non-woven fabric. In this experiment, the conventional standard abradant fabric was replaced with a natural leather to simulate human skin.

The initial weight of the specimen (non-woven fabric treated with B.S.E.) and the abradant (natural leather) was recorded to three decimal points. After 10,000 rubbing cycles the fabric and leather were weighed again. There were no weights added to the specimen as done in abrasion test as it was assumed that the fabric rubs with the human body every 4 seconds, and each rubbing cycle signifies the fabric rubbing with human body.

For both the wet and dry fabrics, no B.S.E. was transferred from the fabric specimen to the leather surface. Given below are the electron microscopic view of the surface of leather with zero rubbing cycles and 10,000 rubbing cycles with the B.S.E. coated non-woven fabric.
Figure 22: The above micrographs show the leather surface before and after the simulated skin was subjected to 10,000 cycles with B.S.E. coated non-woven fabric.
2.3.6. Electron Microscopy

*The following figures show a comparison between non-woven with 0%, 30%, 90%, 120% and 150% B.S.E. coating:*

*Figure 23*

With increase in percentage of B.S.E. a brittle layer is formed on the surface of the non-woven fabric.
The figures 24 given below demonstrate the presence of B.S.E. on the surface of the fabric (400X, uncoated versus 30% B.S.E.)

Figure 24
Figure 25 and figure 26 illustrate the presence of B.S.E. on the surface of the fibre and between the fibres.
Figure 26: Electron Micrographs for 0%, 30%, 90% & 150% B.S.E.
This figure (figure 27) proves that with increase of percentage the property of coating changes and there is crater and crack formation in higher percentage of B.S.E. coating.
The following figures show variation within the fabric coated with B.S.E.:

**Figure 28: Variation within fabric (150% B.S.E. coating)**

**Figure 29: Variation within fabric (120% B.S.E. coating)**
The micrographs below show the view of non-woven fabric coated with different concentrations:

Figure 30: Electron Micrographs for untreated non-woven fabric at 100, 400, 1400 & 5000X
Figure 31: Electron Micrographs for non-woven fabric coated with 30% at 100, 400, 1400, 2000 & 5000X
Figure 32: Electron Micrographs for non-woven fabric coated with 90% at 100, 400, 1000, 2000 & 5000X
Figure 33: Electron Micrographs for non-woven fabric coated with 150% at 100, 400, 1500 & 2000X
2.3.7.

Fourier Transform Infrared Spectrophotometer (FTIR)

Perkin Elmer Spectrum 100 FTIR Spectrum was used to characterize Boswellia Serrata Extract on non-woven fabric by Horizontal ATR using ZnSe crystal.

The IR Spectra of untreated non-woven fabric and also the non-woven fabric coated with 30, 60, 90, 120, 150 and 180% B.S.E. paste are shown in the figures 34, 35 & 36.

In the figure 34, the IR Spectra shows with coating application on non-woven textiles there is a rise in peak between 3050 cm\(^{-1}\) and 3500 cm\(^{-1}\). There is also a fall in peak intensity between 1710 cm\(^{-1}\) & 1730 cm\(^{-1}\) and also between 1140 cm\(^{-1}\) & 1250 cm\(^{-1}\).
Figure 34: IR Spectra of B.S.E. coating on non-woven fabric
Figure 35: IR Spectra for B.S.E. treated and untreated non-woven fabric between 1207 cm⁻¹ and 1720 cm⁻¹
Figure 36: Comparison between non-woven fabric and non-woven fabric coated with 60% B.S.E. paste using IR Spectra
CHAPTER 3

CONCLUSION & RECOMMENDATIONS

3.1

CONCLUSION:

This study has investigated the use of the textile materials as an alternative method for the transdermal delivery of B.S.E., a traditional South Asian anti-inflammatory medicine. Two textile fabrics were used to explore their potential as a carrier for delivery of B.S.E.;

a) A woven pure cotton shirting, in which case the B.S.E. was applied to the fabric by pad application, and

b) A non-woven viscose-rich lining material where the B.S.E. was applied to one face by coating.

The B.S.E. is not wholly soluble in water or alcohol. The long chain aliphatic compounds, said to be solvents for B.S.E., did not improve the solubility. As water is the most commonly used application medium for the textile industry, it was decided to apply the B.S.E. either as dispersion (for the woven fabric) or as a paste (for the non-woven). The dispersions used were visually stable with no discernible sedimentation after 30 minutes.

A high liquor pick-up was used for the application of B.S.E. by padding. This was done to maximize the amount of B.S.E. that could be applied. Furthermore, the use of pick-up of 90-100% (the upper limits of industry practise) causes the applied solution to migrate to the fabric surface during drying. In this way there will be a higher amount of B.S.E. available for transfer from the fabric surface to human skin.

The maximum achievable concentration of a dispersion of B.S.E. in water was 350 g/l (35%). Beyond this concentration the dispersion became too viscous to use.

The maximum concentration achievable by padding was approximately 4-5 mg/sq. cm.
A difference in theoretical and actual weight gain percentage of cotton woven fabric after padding was observed. It was concluded that this difference was due to

a) The presence of volatile substances in B.S.E. powder which were lost during hot air drying, and

b) A negative affinity of B.S.E. for cellulose which resulted in less B.S.E. being applied when coming into contact with the aqueous dispersion

With decreasing drying temperature of the padded cotton fabric, the weight gain of the woven fabric increased. This indirectly confirms the presence of volatile substances in B.S.E.. Hence over the temperature range of 50°C to 110°C, temperature was directly proportional to the weight loss of B.S.E. powder and conversely the weight gain of the fabric was inversely proportional to the drying temperature.

Assessment of the transferability of B.S.E. from the fabric to an adjacent material (simulating human skin) showed there was negligible transfer at 20°C/65% RH. At approximately 100% humidity (damp fabric) the transfer increased but not to an extent sufficient to deliver suitable quantities of B.S.E. to human skin.

The addition of acrylic based binder had no effect on the durability of B.S.E. to laundering. The retention percentage ranged between 1.7% and 2.7%, depending on the percentage of B.S.E. dispersion used for padding.

Padding provided an inefficient way of applying B.S.E. as the distribution is relatively uniform through the fabric. From the above two points it is clear that a more successful approach would be based on a single-side application (e.g. coating).

Coating was found to be more suitable for a higher percentage of solid add-on of B.S.E. onto the textile substrate. A non-woven fabric was more suitable for the application, as B.S.E. could be applied to one side of an inexpensive textile material and the coated fabric could rest against the human body. This can be used as a patch under the conventional clothing.

A concentration of 30% B.S.E. dispersion in water and 180% B.S.E. paste was not suitable for the coating application on non-woven fabric. The 30% B.S.E. dispersion in water gave a non-uniform coating because of low viscosity of the dispersion, whereas the 180% B.S.E. paste gave non-uniform coating because of high viscosity of the B.S.E. paste in water. The
optimum percentage for coating application of B.S.E. on non-woven fabric was of the order of 120%, giving the highest and most uniform amount of solid add-on onto the non-woven fabric. The highest achieveable concentration using coating application was approximately 12 mg/sq. cm.

It was observed that due to higher percentage of B.S.E. on the coated non-woven fabric there was a transfer from the coated fabric under the dry condition (20°C, 65% RH) that was lacking in B.S.E. padded cotton samples. The transfer increased when the adjacent fabric was moist.

Electron scanning micrographs were used to confirm the presence and distribution of B.S.E. on the fabric and fibre surface available for transfer from fabric surface to skin.
3.2

RECOMMENDATIONS:

This is the first known study of the application of B.S.E. on to textile substrates. It is suggested that coating is the most suitable process for application of B.S.E. on the textiles for a high availability of chemical compound to be transferred from fabric surface to the human skin. The recommended carrier for the B.S.E. or similar analgesics is an inexpensive non-woven particularly where there is a high proportion of cellulosic fibre to aid human comfort.

Some options like hydro-gels and high moisture content backing fabric for the non-woven patch can be explored for improved transfer of B.S.E. from fabric to skin upon rubbing.

As Boswellic Acids have two major functional groups, purer forms of Boswellic Acids can be encapsulated and applied on the textile substrate to form chemical bonds for the transdermal drug delivery.

Chemical compounds like beta/gamma cyclodextrin can be used along with pure forms of Boswellic Acid on textile substrates for controlled drug release and transdermal drug delivery.

Due to the ethical requirement of not using live subjects, the transdermal deliver of B.S.E. was assessed using adjacent materials such as cotton and leather. Further research may be done on living animals to assess the transdermal drug delivery and subsequent examination of the content of Boswellic Acid in the blood plasma by HPLC.
Abbreviations & Glossary

GMO: Genetically Modified Organism

TLC: Thin Layer Chromatography

ppm: Parts Per Million

cfu/g: Colony forming unit per gram (for microbiological culture)

AOAC: Association of Analytical Communities

B.S.E.: Boswellia Serrata Extract

AKBA: A constituent of Boswellia Serrata known as acetyl-11-keto-Boswellic Acid.

Apoptosis: Programmed cell death which is an essential process in normal cell cycle.

Bradykinin: Large peptide formed by the action of proteases on kininogens that exerts an effect on blood vessels. Bradykinin is a very potent vasodilator and increases permeability of small vessels. It also causes spasms in some smooth muscles, generating pain.

Bronchial asthma: A form of asthma caused by hypersensitivity to an allergan.

5-lipoxygenase (5-LO): An enzyme that helps in the conversion of arachidonic acid to hydroxyeicosatetraenoic acids (HETE) and leukotrienes (LT).

Histamine: A compound formed by decarboxylation of histidine. It is a potent pharmacological agent acting through receptors in smooth muscle and in secretory systems. Stored in mast cells and released by antigen, as in the process of allergic reaction. It causes smooth muscle contraction of bronchioles and small blood vessels and increases the permeability of capilaries as well as secretion by nasal and bronchial mucus glands.

Human leukocyte elastase: Serine protease enzyme that will digest elastin and collagen type IV.

Leukotrienes: Mediators of various allergic and inflammatory reactions produced by the lipoxygenase pathway in leukocytes, mast cells, platelets and in the lungs.

Malignant glioma: Tumor of non-nervous cells which may be invasive or produce symptoms by pressing on surrounding structures depending on the grade of malignancy.

Non-Steroidal Anti-Inflammatory Drugs: A large group of anti-inflammtory agents that inhibit the production of prostaglandins. (e.g. aspirin, ibuprofen, and pyrazolone derivatives)

Prostaglandins: Products from the cyclooxygenase pathway of arachidonic acid metabolism present in almost all tissues rather than in specialized glands. There are primarily two stable main groups, prostaglandin E and prostaglandin F. They inhibit a variety of T and B-cell functions. They modulate the release of norepinephrine from sympathetic nerve endings. Prostaglandins are chemically a closely related 20-carbon unsaturated fatty acid containing a cyclopentane ring.
Retention Time: of a solute is taken as the elapsed time between the time of injection of a solute and the time of elution of the peak maximum of that solute. It is a unique characteristic of the solute and can be used for identification purposes. The corrected retention time of a solute is the retention time minus the retention time of a completely unretained solute. By multiplying the corrected retention time of a solute by the exit flow rate then the corrected retention volume can be obtained. If the mobile phase is compressible (i.e. the mobile phase is a gas) a pressure correction must be applied which is a function of the column inlet-outlet pressure ratio. Values of the corrected retention volume per ml of stationary phase for a solute measured over a range of temperatures can provide the standard energy of distribution, the standard enthalpy of distribution and the standard entropy of distribution for the solute concerned.

Ulcerative colitis: Inflammation and ulceration of the colon and rectum.

Appendix I:
Aim: To find the mass per unit area (in grams per square meter) of the cotton woven fabric used in the study.

Conditions:

All weighing during the test was performed in a standard atmosphere for testing textiles (20 +/- 1°C (70 +/- 2°F), 65 +/- 2 % RH), after the specimens has been conditioned in the same atmosphere.

Methodology:

The fabric sample was cut of size 10 cm X 10cm by a cutter and then weighed. The weight of the fabric was 1.89 grams.

Fabric sample size: 10 cm X 10 cm = 0.1 m X 0.1 m
Fabric weight: 1.89 grams

Calculations:

Mass per unit area (in gram per sq. meter) = 1.89 grams / 0.1m X 0.1m
= 1.89 X 10 X 10 grams per sq. m
= 189 grams per sq. m

Appendix II:
Aim: to show the co-relation of percentage of B.S.E. solution to the density and viscosity of B.S.E. solution.

Methodology:

A simple method was used to determine the approximate density of the solution. The required percentage of B.S.E. solution was weighed and then the volume of that solution was calculated. The density was calculated by the formula given below:

Density = Mass in grams of the solution / volume of the solution in ml

The viscosity of B.S.E. powder dispersion in water was calculated using a viscometer.

Analysis:

<table>
<thead>
<tr>
<th>Percentage B.S.E. solution</th>
<th>Density of the Solution</th>
<th>Viscosity in Centipoise at 20°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.95</td>
<td>1.0</td>
</tr>
<tr>
<td>5</td>
<td>0.97</td>
<td>2.5</td>
</tr>
<tr>
<td>10</td>
<td>0.98</td>
<td>3.5</td>
</tr>
<tr>
<td>15</td>
<td>1.00</td>
<td>5.0</td>
</tr>
<tr>
<td>25</td>
<td>1.06</td>
<td>15.0</td>
</tr>
<tr>
<td>35</td>
<td>1.12</td>
<td>53.0</td>
</tr>
</tbody>
</table>
The above graph shows that the viscosity increases exponentially with increase in percentage of B.S.E. in the dispersed solution at 20°C.
Appendix III:

Simulated Skin: Natural leather was used to assess the transdermal delivery of B.S.E. from the padded cotton fabric specimen or coated non-woven fabric samples. This was due to the ethical requirement of not using live being. Therefore, the natural leather is referred to as “simulated skin” in the report.
References


[21]: Lee RV, 2000, ‘Pleasure, pain, and prophylaxis: olfaction (the neglected sense)’, Baylor University Medical Center Proceedings, Volume 13, Issue 3, pages 261-266


