CHAPTER 3

MATERIALS AND METHODS

3.1 Introduction

Surgical gowns and drapes are used to protect both patient and surgical personnel from contamination during an operation. The surgical gown must act as a barrier between the sources of infection and the user and must also provide good thermal comfort. The present study has been undertaken to develop surgical gown for its performance improvement. Considering the generally used fabric types for manufactured surgical fabrics required to be studied. Therefore the woven and non-woven fabrics of different structures suitable for surgical gown and drapes have been considered for the characterization and analysis to evaluate the quality and performance parameters. The fabric surface has been modified by application of nano cellulose coating. Various characteristics such as nano cellulose pick up percentage, air permeability etc. of these fabrics have been measured to evaluate the comfort aspect.

Further antimicrobial treatment is also given to the potentially selected fabric specimen. These fabrics have been observed for the performance assessment by studying their anti-microbial properties.

3.2 Materials

3.2.1 Fabrics

For the study, three types of fabrics have been selected commonly required to produce surgical gown viz. one cotton woven, and two types of viscose rayon nonwoven. The cotton woven fabric has been procured from Mahavir Corporation, Ahmedabad. The viscose nonwoven fabrics produced by Ginni Filaments, Gujarat have also been procured from Mahavir Corporation, Ahmedabad. The various structural and physical specifications and mechanical properties of these fabrics have been evaluated and are given Table 3.1.

Table 3.1 Specifications of various fabrics used for performance study of surgical gown

Fabric Code	Fabric type	Yarn/Fibre	Mass density (gsm)	Breaking stress (g/tex)		Air permeability ($cm^3/cm^2/s$)
				m/c direction	cross m/c direction	
C_w	Cotton woven (plain weave)	Warp count 18Ne Weft count 8 Ne Ends/cm 18 Picks/cm 15	160	3.81	2.83	2500
V_p	Viscose spunlaced plain nonwoven	Fibre length 50 Denier 1.5	80	6.03	1.35	3600
V_e	Viscose spunlaced embossed nonwoven	Fibre length 50 Denier 1.5	80	7.14	1.8	3880

3.2.2 Nano cellulose

In the present work cellulose nano whisker is separated from industrial waste viscose rayon filaments. The breaking of the cellulose chain which contains high order crystalline regions connected with low order amorphous regions is having shape like individual rods. These rod-like particles are commonly called as whiskers. The average size of the particles is 348nm. These nanoparticles have to be applied to fabric samples by padding technique using suitable cross-linking agent in some cases.

3.2.3 Cross-linking agent

The nano cellulose particles are required to be applied on the fabric surface using fine layer such that it lasts longer; at the same time fabric should provide comfort to the user. A suitable cross-linking material needs to be used as linking agent for this purpose. ZYPOL-25 of Zydex has been used as a cross-linking agent, which is thermo plastic in nature. This is a high molecular weight anionic polyester resin-based dispersion. It has nano-size particles of polyester resin in water. It is free from alkyl phenol ethoxylates and heavy metal particles like Hg, Cd and Pb. It is a translucent liquid form having pale yellow colour, solid content 25 ± 1.5%. The cross linking agent has been procured from the Textile Chemistry Department, The M. S. University of Baroda

The cross-linking agent provides excellent adhesion to Polyester / Polypropylene / Acrylic/ Cellulose fibre and prevents migration. The nano size particles of the resin in water dispersion help in proper penetration and provide multi-point bonding even in the inner layers of the substrates. Its strong binding is long lasting even after repeated handling. It requires lower curing temperature compared to acrylic resin thereby saves energy cost and possibly

can improve machine productivity. Its viscosity can be altered by special additives for thickening. Moreover, as it is thermoplastic in nature, the final product made from synthetic material becomes recyclable.

3.2.4 Neem seed oil as antibacterial agent

Neem seed oil 'Neembtaila' of Shri Narayan Ayurvedic Pharmacy, India has been used as antibacterial agent. Shree Narayan Ayurvedic Pharmacy, India is an ISO 9001:2015 certified company. Neem seed oil contains different diterpenois and triterpenoids like nimbin, gedunin and azadiretchin. These contents of neem seed oil have properties as an antiseptic, antibacterial and antifungal. So the solution of neem seed oil has been used in this work.

3.3 Methods

Nano cellulose powder has been developed from viscose filament yarns, procured from the industry. Two fabric samples viz. cotton woven (vat dyed) and embossed viscose non-woven have been processed using nano cellulose solution. Two different concentrations of the nano cellulose solutions have been prepared to process the fabric samples using two different concentrations. Thus total 8 specimens have been prepared using the nano cellulose solutions in different baths. The treated samples have been tested for their water repellence and air permeability. To improve water repellence property of fabrics, concentrations of nano cellulose and cross-linking agents have been increased. One more fabric- plain viscose non-woven is also used for similar coating treatment.

3.3.1 Preparation of nano cellulose

The required quantity of nano cellulose has been made for coating of the fabric samples. The nano cellulose in powder form has been prepared from viscose rayon filament by chemical treatment using pure sodium zincate solution as described under.

(a) Preparation of sodium zincate solution: A solution of sodium zincate is required for producing nano crystals from the ground viscose rayon filaments. ZnO is added to mixture of NaOH and water in suitable proportion. First, 180g of NaOH is added to 200ml of distilled water. 80g of ZnO has then gradually added with constant stirring. The solution is then stored at room temperature for 24 hours in a closed container. Thereafter it is filtered using Whatman No.1filter to get pure zincate solution.

(b) Preparation of nano cellulose powder: First, the viscose rayon filaments have been ground to smaller than 20 mesh powder. The ground viscose powder has been mixed with sodium zincate in a ratio of 1:9 (g/ml) to make suspensions of nano cellulose crystals. A reaction temperature of 50°C has been maintained for the diffusion of sodium zincate into the amorphous region of the filaments resulting in a subsequent cleavage of the glycosidic bonds. After 1 hour, the particles have been neutralized by glacial acetic acid solution. The suspension has been washed and further filtered by Whatman No.1 filter paper. The colloidal suspension has been evaporated and converted in powder form. The powder has been washed with distilled water and dried.

3.3.2 Coating of nano cellulose on fabric surface

The comparison of various physical and mechanical characteristics of coated fabrics has been made by coating the fabric using nano cellulose solution which is prepared in distilled water heated at 50°C. The nano cellulose powder has been prepared in Textile Chemistry Laboratory. The powder has been added with constant stirring to prepare the solution of various concentrations; without cross linking agent. These solutions have also been prepared with addition of cross-linking agent ZYPOL-25; of various concentrations as required.

The fabric samples of size 40cm x 30cm have been cut from all the fabrics. Each fabric sample is conditioned for 2 hours at room temperature; there after it is treated with the cellulose solution at room temperature with one-dip-one-nip condition in padding mangle machine. First the machine required to be cleaned thoroughly with water; there after the liquor of particular cellulose concentration without adding cross linking agent in to the solution have been taken in the machine. The all the fabric specimens viz. cotton woven, viscose plain nonwoven and viscose embossed nonwoven required to be coated are processed. After the application of liquor on samples, the samples have been collected and kept at room temperature on a clean surface to avoid any type of stress on the sample and to initiate drying. They have been then placed in conditioning oven, set at 80°C temperature for 20 minutes. There after all these treated samples have been cured in oven for 3 minutes at 115°C temperature. Then the padding mangle has been washed thoroughly and similar procedure has been carried out to treat with another nano cellulose solution prepared with cross linking agent. The procedure is repeated for various concentrations.

3.3.3 Preparation of fabric specimen of coated fabric

First cotton woven and viscose nonwoven (embossed) fabrics have been selected for surface modification, by coating of nano cellulose solution of 2.5gpl and 5gpl concentration without cross linking material. To improve the adhesion of the coat, the cross linking agent having 5gpl concentration by weight have been added in these solutions; and the fabric samples have been treated in these solutions. Thus, total of 8 fabric specimens have been coated as listed in Table 3.2. These fabric specimens have been evaluated for air permeability and water repellence. There was not much improvement in water repellence and other characteristics. Therefore fabrics have been treated with higher concentration of nano cellulose to improve these characteristics.

Further all the 3 fabrics including viscose nonwoven (plain) have been processed in nano cellulose solution having three different concentrations i.e. 10gpl, 20gpl and 30gpl; without cross-linking agents and two concentrations of cross-linking agent i.e. 5gpl and 10gpl. Thus, total of 27 fabric specimen have been prepared using parameters as given in Table 3.3.

Table 3.2Various parameters of nano cellulose solution for fabric coating

No.	Sample fabric	Nano cellulose concentration (gpl)	Cross linking agent concentration (gpl)
1	Cotton	2.5	0
2	Cotton	2.5	5
3	Cotton	5	0
4	Cotton	5	5
5	Viscose	2.5	0
6	Viscose	2.5	5
7	Viscose	5	0
8	Viscose	5	5

Table 3.3Various specifications of cellulose solution for coating and fabric code

No.	Sample code	Nano concentration (gpl)	Cross linking agent (gpl)	Fabric code
1	1A	10	0	Cw
2	1B	10	0	Ve
3	1C	10	0	Vp
4	2A	10	5	Cw
5	2B	10	5	Ve
6	2C	10	5	Vp
7	3A	10	10	Cw
8	3B	10	10	Ve
9	3C	10	10	Vp
10	4A	20	0	Cw
11	4B	20	0	Ve
12	4C	20	0	Vp
13	5A	20	5	Cw
14	5B	20	5	Ve
15	5C	20	5	Vp
16	6A	20	10	Cw
17	6B	20	10	Ve
18	6C	20	10	Vp
19	7A	30	0	Cw
20	7B	30	0	Ve
21	7C	30	0	Vp
22	8A	30	5	Cw
23	8B	30	5	Ve
24	8C	30	5	Vp
25	9A	30	10	Cw
26	9B	30	10	Ve
27	9C	30	10	Vp

(Cw: Cotton woven, Ve: Viscose embossed- nonwoven, Vp: Viscose plain-nonwoven)

3.3.4 Evaluation of various characteristics

The comparison of various characteristics of surface treated fabric samples with that of untreated fabrics has been made to evaluate the performance improvement as gown fabric. Physical properties such as mass density, fabric thickness have been measured. The various mechanical properties such as tensile strength, air permeability, water repellence and fabric stiffness have been measured.

(a) Physical parameters of fabric: Fabric parameters viz. its thickness, pore size and mass density are important in its mechanical characteristics such as tensile strength, stiffness, air permeability and water repellence of fabric which ultimately affect the performance of the fabric. Fabric structure and pore size also affects the finishing characteristics such as sizing, dyeing, coating etc. In the present work; the fabrics have been treated with nano cellulose solutions to produce a coating of nano cellulose on specimen. This coating increases the mass density of the specimen which can influence the drape and comfort properties of the fabric. The mass density and thickness of nominal fabric and chemically treated fabric has been measured.

Mass density of fabric (GSM) has been measured (i.e. weight of fabric in gram per square metre) using ASTM D3776 standard. A special GSM-cutter is used to cut a uniform circular fabric of 100 cm^2 area and weight of specimen in gram (w) is measured using an electric balance. 10 different specimens from different location on the fabric have been taken and average mass (w_a) has been calculated. Fabric mass after coating the fabric surface also has been measured using the same procedure as (w_c).

- Fabric mass density has been obtained by the formula: (GSM) = w_a*100
- Change in the mass density have been calculated and expressed as % increase in mass density as : (w_c - w_a) /w_a *100

Fabric thickness i.e. distance between both surfaces of fabric under a specified applied pressure have been measured using ASTM D1777 – 96 standard. The thickness gauge has analog dial for measuring thickness in millimeters with maximum capacity 10mm and accuracy 0.01mm. The fabric sample has been kept on an anvil of the gauge. Then the presser foot is gently lowered onto the specimen and dial-reading is taken to obtain the

thickness of specimen. The procedure is repeated to obtain the values of thickness at 10 different locations. Average thickness (T_a) is calculated to evaluate the fabric stiffness.

(b) Evaluation of tensile strength: Tensile strength measurement is the most common mechanical measurement of the fabrics. This test provides information about general strength of the fabric. The specimen is axially loaded and stretched in this test. Tensile strength depend on the type of fibre, arrangement of fibres and yarns in the specimen and fabric structure. Hence tensile strength test have been carried out for the fabrics.

The British Standard for fabric tensile strength measurement has been followed. This involves extending a strip of fabric to its breaking point by a suitable mechanical means which can record the breaking load and extensions. Five fabric samples have been extended in a direction parallel to warp and five parallel to the weft, no two samples to contain the same longitudinal threads.

The specimens are cut to a size of 600mm x 300mm and then frayed down in the width equally at both sides to give samples which are exactly 50 mm wide. This ensures that all the threads run the full length of the sample so contributing to the strength and also that the width is accurate. The rate of extension has been set to 50mm/min and the distance between the jaws (gauge length) has been set to 200 mm. The sample has been pretensioned to 1% of the probable breaking load. Any breaks that occur within 5 mm of the jaws and that load sustainably less than the average have been rejected. Then mean breaking force and mean extension as a percentage of initial length have been reported.

(c) Measurement of air permeability: Air permeability decides fabric comfort of the fabric and garments. Air permeability is used to compare the breathability of different types of coated and uncoated fabrics and garments. Air permeability of the fabric generally depends on the pore size of the fabric. Air permeability test is equally important for technical textiles like suiting and shirting fabrics. Hence this test has been carried out. The purpose of this test is to find out air permeability of the fabric samples, which is one of the functions of comfort as well as pore size of the fabric. The air permeability of a fabric is a measure of how well it allows the passage of air through it. The ease or otherwise of passage of air is of importance for a number of fabric end uses.

Air permeability is defined as the volume of air passed per second through 100mm^2 area of fabric at a pressure difference of 10mm head of water. British standard test is followed for measurement of the airflow through a given area of fabric at a constant pressure drop of 10mm head of water. Specimen be clamped over the air inlet of the apparatus with the use of rubber gaskets and air is sucked through it by means of pump. The air valve is adjusted to give a pressure drop across the fabric of 10mm head of water and the air flow is then measured using a flow meter. Five specimens have been used each with a test area of 507mm^2 (25.4mm diameter) and the mean air flow is calculated in cm^3/cm^2/s.

(d) Evaluation of water repellence: Water repellence of the fabric decides the tendency of the fabric to allow the water droplets to pass through it. This property decides the fabric wetting tendency and fabric comfort. The evaluation of water repellency test has been carried out using AATCC TM22-2017 test method. This method is applicable to any textile fabric, which may or may not have been given a water-repellent finish. It measures the resistance of fabrics to wetting by water. It is especially suitable for measuring the water-repellent efficacy of finishes applied to fabrics.

In this test, fabric specimen is fitted in fabric holding frame and water is sprayed against the taut surface of a test specimen under controlled conditions. It produces a wetted pattern on the fabric surface on both side of the fabric whose size depends on the relative repellence of the fabric. Evaluation is accomplished by comparing the wetted pattern with pictures on a standard chart. For each fabric ten specimens have been tested and average rating number is reported corresponding to Standard Spray Test Rating Chart shown in Table 3.4.

Table 3.4 Standard spray test rating chart

Rating	Evaluation
100	No sticking or wetting of upper surface
90	Slight random sticking or wetting of upper surface
80	Wetting of upper surface at spray points
70	Partial wetting of whole of upper surface
50	Complete wetting of whole of upper surface
0	Complete wetting of whole upper and lower surfaces

(e) Determination of stiffness: The bending of the fabric is one of the important fabric properties. It measures the severity of bending or flexing action of the fabric. Resistance to bending is called flexural rigidity which affects the comfort as well as aesthetic appeal of the fabric and garment. In the present work nano cellulose coating has been applied to the fabric, which may affect the fabric stiffness. So, the stiffness test has been carried out.

Shirley stiffness tester has been used for the test. This is a form of the cantilever stiffness test, explained in B. S. Handbook No.11. The test specimen is each 25mm wide and 200mm long have been cut parallel to the warp and three parallel to the weft so that no two warp specimens contain the same warp threads, and no two weft specimens contain the same weft threads. A horizontal strip of fabric is clamped at one end and the rest of the strip is allowed to hang under its own weight to 41.5°.The advanced length of the strip is denoted as Lcm.

- Bending length (C) has been calculated as: $C = L (\cos ½ \Theta / 8 \tan \Theta)^{1/3}$
- Flexural rigidity (G) of the fabric is calculated as $G = MC^{3*}10^3$ mg/cm

  where Θ = angle to which fabric strip bends

  M = fabric mass per unit area (GSM)
- Bending modulus q is calculated as: $(q) = 12 * G * 10^{(-6)} / T_a^3$ kg/cm^2

  where T_a = fabric thickness (cm)

Four readings have been taken from each specimen, one face up and one face down on the first end, and then the same for the second end. The mean bending moduli for warp and weft way samples have been calculated using above formula.

3.3.5 Evaluation of antibacterial characteristic of fabric

AATCC test method 147 has been followed for evaluation of antibacterial characteristic of fabric sample treated with application of antibacterial finishes. This test method is used for antibacterial activity assessment of textile materials. Among the various coated fabric specimens evaluated for mechanical characteristics, the four samples (i.e. 8A, 8C, 9A and 9C) have been considered for the study. The processing parameters of these sample fabrics are given in Table 3.5.

Table 3.5Various fabric samples used for antibacterial treatment

Sample code	Sample fabric	Nano cellulose concentration (gpl)	Cross linking agent concentration (gpl)
8A	Cotton	30	5
8C	Plain viscose	30	5
9A	Cotton	30	10
9C	Plain viscose	30	10

This study is carried out at Microbiology Laboratory, Faculty of Pharmacy, The M. S. University of Baroda. This laboratory is equipped with sterilization autoclave, incubator, controlled atmospheric conditions and other required facilities required for the culture test.

This test is useful for obtaining a rough estimate of bacterial activity in which the growth of the inoculum organism decreases from one end of each streak to the other and from one streak to the next resulting in increasing degrees of sensitivity. The antibacterial treatment of the fabric specimen using neem seed oil solution has been given for this culture test.

(a) Preparation of neem seed oil solution: Initially, Neem seed oil solution sample has been prepared to treat one fabric sample. To prepare neem seed oil solution in this case, 195ml distilled water is taken in a glass beaker. Then 5ml neem seed oil has been added to the beaker. The neem seed oil has been then thoroughly mixed with distilled water at room temperature in the beaker using magnetic stirrer for 20 minutes and thus homogenous solution has been prepared. The solution is then heated to 50°C temperature for 15 minutes. Initial trial sample preparation is carried out and there after more quantity of neem seed oil solution has been prepared using the above-mentioned method.

(b) Application of neem seed solution on samples: Both, nominal untreated fabric and cellulose coated fabrics have been taken for the comparative study. The fabric samples of plain viscose non-woven and plain cotton woven have been cut in to narrow strips of 15mm x 60mm size from the roll in strips. The strips have been then immersed in the warm neem seed oil solution for 15 minutes. During this treatment the fabric strips have absorbed the solution thoroughly as the medium is warm. After 15 minutes, fabric strips have been taken out of the solution using a forceps. The fabric strips are then dried at room temperature by laying them down on the clean surface.

(c) Sterilization of samples: Sterilization process is necessary before performing any experiment. It is used to kill all the living microorganisms, including bacterial spores. The fabric samples used in the study have been sterilized in autoclave. A simple autoclave consists of a vertical cylindrical body with a heating element has been taken. It is provided with a tray to keep the articles in proper condition which can be closed by a lid that can be fastened. The safety valve, pressure gauge, and pressure release valve are other attachments provided for proper operation of the autoclave.

The fabric samples have been placed inside the autoclave. The lid is closed but the pressure release valve has been kept open and the water is heated. As the water started boiling, the steam drives the air out of the pressure release valve. When all the air is displaced and steam started appearing through the pressure release valve, the valve is closed. The pressure inside is allowed to rise up to 15lbs per square inch. At this pressure the articles are held for 15 minutes, after which the heating has been stopped and the autoclave is allowed to cool.

Once the pressure gauge shows the pressure equal to atmospheric pressure, the discharge tap is opened to let the air in. The lid is then opened and samples are removed. The sterilized fabrics are properly stored in air tight chamber for preparation of specimen for the microbiological culture test.

(d) Sample preparation for culture test: To study the comparative growth of bacteria on the fabric, untreated (control) fabric strip and a treated fabric strip have been to be placed in intimate contact with the agar surface of the Petri dishes which has been previously streaked with an inoculum of a test bacterium. First, 3 lines of bacteria have been drawn on Petri dish to allow the development of bacteria on and around the strips.

Two types of bacterium viz. gram positive, Staphylococcus Aureus (S.A.) and gram negative Escherichia Coli (E.C.) have been used on the strip to study their growth. Thus eight fabric samples are prepared as shown in Table 3.6. Three specimens of each fabric sample were required to prepare for proper estimation by averaging the results. Thus total of 48 Petri dishes have been prepared using both types of strips for the culture test. The detail procedure followed for preparation of Petri dish is described in next section.

Table 3.6 Various bacterium used for culture test of antibacterial finished fabrics

Sample code	Bacterium type	Name of the bacterium
8A	Gram positive	Staphylococcus Aureus (S.A.)
8A	Gram negative	Escherichia Coli (E.C.)
8C	Gram positive	Staphylococcus Aureus (S.A.)
8C	Gram negative	Escherichia Coli (E.C.)
9A	Gram positive	Staphylococcus Aureus(S.A.)
9A	Gram negative	Escherichia Coli(E.C.)
9C	Gram positive	Staphylococcus Aureus(S.A.)
9C	Gram negative	Escherichia Coli (E.C.)

(e) Preparation of culture medium and petri dishes: 13gpl nutrient broth has been taken with following contents:

Peptic digest of animal tissue - 5gpl,
Beef extract - 1.5gpl,
NaCl - 5gpl,
Yeast extract - 1.5gpl

- It has been heated to boil and pH was adjusted to 6.8 with 1N NaOH solution with continuous stirring.
- Then it is dispensed in 10ml amounts in conventional bacteriological culture tubes (125mm × 17mm).
- Tubes have been then Plugged with cotton and sterilized (autoclaving) at 103 kPa (15psi) for 15 minutes.
- 1.5% bacteriological agar is then added to nutrient broth, heated to boil, pH adjusted to 7.1 using NaOH solution.
- Then it has been dispensed in 15ml amounts in conventional bacteriological culture tubes, then plugged with the cotton and sterilize (autoclaving) at 103kPa (15psi) for 15 minutes. The sterilized 20ml amount has been poured from conventional bacteriological culture tubes to Petri dish.
- The environment has been kept sterile while making Petri dish for which working on the lamellar air flow instrument desk is required.

- The plates have been dried in the lamellar flow with UV light and the lid slightly off for 15 minutes. Drying the plate is very important for storing the plates and growing colonies on them. If not dried, the moisture will evaporate and condense on the lid during storage or during incubation and gives wet plates to work with. At worst the moisture can affect the plating of cells.
- Once prepared, media has been held at 4-5°C in the refrigerator for 1-2 weeks.

(f) Bacterial growth observations: To assess the antimicrobial characteristic of coated fabric, the bacterial growth on the fabric or resistance of bacterial colonies has been observed. Petri dishes have been prepared for anti-bacterial testing (Fig. 3.1). The AATCC test method 147 has been followed. This test method is used for antibacterial activity assessment of textile materials. The objective is to detect bacteriostatic activity on the fabrics. The method is useful for obtaining a rough estimate of bacterial activity in which the growth of the inoculum organism decreases from one end of each streak to the other and from one streak to the next resulting in increasing degrees of sensitivity. Development or resistance of bacterial colonies has been observed in the samples. The Petri dish prepared with specimen has been placed in the incubator. A thin glass slide is placed to apply required load on the fabric strips for their proper contact with agar surface. Petri dishes have been covered and placed in an incubator for 24 hours at 37°C temperature.

After incubation of 24 hours, Petri dishes have been taken out from the incubator and observed for the growth of Gram-positive bacteria (Staphylococcus aureus) and Gram-negative bacteria (Escherichia coli). The pictures of the dishes showing the bacterial growth have been taken after completion of each test.

Fig 3.1 Dishes prepared for anti-bacterial testing