CHAPTER – 3

MATERIALS AND METHODS

OVERVIEW:

The present chapter describes the polymerization processes of super absorbent pH-responsive polymers based on MMA, AA, MAA, AN, and ACR copolymers. The prepared pH-responsive polymers were microencapsulated with n-eicosane for application on sanitary napkins. Characterizations of prepared encapsulated were analysed by thermogravimetric analysis (TGA), scanning electron microscopy (SEM), and Fourier transfer infrared spectroscopy (FTIR). Coated napkins were quality checked with various parameters like water absorbency and saline solution absorbency for free swell capacity, absorption under load, rewet, striking through, antimicrobial, thickness, and pH.

3.1 Present scenario in the manufacturing of sanitary napkin

The production of sanitary napkins is economically feasible and holds substantial social importance. The Indian sanitary napkin industry exemplifies this, attaining an impressive US$ 0.758 billion in 2023. The marketplace is anticipated to attain US$ 16500 million by 2032, reflecting a healthy growing rate of 9.09%. Consistent market expansion indicates a rising awareness of and demand for high-quality sanitary goods.

The manufacturing of hygienic pads is both a lucrative economic venture and a socially important initiative. Manufacturers enhance the health and dignity of women by supplying needed hygiene goods. This venture has exciting opportunities for entrepreneurs due to an expanding market and the possibility for substantial earnings.

Investing in contemporary machinery and effective manufacturing methods can result in the manufacture of superior sanitary products, addressing the rising demand and enhancing women's lives. A meticulously crafted prototype project report and a tailored bankable project report are crucial for guaranteeing the success and sustainability of the manufacturing establishment. These reports facilitate the precise planning of financial and technical elements, so ensuring a successful enterprise in the sanitary napkin manufacturing sector (*Sanitary Napkin Manufacturing Business with Social Impact & Awareness*, n.d.)

The worldwide marketplace for sanitary pads or napkins was appreciated at 24.581 USD Billion in 2023 and is predictable to attain 35.96 USD Billion by 2030, exhibiting a growth rate of 4.50%. Sanitary napkins, towels, pads, and menstrual pads are utilized by ladies experiencing menstruation, postpartum bleeding, recovery from genital operations, premature delivery, abortion, or any circumstance requiring the absorption of vaginal blood. The market research for sanitary pads or sanitary napkins encompasses a brief description, categorization, present circumstances and patterns, possibilities and obstacles, industrial cycle assessment, comparative evaluation, business descriptions, and trade data. Before confirming the transaction, it conducts a detailed analysis of each kind, implementation, participant, five major areas, subdivisions of key countries, end users, channels, technologies, and additional information (*Sanitary Pads or Sanitary Napkin Market Report 2024 (Global Edition)*, n.d.).

Looking into the huge market potential the present research work has been designed in two phases as follows;

Phase I: Synthesis and characterization of pH-responsive polymers; Formulate microencapsulated phase change materials containing pH-responsive polymer.

Phase II: The polymer produced has been applied to the fabrics used to make female and infant hygiene products. Finally, the quality of the prepared product has been assessed for identical textiles for their commercial use.

3.2 Materials

All the chemicals were of laboratory grade and utilized after their purification. Solvents used in experimental work were distilled before their use.

Methyl methacrylate (CAS number: 80-62-6) (MMA), methacrylic acid (CAS number: 79-41-4) (MAA), diallyl maleate (CAS number: 999-21-3) (DAM), and benzoyl peroxide (CAS number: 94-36-0) (BP) were collected from A. B. Enterprise, Mumbai. Triallylamine (CAS number: 102-70-5) (TAA) and triallylphosphite (TAP) were imported from Shandong Luyue Chemical, China. Acrylonitrile (CAS number: 107-13-1) (AN or ACN) and toluene were acquired from Solvochem, Ahmedabad. The monomers' structures are as follow:

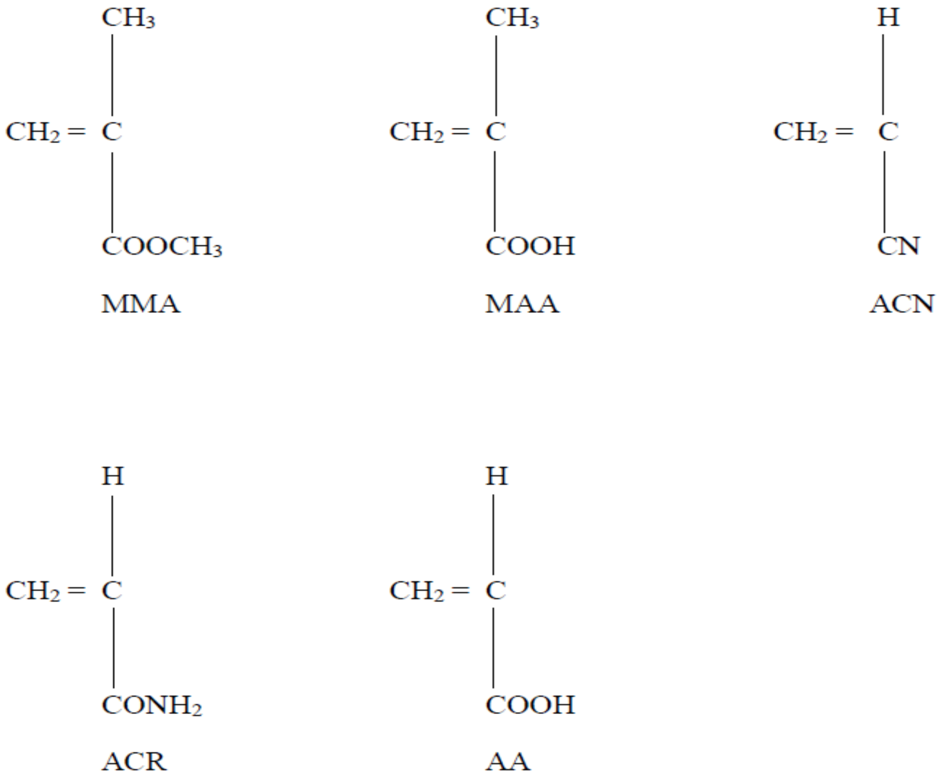

Figure 3.1: Structure of Monomers

While acrylic acid (AA) (CAS number: 79-10-7), acrylamide (CAS number: 79-06-1) (AM or ACR), diallyl phthalate (CAS number: 131-17-9), ethyl Acetate (CAS number: 141-78-6) (EA), tert-bytylhydroperoxide (CAS number: 75-91-2) (TBHP), aqueous ammonia and nitrogen gas were procured from Mumbai-Madhu Chemicals, Pawan Chemicals-Mumbai, Yash Organic-Mumbai, Arihant Chemicals-Vapi, Triveni Chemicals-Vapi, Ahmedabad-Sunshine Industrial Gase, Vapi-Suvidhi Industry, respectively.

3.3 Procedure of Polymerization

Synthesis of novel superabsorbent polymers (SAPs) has been performed via the following general homogeneous free radical solution polymerization procedure using two or more different monomers. The photograph of the polymerization assembly set up was given away in Figure 3.2.

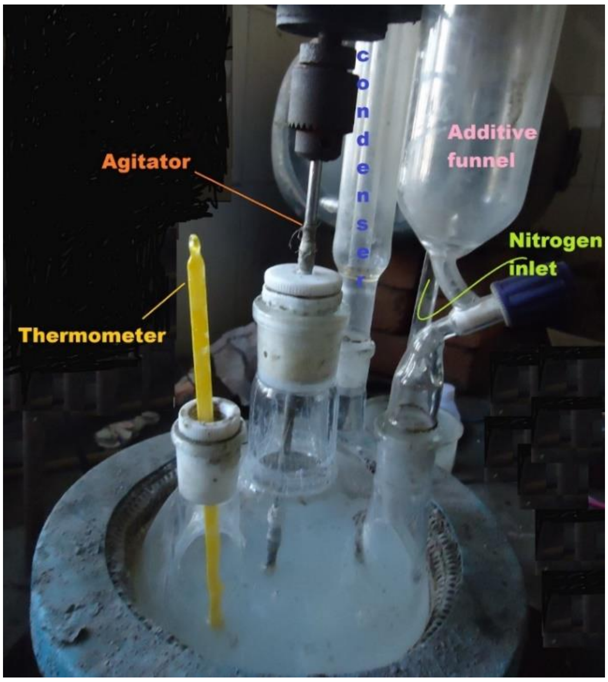

Figure 3.2: Assembly setup for the polymerization process

Five-necked round bottom flask is fitted with nitrogen gas inlet tube, additive funnel, thermometer, a double helical parallel impeller agitator with the capacity of 1000 cc. The flask is filled with porcelain chips approximately 2 small pieces, and solvent mixture with the quantity of 200 ml. Now unhurriedly heat the solvent combination to

40º C temperature and then permit the nitrogen gas to sprinkle the flask fillings to eject the air. 50 ml of monomer feed (cross-linker, monomers, and solvent mixture) is taken in additive funnel, and add the mixture dropwise with the rate of 3.0 ml/min beneath a nonstop watercourse of nitrogen gas. At the same time add 0.01 % initiator to initiate the polymerization and gradually increased the temperature to 80°C. As 50 ml monomer mixture addition is completed, the residual 200 ml of the mixture of monomers is transported to the additive funnel and dropwise addition of the feed is has been completed in 1.5 h. Throughout the process of polymerization, 80 – 90 ºC is maintained. At the termination of the reaction, inhibitor 0.01 % is added to inhibit the polymerization reaction.

3.3.1 General reaction scheme of polymerization

A proposed general reaction scheme for polymers is presented in figure 3.3. The various pendant groups R_1, R_2, R_3 and R_4 are defined in table 3.1.

Table 3.1: Components of monomers in reaction scheme-3.3

Polymers (SAPs)	**Monomers**	**R_1**	**R_2**	**R_3**	**R_4**	**R_5**	**R_6**
1A – 1E	AA and ACN	H	COOH	H	CN	---	---
2A – 2E	MMA and MAA	CH_3	$COOCH_3$	CH_3	COOH	---	---
3A – 3E	AA and MMA	H	COOH	CH_3	$COOCH_3$	---	---
4A – 4E	MAA and ACR	CH_3	COOH	H	$CONH_2$	---	---
5A – 5E	MMA and ACR	CH_3	$COOCH_3$	H	$CONH_2$	---	---
6A – 6E	AA and ACN	H	COOH	H	CN	Sodium Acrylate	
7A – 7E	MAA, ACR and ACN	CH_3	COOH	H	$CONH_2$	H	CN
The group R is the structural component of cross-linker							

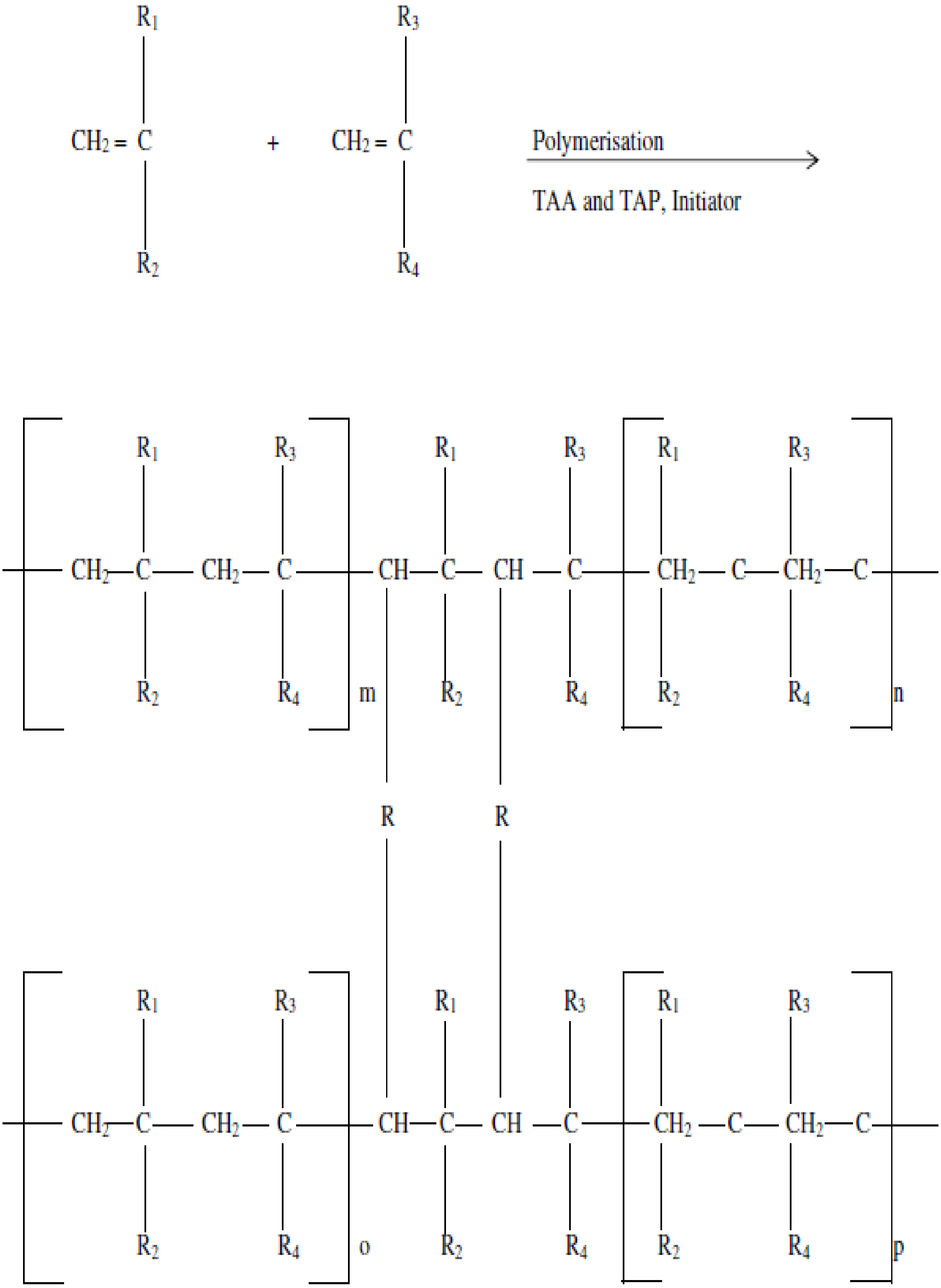

Figure 3.3: Reaction scheme for super absorbent polymers

3.4 Synthesis of AA and ACN based Polymers [1A-1E]

The various components used in the polymerization process for the synthesis of AA and ACN based SAP [1A-1E] are as follows:

Monomers	Acetic Acid (Monomer–a) and Acrylonitrile (Monomer-b)
Inhibitor	Tert-butyl hydroperoxide (TBHP)
Initiator	Benzoyl peroxide
Cross-linker	Triallylphosphite and Triallylamine In equivalent amounts
Solvent	Toluene (Solvent-I) and Ethyl Acetate (Solvent-II) (Solvent mixture of I:II is 60:40)

3.4.1 Polymerization procedure for SAPs 1A - 1E

A round-bottom flask having five-necked with a capacity of one thousand milliliters is equipped with a nitrogen gas inlet tube, thermometer, a water condenser, and an additive funnel. Additionally, a dual spiral parallel impeller campaigner is also included. After transferring solvent mixture (200ml) with a volume-to-volume ratio of 60:40 (toluene-120 ml and ethyl acetate-80 ml), the porcelain chips viz. two small pieces are added to the flask using a transfer method. The solvent combination should now be heated gradually to a temperature of forty degrees Celsius, and then the nitrogen gas should be passed through the container in order to banish the air. While the additive funnel is being filled with fifty milliliters of monomer feed (including solvent, cross-linker, and a mixture of monomers), the feed is being supplied drop by drop at a rate of three milliliters per minute while a steady stream of nitrogen gas is being introduced. In the meantime, 0.01 percent by weight of benzoyl peroxide is added in order to kickstart the polymerization process, and the temperature is gradually elevated to 80 degrees Celsius. In the event that the addition of fifty milliliters of monomer feed has been finished, the remaining two hundred milliliters of monomer feed are deposited into the additive funnel and added dropwise over the course of one and a half hours. During the entirety of the polymerization process, 80- and 90-degrees Celsius temperature must be kept constant. In order to prevent the polymerization reaction from occurring, 0.01% by weight of TBHP is added at the conclusion of the procedure. Table 3.2 has a variety of different mixtures that can be used for a series of experiments.

Table 3.2: Chemicals and quantity are set to formulate polymers 1A-1E

Experiment	**SAP**	**Solvent (ml)**		**Temperature (ºC)**	**Monomer (g)**		**Cross-linker (g)**
		I	**II**		**a**	**b**	
I	**1A**	240	160	80 – 90	80	20	0.01
II	**1B**	240	160	80 – 90	70	30	0.01
III	**1C**	240	160	80 – 90	60	40	0.01
IV	**1D**	240	160	80 – 90	50	50	0.01
V	**1E**	240	160	80 – 90	40	60	0.01

After completion of polymerization, the polymer product is distilled in a rotary evaporator under a vacuum to remove thinners. The resultant amount of the polymer product is practically quantitative.

3.5 Synthesis of MMA and MAA based Polymers [2A-2E]

The recipe for the copolymerization of synthesis of the SAP [2A-2E] based on MMA and MAA monomers are as follows:

Monomers	Methyl methacrylate (Monomer–a) and Methacrylic acid (Monomer-b)
Solvent	Toluene (Solvent-I) and Ethyl Acetate (Solvent-II) (Solvent mixture of I:II is 60:40)
Cross-linker	Triallylphosphite and Triallylamine In equivalent amounts
Initiator	Benzoyl peroxide
Inhibitor	Tert-butyl hydroperoxide (TBHP)

3.5.1 Polymerization procedure for SAPs 2A - 2E

Flask with five necks and a round bottom, with a capacity of one thousand milliliters, equipped with a nitrogen gas inlet tube, thermometer, a water condenser, a double helical impeller agitator, and an additive funnel. Two small pieces of porcelain chips were added to the flask, and then 200 milliliters of a solvent mixture consisting of 120 milliliters of toluene and 80 milliliters of ethyl acetate were used. Now, run the nitrogen gas through the contents of the flask in order to expel the air, and in the meantime, gradually heat the mixture of solvents to a temperature of forty degrees Celsius. While the additive funnel is being filled with fifty milliliters of monomer feed (including solvent, cross-linker, and a mixture of monomers), the feed is being supplied drop by drop at a rate of three milliliters per minute while a steady stream of nitrogen gas is being introduced. In order to start the polymerization process, you should gradually raise the temperature to 80 degrees Celsius after the addition of 0.01% benzoyl peroxide by weight. Following the conclusion of the addition of fifty milliliters of monomer feed, the remaining two hundred milliliters of monomer feed are poured into the additive funnel, and during the course of one and a half hours, they are added drop by drop. During the entirety of the polymerization process, 80- and 90-degrees Celsius temperature must be kept constant. In order to prevent the polymerization reaction from occurring, 0.01% by weight of TBHP is added at the conclusion of the procedure. Table 3.3 has a variety of different mixtures that can be used for a sequence of experiments.

Table 3.3: Chemicals and quantity are set to formulate polymers 2A-2E

Experiment	**Polymer**	**Solvent (ml)**		**Temperature (ºC)**	**Monomer (g)**		**Cross-linker (g)**
		I	**II**		**a**	**b**	
VI	**2A**	240	160	80 – 90	80	20	0.01
VII	**2B**	240	160	80 – 90	70	30	0.01
VIII	**2C**	240	160	80 – 90	60	40	0.01
IX	**2D**	240	160	80 – 90	50	50	0.01
X	**2E**	240	160	80 – 90	40	60	0.01

After completion of polymerization, the polymer product is distilled in a rotary evaporator under a vacuum to remove thinners. The resultant amount of the polymer product is practically quantitative.

3.6 Synthesis of AA and MMA based Polymers [3A-3E]

The following recipe is employed for the copolymerization of synthesis of the SAP [3A-3E].

Monomers	Acrylic acid (Monomer–a) and Methyl methacrylate (Monomer-b)
Inhibitor	Tert-butyl hydroperoxide (TBHP)
Initiator	Benzoyl peroxide
Cross-linker	Triallylphosphite and Triallylamine In equivalent amounts
Solvent	Toluene (Solvent-I) and Ethyl Acetate (Solvent-II) (Solvent mixture of I:II is 60:40)

3.6.1 Polymerization procedure for Polymers 3A - 3E

A round-bottom flask having five-necked with a capacity of one thousand milliliters is equipped with a nitrogen gas inlet tube, thermometer, a water condenser, and an additive funnel. Additionally, a dual spiral parallel impeller campaigner is also included. After transferring solvent mixture (200ml) with a volume-to-volume ratio of 60:40 (toluene-120 ml and ethyl acetate-80 ml), the porcelain chips viz. two small pieces are added to the flask using a transfer method. The solvent combination should now be heated gradually to a temperature of forty degrees Celsius, and then the nitrogen gas should be passed through the container in order to banish the air. While the additive funnel is being filled with fifty milliliters of monomer feed (including solvent, cross-linker, and a mixture of monomers), the feed is being supplied drop by drop at a rate of three milliliters per minute while a steady stream of nitrogen gas is being introduced. In the meantime, 0.01 percent by weight of benzoyl peroxide is added in order to kickstart the polymerization process, and the temperature is gradually elevated to 80 degrees

Celsius. In the event that the addition of fifty milliliters of monomer feed has been finished, the remaining two hundred milliliters of monomer feed are deposited into the additive funnel and added dropwise over the course of one and a half hours. During the entirety of the polymerization process, 80- and 90-degrees Celsius temperature must be kept constant. In order to prevent the polymerization reaction from occurring, 0.01% by weight of TBHP is added at the conclusion of the procedure. A variety of compositions for a sequence of experiments are documented in Table 3.4.

Table 3.4: Chemicals and quantity are set to formulate polymers 3A-3E

Experiment	**Polymer**	**Solvent (ml)**		**Temperature (ºC)**	**Monomer (g)**		**Cross-linker (g)**
		I	**II**		**a**	**b**	
XI	**3A**	240	160	80 – 90	80	20	0.01
XII	**3B**	240	160	80 – 90	70	30	0.01
XIII	**3C**	240	160	80 – 90	60	40	0.01
XIV	**3D**	240	160	80 – 90	50	50	0.01
XV	**3E**	240	160	80 – 90	40	60	0.01

After completion of polymerization, the polymer product is distilled in a rotary evaporator under a vacuum to remove thinners. The resultant amount of the polymer product is practically quantitative.

3.7 Synthesis of MAA and ACR based Polymers [4A-4E]

The various components used in the polymerization process for the s synthesis of the SAP [4A-4E] are as follows:

Monomers	Methacrylic acid (Monomer–a) and Acrylamide (Monomer-b)
Inhibitor	Tert-butyl hydroperoxide (TBHP)
Initiator	Benzoyl peroxide
Cross-linker	Triallylphosphite and Triallylamine In equivalent amounts

Solvent	Toluene (Solvent-I) and Ethyl Acetate (Solvent-II) (Solvent mixture of I:II is 60:40)

3.7.1 Polymerization procedure for Polymers 4A - 4E

Flask with five necks and a round bottom, with a capacity of one thousand milliliters, equipped with a nitrogen gas inlet tube, thermometer, a water condenser, a double helical impeller agitator, and an additive funnel. Two small pieces of porcelain chips were added to the flask, and then 200 milliliters of a solvent mixture consisting of 120 milliliters of toluene and 80 milliliters of ethyl acetate were used. Now, run the nitrogen gas through the contents of the flask in order to expel the air, and in the meantime, gradually heat the mixture of solvents to a temperature of forty degrees Celsius. While the additive funnel is being filled with fifty milliliters of monomer feed (including solvent, cross-linker, and a mixture of monomers), the feed is being supplied drop by drop at a rate of three milliliters per minute while a steady stream of nitrogen gas is being introduced. In order to start the polymerization process, you should gradually raise the temperature to 80 degrees Celsius after the addition of 0.01% benzoyl peroxide by weight. Following the conclusion of the addition of fifty milliliters of monomer feed, the remaining two hundred milliliters of monomer feed are poured into the additive funnel, and during the course of one and a half hours, they are added drop by drop. During the entirety of the polymerization process, 80- and 90-degrees Celsius temperature must be kept constant. In order to prevent the polymerization reaction from occurring, 0.01% by weight of TBHP is added at the conclusion of the procedure. Table 3.5 provides a variety of compositions for a succession of experiments.

Table 3.5: Chemicals and quantity are set to formulate polymers 4A-4E

Experiment	**Polymer**	**Solvent (ml)**		**Temperature (ºC)**	**Monomer (g)**		**Cross-linker (g)**
		I	**II**		**a**	**b**	
XVI	**4A**	240	160	80 – 90	80	20	0.01
XVII	**4B**	240	160	80 – 90	70	30	0.01

XVIII	**4C**	240	160	80 – 90	60	40	0.01
XIX	**4D**	240	160	80 – 90	50	50	0.01
XX	**4E**	240	160	80 – 90	40	60	0.01

After completion of polymerization, the polymer product is distilled in a rotary evaporator under a vacuum to remove thinners. The resultant amount of the polymer product is practically quantitative.

3.8 Synthesis of MMA and ACR based Polymers [5A-5E]

The recipe used in the polymerization process for the synthesis of the SAP [5A-5E] based on MMA and ACR monomers is as follows:

Monomers	Methyl methacrylate (Monomer–a) and Acrylamide (Monomer-b)
Inhibitor	Tert-butyl hydroperoxide (TBHP)
Initiator	Benzoyl peroxide
Cross-linker	Triallylphosphite and Triallylamine In equivalent amounts
Solvent	Toluene (Solvent-I) and Ethyl Acetate (Solvent-II) (Solvent mixture of I:II is 60:40)

3.8.1 Polymerization procedure for Polymers 5A - 5E

A round-bottom flask having five-necked with a capacity of one thousand milliliters is equipped with a nitrogen gas inlet tube, thermometer, a water condenser, and an additive funnel. Additionally, a dual spiral parallel impeller campaigner is also included. After transferring solvent mixture (200ml) with a volume-to-volume ratio of 60:40 (toluene-120 ml and ethyl acetate-80 ml), the porcelain chips viz. two small pieces are added to the flask using a transfer method. The solvent combination should now be heated gradually to a temperature of forty degrees Celsius, and then the nitrogen gas should be passed through the container in order to banish the air. While the additive funnel is being filled with fifty milliliters of monomer feed (including solvent, cross-linker, and a mixture of monomers), the feed is being supplied drop by drop at a rate of three milliliters per minute while a steady stream of nitrogen gas is being introduced. In the meantime, 0.01 percent by weight of benzoyl peroxide is added in order to kickstart the polymerization process, and the temperature is gradually elevated to 80 degrees Celsius. In the event that the addition of fifty milliliters of monomer feed has been finished, the remaining two hundred milliliters of monomer feed are deposited into the additive funnel and added dropwise over the course of one and a half hours. During the entirety of the polymerization process, 80- and 90-degrees Celsius temperature must be kept constant. In order to prevent the polymerization reaction from occurring, TBHP is added 0.01% by mass at the conclusion of the procedure. Table 3.6 lists different mixtures that can be used in a number of experiments.

Table 3.6: Chemicals and quantity are set to formulate polymers 5A-5E

Experiment	Polymer	Solvent (ml)		Temperature (°C)	Monomer (g)		Cross-linker (g)
		I	II		a	b	
XXI	**5A**	240	160	80 – 90	80	20	0.01
XXII	**5B**	240	160	80 – 90	70	30	0.01
XXIII	**5C**	240	160	80 – 90	60	40	0.01
XXIV	**5D**	240	160	80 – 90	50	50	0.01
XXV	**5E**	240	160	80 – 90	40	60	0.01

After completion of polymerization, the polymer product is distilled in a rotary evaporator under a vacuum to remove thinners. The resultant amount of the polymer product is practically quantitative.

3.9 Synthesis of AA and ACN based Polymers grafted with sodium acrylate [6A-6E]

The following recipe is employed for the copolymerization of synthesis of the SAP s [6A-6E].

Monomers	Acrylic acid (Monomer–a) and Acrylonitrile (Monomer-b)
Inhibitor	Tert-butyl hydroperoxide (TBHP)

Initiator	Benzoyl peroxide
Cross-linker	Triallylphosphite and Triallylamine In equivalent amounts
Solvent	Toluene (Solvent-I) and Ethyl Acetate (Solvent-II) (Solvent mixture of I:II is 60:40)
Grafting with	Sodium Acrylate (SA)

3.9.1 Polymerization procedure for Polymers 6A - 6E

The procedure followed was to synthesize a series of super absorbents from ACN, AA, and SA. An suitable amount of ACN was liquified in 30ml of ml of 1% concentrated CH_3COOH solution in a five-neck flask equipped with a funnel, mechanical stirrer, a nitrogen line, thermometer and reflux condenser with the capacity of 1000 ml. Nitrogen was passed for 30 minutes to remove the dissolved oxygen from the system and then added KPS to initiate ACN and generate radicals. After ten minutes, a solution was made up of AA, a certain amount of SA, and 10 ml of water. The polymerization process was finalized by keeping the water bath at 80°C for 3 hours. Sodium hydroxide solution was utilised to neutralised the prepared product, and then the prepared material was dry to reach its constant mass on the dish at 70°C set with oven. Table 3.7 contains the feed compositions of every sample.

Table 3.7: Chemicals and quantity are set to formulate polymers 6A-6E

Experiment	**Polymer**	**Solvent (ml)**		**Temperature (ºC)**	**Monomer (g)**		**Cross-linker (g)**
		I	**II**		**a**	**b**	
XXVI	**6A**	240	160	80 – 90	80	20	0.01
XXVII	**6B**	240	160	80 – 90	70	30	0.01
XXVIII	**6C**	240	160	80 – 90	60	40	0.01
XXIX	**6D**	240	160	80 – 90	50	50	0.01
XXX	**6E**	240	160	80 – 90	40	60	0.01

After completion of polymerization, the polymer product is distilled in a rotary evaporator under a vacuum to remove thinners. The resultant amount of the polymer product is practically quantitative.

3.10 Synthesis of MMA, ACR and ACN based Polymers [7A-7E]

Components used in the polymerization process for the synthesis of the polymeric thickeners [7A-7E] based on MAA, ACR and ACN monomers are as follows:

Monomers	Methacrylic acid (Monomer–a) Acrylamide (Monomer-b) and Acrylonitrile (Monomer-c)
Inhibitor	Tert-butyl hydroperoxide (TBHP)
Initiator	Benzoyl peroxide
Cross-linker	Triallylphosphite and Triallylamine In equivalent amounts
Solvent	Toluene (Solvent-I) and Ethyl Acetate (Solvent-II) (Solvent mixture of I:II is 60:40)

3.10.1 Polymerization procedure for Polymers-7A-7E

A round-bottom flask having five-necked with a capacity of one thousand milliliters is equipped with a nitrogen gas inlet tube, thermometer, a water condenser, and an additive funnel. Additionally, a dual spiral parallel impeller campaigner is also included. After transferring solvent mixture (200ml) with a volume-to-volume ratio of 60:40 (toluene-120 ml and ethyl acetate-80 ml), the porcelain chips viz. two small pieces are added to the flask using a transfer method. The solvent combination should now be heated gradually to a temperature of forty degrees Celsius, and then the nitrogen gas should be passed through the container in order to banish the air. While the additive funnel is being filled with fifty milliliters of monomer feed (including solvent, cross-linker, and a mixture of monomers), the feed is being supplied drop by drop at a rate of three milliliters per minute while a steady stream of nitrogen gas is being introduced. In the meantime, 0.01 percent by weight of benzoyl peroxide is added in order to kickstart the polymerization process, and the temperature is gradually elevated to 80 degrees Celsius. In the event that the addition of fifty milliliters of monomer feed has been finished, the remaining two hundred milliliters of monomer feed are deposited into the additive funnel and added dropwise over the course of one and a half hours. During the entirety of the polymerization process, 80- and 90-degrees Celsius temperature must be kept constant. In order to prevent the polymerization reaction from occurring, 0.01% by weight of TBHP is added at the conclusion of the procedure. Table 3.8 has a variety of different compositions that can be used for a series of tests.

Table 3.8: Chemicals and quantity are set to formulate polymers 7A-7E

Experiment	**Polymer**	**Solvent (ml)**		**Temperature (°C)**	**Monomer (g)**			**Cross Linker (g)**
		I	**II**		**a**	**b**	**c**	
XXXI	**7A**	240	160	80 – 90	0.01	20	1	0.01
XXXII	**7B**	240	160	80 – 90	0.01	30	1	0.01
XXXIII	**7C**	240	160	80 – 90	0.01	40	1	0.01
XXXIV	**7D**	240	160	80 – 90	0.01	50	1	0.01
XXXV	**7E**	240	160	80 – 90	0.01	60	1	0.01

After completion of polymerization, the polymer product is distilled in a rotary evaporator under a vacuum to remove thinners. The resultant amount of the polymer product is practically quantitative.

3.11Preparation of pH-stimuli microcapsules

In-situ polymerization method was applied for microencapsulating n-eicosane as a core and pH-responsive superabsorbent polymers, and the preparation steps were performed as shown in Figure 3.4.

Three steps correspond to the formulation shown in Table 3.9.

Table 3.9: Steps for Preparation of pH-Stimuli microcapsule

Steps	**Process**
1	• Dispersion of AA, DAM in oil, and n-eicosane in water for preparing the emulsion (oil-in-water) by adding ADS.

	• BP as initiator had been added for initiating the polymerization of AA and DAM, in oil and the O/W emulsions. • Temperature of the process is started from warm to 75°C.
2	• Monomers are added with the addition of $K_2S_2O_8$
3	• n-eicosane core covered with SAPs

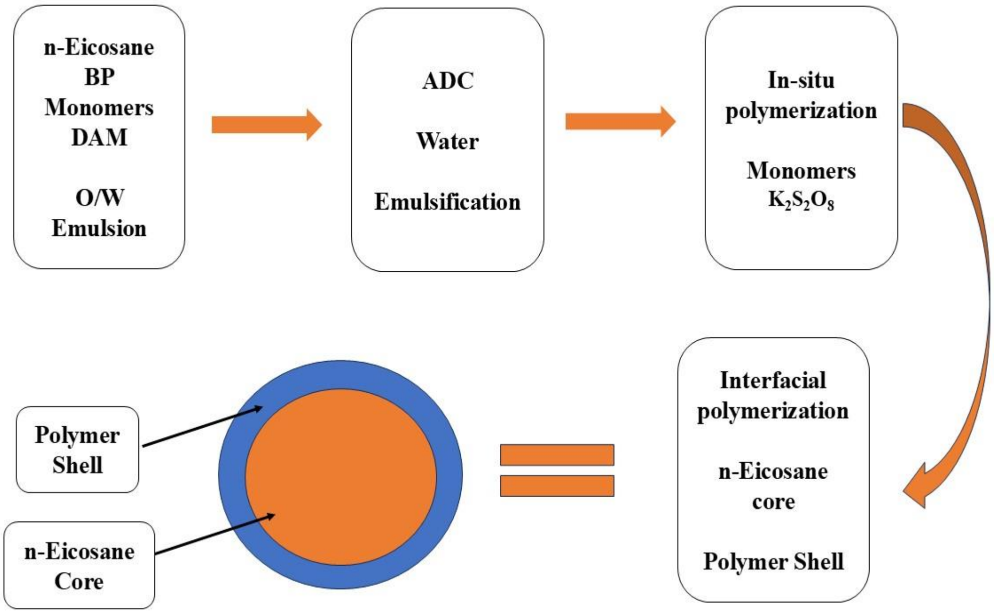

Figure 3.4: Synthetic route for preparing pH-responsive microcapsules

3.11.1 Preparation of pH-stimuli microcapsules with polymer [1C, 2C, 3D, 4B, 5B, 6C, 7C]

For the purpose of microencapsulation, the superabsorbent polymers that had been prepared were chosen on the basis of their characteristic powder form that was obtained after the dyeing process. Subsequently, the microencapsulation process was followed by in-situ polymerization of the quantity that was taken for the preparation of polymers 1C, 2C, 3D, 4B, 5B, 6C, and 7C. Table 3.10 provides a list of components that are recommended for the formulation of pH-sensitive microcapsules. The technique for the synthetic procedure involves injecting deionized water about 400 mL into a flask with round bottom having1000 mL capacity and five necks. In the flask, monomers, 1.0 grams of DAM, 0.1 grams of BP, and 20.0 grams of n-eicosane were added while the temperature was maintained at 55 °C and the mixture was stirred continuously without interruption.

Table 3.10: Chemicals and quantity are set to formulate pH-stimuli microcapsules [1C, 2C, 3D 4B, 5B, 6C, 7C]

Experiment	n-eicosane (g)	Monomer (g)	Initiator (BP and $K_2S_2O_8$) (g)	Diallyl maleate (DAM) (g)	Ammonium Dodecyl Sulfate (ADS) (g)	Temper-ature (ºC)
XXXVI (1C)	20	18 AA 12 ACN	0.1 0.03	1.0	2.5	55 - 82
XXXVII (2C)	20	18 MMA 12 MAA	0.1 0.03	1.0	2.5	55 - 82
XXXVIII (3D)	20	6 AA 24 MMA	0.1 0.03	1.0	2.5	55 - 82
XXXIX (4B)	20	24 MAA 6 ACR	0.1 0.03	1.0	2.5	55 - 82
XL (5B)	20	24 MMA 6 ACR	0.1 0.03	1.0	2.5	55 - 82
XLI (6C)	20	18 AA 12 ACN	0.1 0.03	1.0	2.5	55 - 82
XLII (7C)	20	18 MAA 11 ACR 1 ACN	0.1 0.03	1.0	2.5	55 - 82

Following the addition of 2.5 grams of ADS to the combination described above, the mixture was allowed to stand at a temperature of 55 °C for 2.5 hours while nitrogen was removed. During the process of producing the stable O/W emulsion, the temperature progressively climbed to 75 °C. This allowed for free radical polymerization to occur in the existence of nitrogen gas. The combination was then permitted to remain at the same temperature for three hours while being continuously stirred. The following system was supplemented with a mixture of monomers and 0.03 g $K_2S_2O_8$ after three hours. To achieve interfacial polymerization, the flask was heated to 82 °C for 2.5 hours while swirling continuously after mixing. The white granules, which are pH-responsive polymer microcapsules, were recovered by filtration after washing the yield with deionized water at the end of the reaction. Room temperature is used for drying them.

3.12 Characterization

3.12.1 Thermogravimetric analysis (TGA)

TGA gives the information about mass loss of a product as the effect of temperature. The sample is analyzed in well-defined conditions of the environment and pre-programmed tuning is done with temperature used to illustrate a widespread diversity of materials, also DSC, the most commonly used technique, compared to TGA provides supplementary and complementary information. This measurement is mostly done for solid materials and the sample should made powdered and spread in the crucible for ideally small content. The initial mass of the sample has been taken as 8 mg. Atmosphere of the furnace can be covered with nitrogen, air, or inert gas. In this experiment, TGA offers an empirical evaluation of the amount of mass changes and its rate (velocity) in a sample as a function of time or temperature under a regulated atmosphere. This technique is employed to assess the characteristics of substances and their ability to oxidize and thermally stabilization. Mass loss can be evaluated as a result of dehydration, breakdown, and oxidation of the material over time and temperature. It is specifically utilized for the examination of polymers, including composites, thermosets, adhesive, thermoplastics, elastomers, fibers, films, paints, biopolymers, and coatings. Distinct thermogravimetric curves are provided for certain materials and chemical substances, resulting from distinctive patterns of physicochemical reactions that transpire within defined intervals of temperature and heating rates. The distinctive traits are associated with the molecular composition of the sample. The TGA curves yield critical insights into the thermal behavior of polymers, which can be utilized to choose substances for specific applications, anticipate performance of goods, and enhance the overall quality of the product (Menczel & Prime, 2009; Mortezaeikia et al., 2021; Ng et al., 2018; Nurazzi et al., 2021). TGA includes measuring the sample properties while the sample temperature is computer program-controlled as shown in Figure 3.5

Set the nitrogen and oxygen vapor current rates to deliver the heating rate of 20 K/min. Place 1.0 g of sample polymer material in the sample container and increase the heater temperature. Set the preliminary mass (1.0 g) interpretation to 100%, and then pledge the heating system. TGA counts weight alteration in the sample, and it can be used to spot oxidation, decomposition, evaporation, and other effects occurs due to temperature alteration which is responsible for alteration in mass. Data are recorded as a plot of out of a hundred mass loss against temperature (Mortezaeikia et al., 2021; Nurazzi et al., 2021).

TGA investigations of all SAPs and microencapsulated SAPs were carried out with 1.0 g of initial sample material at the heating rate of 20 K/min using Parkin Elmer TGA model 4000 (Menczel & Prime, 2009).

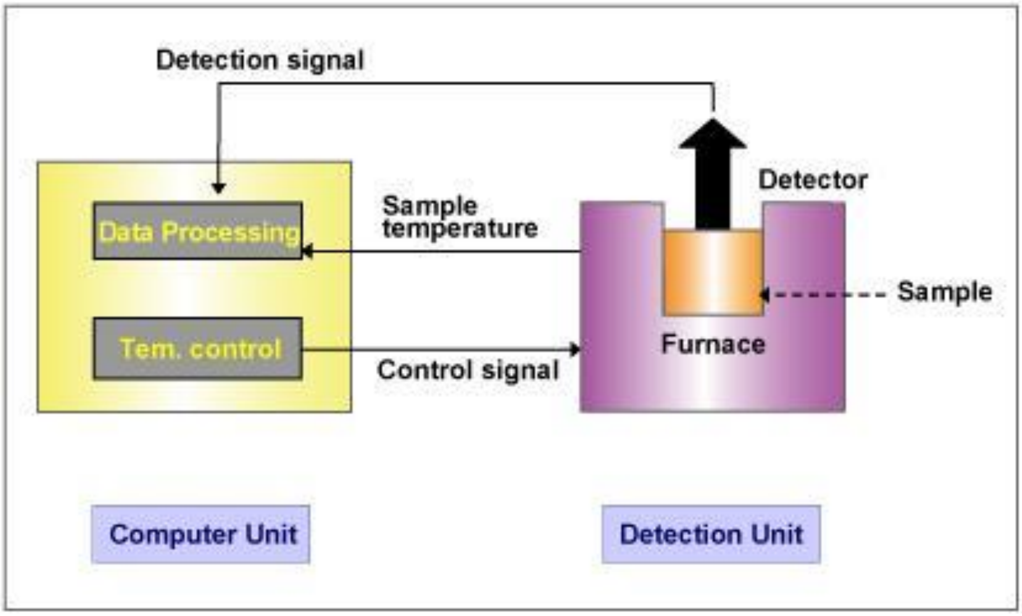

Figure 3.5: Block diagram of an instrument for TGA

3.12.2 FT-IR Spectrometric Studies of Polymers

Knowing the kinds of sample cells or holders is vital for determining the IR FTIR spectrum of a chemical. It will obtain a better and more accurate infrared spectrum with the right sample cell or holder. Depending on the sample's condition, a different method of preparation was needed. For example, salt plates made of potassium bromide or sodium chloride can be used to hold liquid samples like drug solutions. Gently squeezing the plates can cause a little film to form from a single drop of liquid. The spectrometer is then fitted with a holder that receives the matched plates. Since no solvent is employed in this approach, the resulting spectrum is commonly called a clean spectrum. In addition, three standard procedures exist for getting solid samples ready for Fourier transform infrared spectroscopy (Mohamed et al., 2017). Crushing the prepared product till it becomes a finely solid powder is one way to get the solid powder from a membrane sample. Under high pressure at approximately 12,000 psi for 1 to 2 minutes, the mixture is pressed is the next step after mixing the sample powder with powdered potassium bromide (KBr). To create a highly uniform KBr pellet, 1:100 is the ratio of powder of sample to KBr. The final KBr pellet can be placed into a specific holder within the FTIR spectrometer (Chércoles Asensio et al., 2009).

The insolubility of the polymeric thickener that has been prepared renders its characterization exceedingly challenging. The NMR analysis of these prepared polymers was nearly impossible; though, FTIR bands were noted by making the polymer sample's film on the glass using its latex surface, and subsequently detaching it from the same base. To analyse the structure of chemicals and all prepared polymers configuration PerkinElmer Spectrum^{TM} 3 FT-IR spectrometer was used.

3.12.3 SEM and Particle Size Analysis

The profile of the microcapsules was examined applying scanning electron microscopy right after they were spray-dried. Powder particles were fastened to an aluminum stub using double-sided sticky tape. The JSM 7001F scanning electron microscope (SEM) was employed to analyse the morphology of pH-responsive microcapsules. When doing the exams, the scanning electron microscope has been utilized, and a hastening voltage of 10 kV was utilized. Additionally, magnifications of 600× were utilized (XIE et al., 2010; H. Zhao et al., 2020).

Nano Measure^{TM} software was employed to measure the mean size of particle and distribution of particle size of the pH-sensitive microcapsule. The particles that were suspended in alcohol were used to conduct a quadruplicate analysis of the determination of the average particle size as well as the particle size distribution. Prior to the measurements being taken, the samples that were suspended were sonicated for one minute, and the analyses were carried out while the samples were being stirred continuously (Tian et al., 2020; Xu et al., 2020; R. Zhao et al., 2008).

3.13 Application of prepared SAPs/mSAPs to Sanitary Napkin

Five layers have been formulated and fabricated for preparing the pad, including a self-adhesive tape attached to the undergarment. The tape is covered using cotton coated with paraffin wax to inhibit leakage. The third layer is made with developed super adsorbent polymer. Again, the thin cotton has been pressed and attached to the third layer. Outermost sheet of the pad has been made with cotton coated with formulated microencapsulated SAP (Figure 3.6).

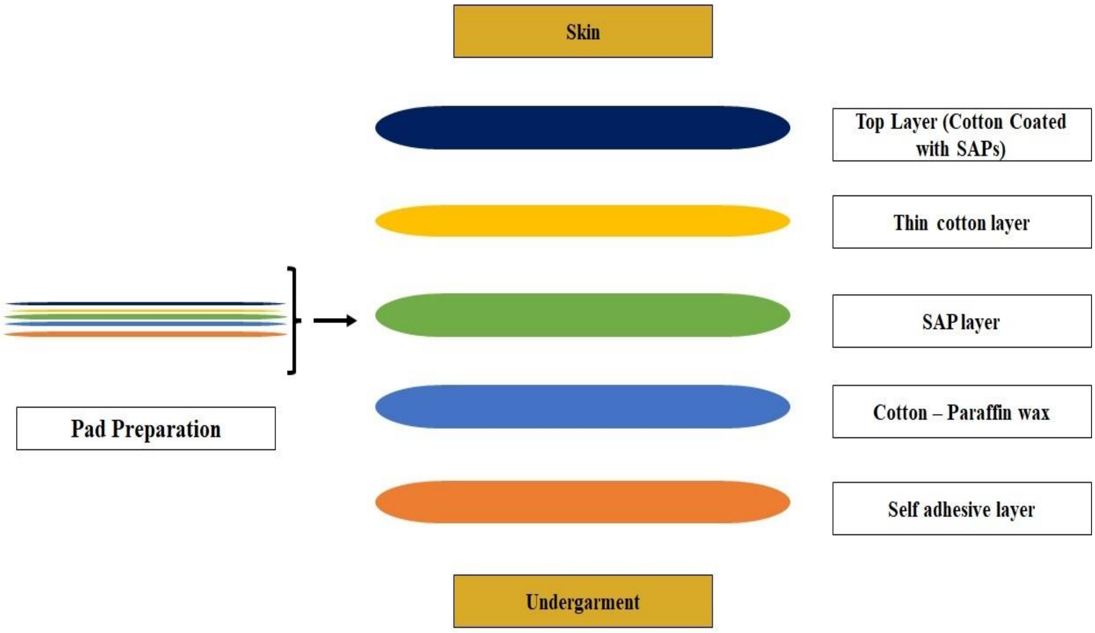

Figure 3.6: Pad preparation with layers

The uppermost sheet of the prepared sanitary napkin was dipped in pH-sensitive dispersed solution (20 g) and then padding was performed utilising two-bowl padding mangle as shown in Figure 3.7. The padded layer was once more immersed in the SAP dispersal and subsequently cushioned. The procedure is recurred thrice to create a coating of SAP atop the sanitary napkin. All prepared SAP samples were covered using the layer-by-layer approach. Ultimately, all stratified napkins were subjected to baking at 82°C for a duration of 3 minutes. The said process was repeated three times. The samples were prepared as CS (control sample) for n-eicosane, SAP-1C, SAP-2C, SAP-3D, SAP-4B, SAP-5B, SAP-6C and SAP-7C for super absorbent polymer 1C, 2C, 3D, 4B, 5B, 6C, and 7C; and mSAP-1C, mSAP-2C, mSAP-3D, mSAP-4B, mSAP-5B, mSAP-6C and mSAP-7C pH-responsive microencapsulated polymers 1C, 2C, 3D, 4B, 5B, 6C, and 7C respectively selected on the basis of free swell capacity and absorption under load test received the highest values.

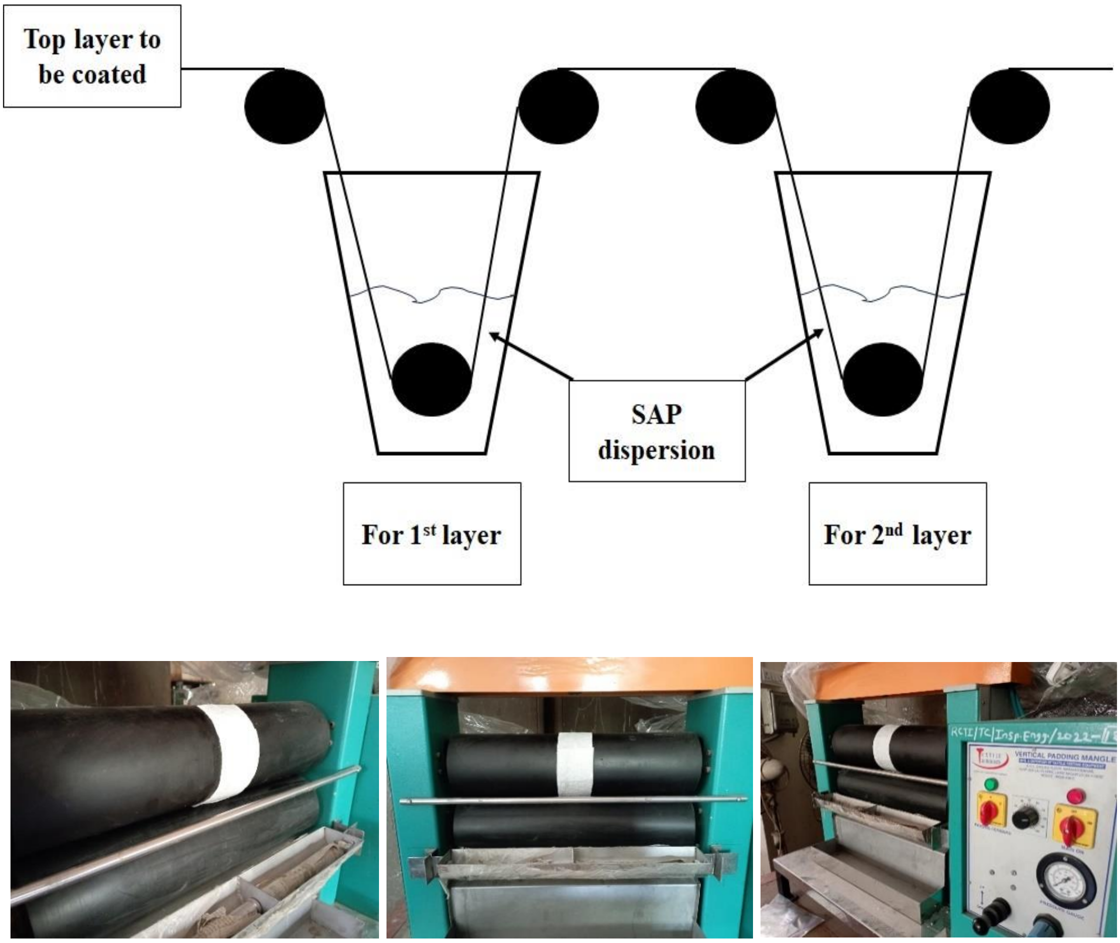

Figure 3.7: Layer-by-layer application of SAP on the top layer of sanitary napkin

3.14 Free Swell Capacity (FSC)

It is generally accepted that when the phrases absorbency or swelling are utilised without describing its circumstances, it indicates that the sample is absorbing purified water while it is spontaneously swollen, which means that there is no weight placed on the sample that is being tested. There are a few straightforward approaches to free absorbency testing, the majority of which are determined by the quantity of the sample that is readily available, the absorbency level of the sample, as well as the precision and accuracy of the aforementioned strategies. Three methods for the free swelling testing are (1) the Centrifuge method the, (2) the Sieve method, and (3) Tea-bag method. The tea-beg method is utilized for measuring the swelling of samples prepared and applied on sanitary napkins.

This test result is used to determine the quantity of distilled liquid that is captivated by the sample while it is in a condition in which it is freely swollen. According to (EDANA, 2019b; Qin, 2016), the tea-bag method is the solution that offers the greatest convenience and quickness when it comes to analysing samples that are in restricted quantities. Following the preparation of the samples, they were placed inside of a teabag and water-logged in either a saline solution or water for a period of one hour. As soon as the tea bag had reached its maximum capacity for swelling, it was weighed once more, and its swelling capacity was determined.

$$Fa\ (\frac{g}{g}) = \frac{(\mathrm{T2} - \mathrm{T1})}{\mathrm{T1}} \tag{3.1}$$

$$FSC\ \left(\frac{g}{g}\right) = \frac{(\mathrm{W2}-(\mathrm{W1} - \mathrm{Fa})-\mathrm{W1}-\mathrm{W0})}{\mathrm{W0}} \tag{3.2}$$

Where, T_1 and T_2 are the tea bag absorption factor, W_0 is prepared sample of sanitary napkin's weight, W_1 is dry empty tea bag weight, and W_2 is the swollen sample's weight.

3.15 Absorption under load (AUL)

Typically, the absorbency under load (AUL) data is provided by industrial SAP manufacturers in the patent literature as well as in the technical data sheets. When the word AUL is applied short of defining its swelling area, it indicates that the testing sample is under pressure by approximately loads (which are typically definite to be pressures of 0.3 or 0.9 psi). This means that the sample is absorbing a solution of 0.9% sodium chloride. A typical AUL tester is a device that is not only straightforward but also of high quality. It consists of a macro-porous sintered glass screen plate that is located inside a petri saucer. The SAP sample that has been dried and weighed is then placed in a uniform manner on the exterior of the polyester gauze that is mounted on the sintered crystal. A dense load in the shape of a cylinder is applied on dry SAP particles while the particles are allowed to freely slide inside a glass cylinder.

The volume of SAP samples to absorb liquids subjected to precise weights is evaluated using the AUL value. For the purpose of producing an even bed, a sample of SAP weighing 0.9 grams was allowed to plug the sieve screen. An aluminum piston was positioned on the bed, and it was placed there. A total of W1 was the weight of the assembly. Following the settlement of a tap water, permeable disk in a platter, and saline solution were added to the mixture (figure 3.8). After placing the whole assemblage on the screen paper, a weight of 0.3 pounds per square inch was applied to it (Bachra et al., 2020; EDANA, 2019a). It was decided that the gathering could continue in its existing state for a period of one hour. Following the completion of the predetermined time period, the assemblage was elevated, the weight was detached, and the piston was reweighted as W2. The AUL was determined by applying the equation that is presented below.:

$$AUL\ \left(\frac{g}{g}\right) = \frac{(W2 - W1)}{W1} \tag{3.3}$$

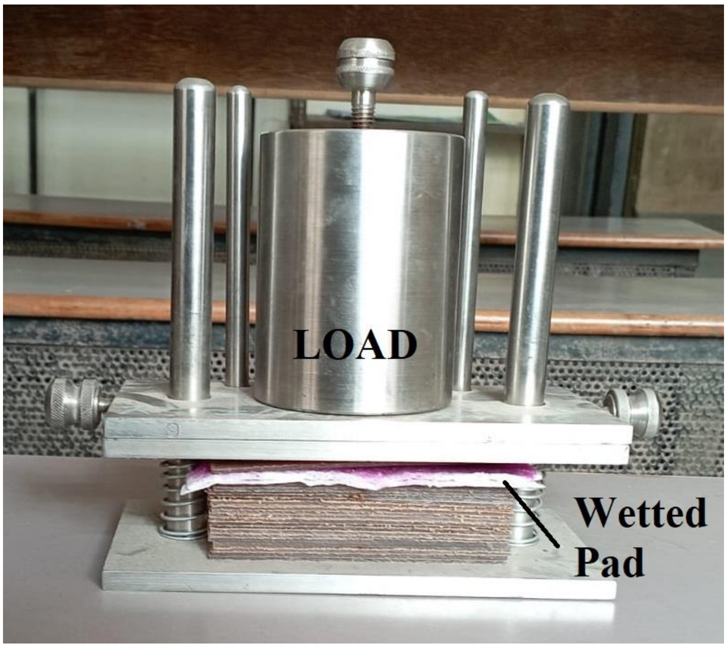

Figure 3.8: Absorbent capacity measurement assembly with load

3.16 Absorbent capacity

Absorbnecy speed and absorbency were measured for the measurement of absorbent capacity of the superabsorbent polymer (SAP), and is measured in accordance with the standard test technique outlined in EAS 96:2008-Annex C. A dry mass of the SAP-coated napkin was measured and noted before the addition of 0.9% saline solution till the pad reached saturation. Following the completion of the saturation process, a 3.4 kilograms mass was positioned on the napkin, as depicted in Figure 3.9. In order to remove any surplus fluid, we made use of filter paper, and after that, we determined the weight of the pad. For the purpose of this investigation, for an alternative to blood, a briny solution was used (Mukhila & Amrutha, 2023; Yadav et al., 2016). The formula that is offered in equation 3.4 was applied for the purpose of getting measurements.

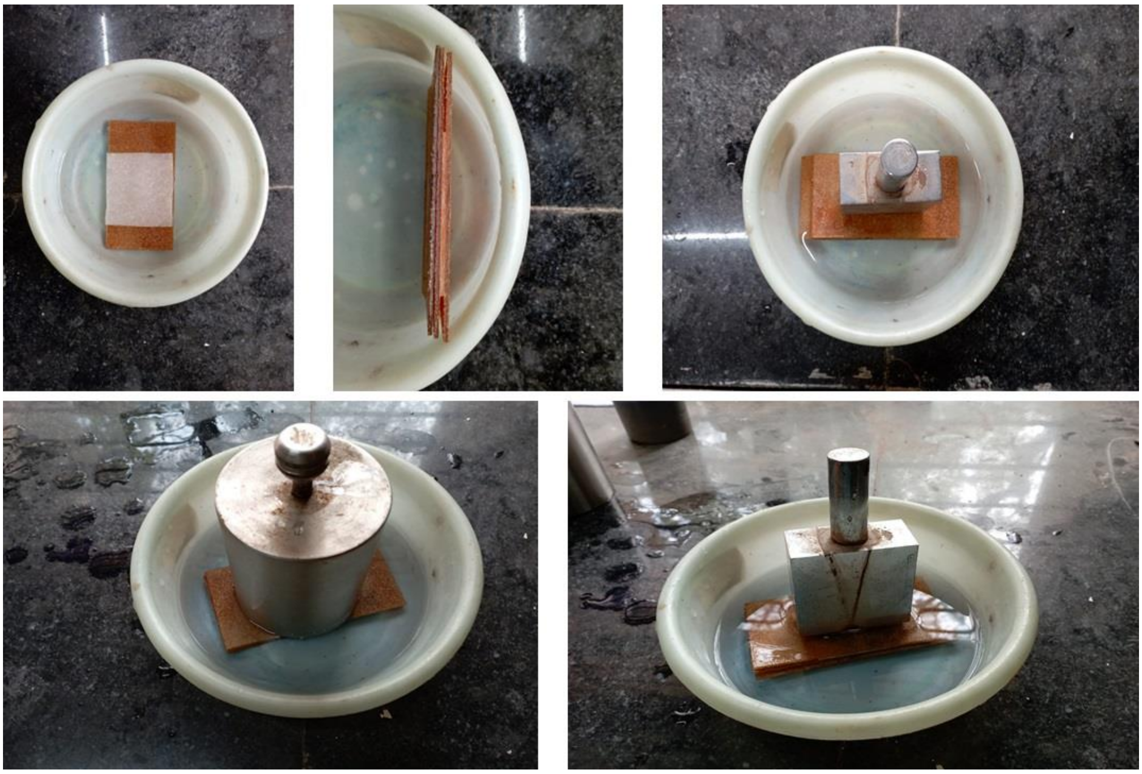

Figure 3.9: Absorption capacity measurement assembly setup

$$Absorption\ Capactiy\ (A) = (W - X)g \tag{3.4}$$

Where, X = Sanitary napkin dry weight (g)

W = weight of the sanitary napkin (g) after fullness

3.17 Wet back

The moisture retention or rewet of a sanitary napkin which was prepared during the study, evaluated in compliance with ISO/IEC 17025. This process entails administering 5 millilitres of a 0.9% saline solution onto the central part of the napkin and allowing it to sit for one minute. The aforementioned steps are executed. The subsequent stage involves positioning three grams of dehydrated screen paper at the centre of the apparatus for a duration of fifteen seconds. This will ascertain the mass of the first screen paper, and reiterating the method will ascertain the weight of the second filter paper. Each sample was exposed to typical atmospheric conditions, comprising a relative humidity of 65 $\pm$ 2 %. And temperature of 20 $\pm$ 2 °C. The weight differential between the first and second filter paper was recorded for each sample (EDANA, 2018).

3.18 Strikethrough

When conducting the experiment, a tiny volume of saline solution with a concentration of 0.9% was used. The objective of the test was to ascertain the rate at which a solitary drop of liquid traverses' specimens of sanitary napkins. To conduct this trial, a droplet of the test fluid was allowed to drop onto the sanitary napkin sample, and the proportion of fluid diffusion into the napkin was meticulously observed. A measurement was conducted to determine the duration required aimed at the body fluid substitute to permeate from the outermost film of the napkin to the innermost sheet, in order to assess the pad's efficacy. The drop was meticulously examined until the test fluid manifested on the napkin sample as a cloudy spot, and the napkin was monitored for the similar duration as the drop (EDANA, 2018)(figure 3.10).

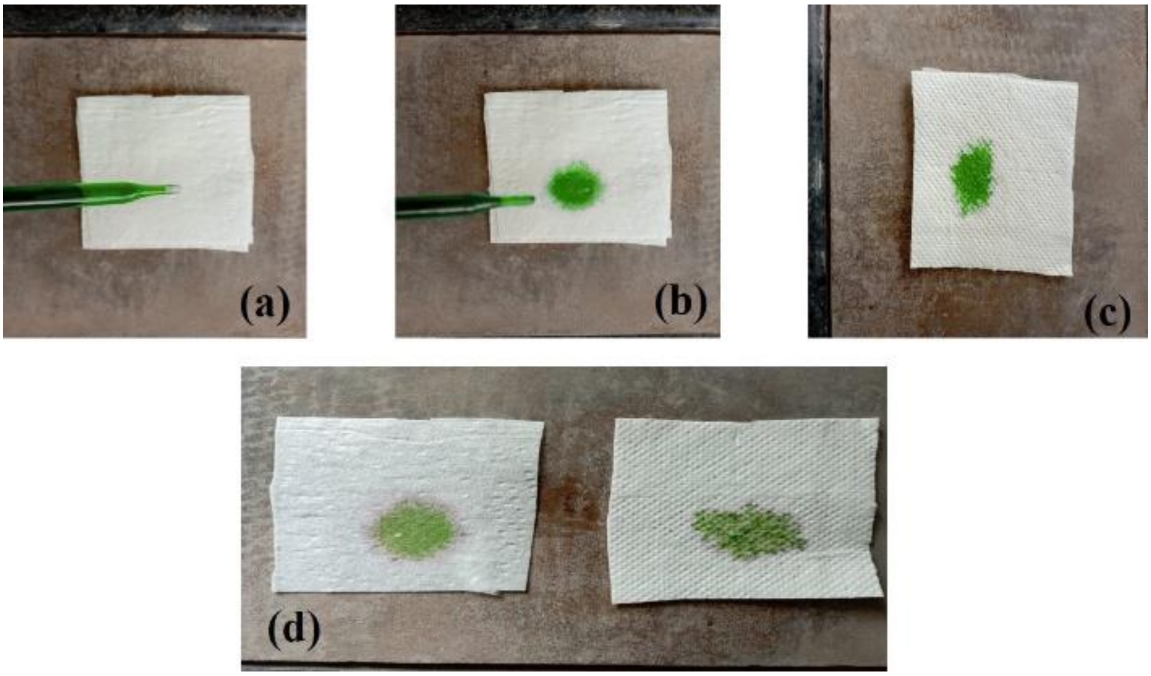

Figure 3.10: Strike through measurement (a) Assembly set up, (b) Drop at the top layer, (c) striking through top to inner layer, and (d) top and inner layer

3.19 Wiking height test

For vertical wicking measurement, column experiment was performed, which was headed by the AATCC TM 197-2011 normal, an evaluation of the various samples with the absorption distribution was carried out. The purpose of this examination technique is to measure the capacity of vertically associated napkin specimens to passage fluid through and/or along them through the act of capillary action. This test method is also applicable to woven, knitted, and non-woven textiles. For the purpose of the wicking test, a test sample measuring 5 centimeters by 18 centimeters is taken, and a mark is made about one centimeter from the bottom. After that, a piece measuring one centimeter is submerged in a direct dye solution containing one percent for a period of five minutes. After that, the distance that the colored solution travels beyond the one-centimeter mark is measured in millimeters (Shibly et al., 2021).

3.20 Determination of pH of the top layer

pH meter was utilised to measure the saline solution’s pH (figure 3.11), and that was used to determine the pH. An amount of 0.5 grams of the taster was gradually added to a saline solution that was 100 milliliters in volume while the mixture was continually stirred. A measurement of the pH was taken after the solution had had time to settle. (EDANA, 2019b).

Figure 3.11: pH meter

3.21 Antimicrobial activity

SAP/mSAP-coated samples were spread out on a nutrient culture medium plate and then incubated for 24 to forty-eight hours at 37 ℃ in order to determine whether or not they have antibacterial activity contrary to gram-negative E. coli. During the course of the investigation into the action against bacteria, the diameter of the inhibitory region was measured. (Hassan Shibly et al., 2019). Following the contact of organisms with the samples that had been prepared, a percentage decrease was determined for the purpose of antimicrobial analysis by utilizing equation 3.5.

$$\% \, Reduction = \frac{(\mathrm{X}-\mathrm{Y})}{\mathrm{X}} \mathrm{X}\ 100 \tag{3.5}$$

Where, the cells X and Y are the ones that were able to survive in the CFU/ml unit that contained the prepared samples, as well as the control cell that was not coated with SAP.

3.22 Physical Testing of SAP/mSAP-coated samples

The general physical qualities of sanitary napkins include weight, length, thickness, and width, which are evaluated in agreement with ISO/IEC 17025. The designed sanitary napkin is assessed based on the commercial standards for length, width, and weight. Ana Thick, a thickness tester (Model MAG-C1001, India, MAG) Analog Thickness Gauge has a dimension measure range of 0.010–10.00 mm and it is utilized to ascertain the width of various napkins in accordance with the IS 7702 test procedure. A range of menstruation pads, comprising SAPs/mSAPs coated and commercially available CAs, underwent the IS 7702 test to ascertain their thickness. The MAG-C1001 thickness tester was employed for this purpose with a range of 0.010 to 10.00 millimeters (Shibly et al., 2021).

3.23 Safety Assurance

The chemical safety of coated sanitary napkins has been evaluated by the exposure assessment. The three types of skin contact that have been identified are (i) direct skin contact (top sheet), (ii) absorbent core (indirect skin contact), and (iii) backsheet which has negligible skin contact, as shown in figure 3.12.

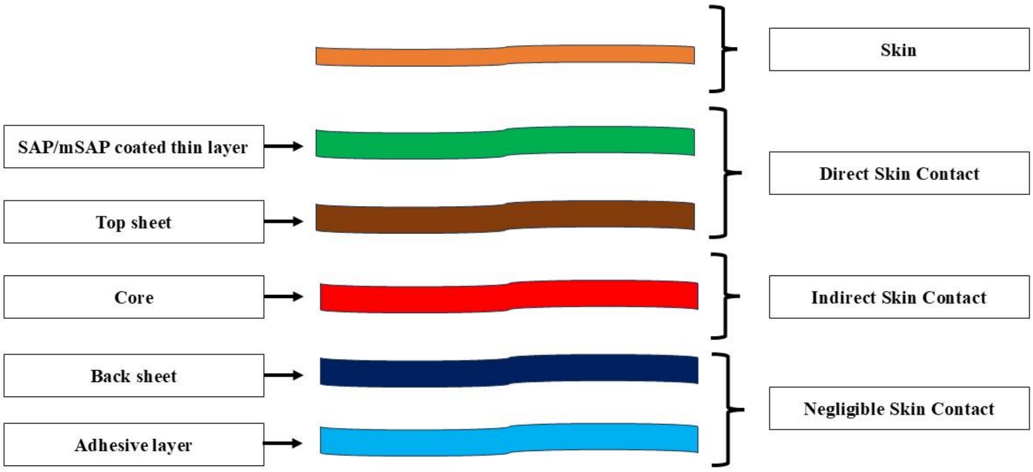

Figure 3.12: Sanitary napkin apparatuses with consumer exposure

3.23.1 Direct skin contact

Additionally, the emollient and the SAP/mSAP coated top sheet come into touch with the skin; however, solitary a share of the material will transport while the product is being used. The transfer of emollient to the skin was shown to be less than twenty percent in replicating tests (Farage, 2010). A successive approximation of the extreme quantity of the functional emollient that will pass transfer to the body was adopted, and this 20% number was chosen as the highest possible value. A conservative estimation of the maximum skin transmission of short molecular mass top sheet components that are supposed to stay on the napkin but it might be transport during the application and so, require a conservative assessment is another application of this technique(Woeller & Hochwalt, 2015).

3.23.2 Negligible skin contact

The back sheet and adhesive exhibit minimal skin contact and no exposure via rewetting. The product integrity standard and analytical leachability assessment substantiate the presumption of little skin exposure to sanitary napkins.

3.23.3 Quantitative exposure assessment

The parameters that were utilized in the quantitative exposure evaluation are suggested in Table 3.11, and they are based on the assumptions that were made in sections 3.23.1 to 3.23.2 for the purpose of gaining an understanding of menstruation behaviours and routines. Systematic exposures are assessed based on the body weight of the individual; the amount per unit zone of skin exposed is the essential experience for determining the risk of skin sensitization. Equations 3.6 and 3.7 (Woeller & Hochwalt, 2015)have been utilized in order to conduct an analysis of quantitative exposure.

Table 3.11: Parameters for measurable exposure valuation of sanitary napkin

Parameters	Values and Units
Weight of raw materials (RMw)	g/sanitary napkin
Constituents' concentration (CC)	% constituent/100 g sanitary napkin
Regularity of use (RU)	5 sanitary napkins/day
Transfer to skin (TS)	20% of superficial functional components
Rewet transfer (RE)	5% of absorbed fluid
Dermal absorption (DA)	100%
Duration of exposure (DE)	100%
Body weight (Female) (BW)	50 kg (assumed)
Surface area of napkin (SA)	cm^2

$$\text{Systemic exposure}\left(\frac{\frac{\mu\mathrm{k}}{\mathrm{kg}}}{\mathrm{day}}\right) = \mathrm{RMw} \times \mathrm{CC} \times \mathrm{RU} \times \mathrm{DE} \times \mathrm{TS} \times \mathrm{DA} \times \mathrm{BW} \times 10^6 \quad (3.6)$$

$$\text{Dermal exposure}\left(\frac{\frac{\mu\mathrm{g}}{\mathrm{cm}^2}}{\mathrm{day}}\right) = \mathrm{RMw} \times \mathrm{CC} \times \mathrm{RU} \times \mathrm{DE} \times \mathrm{TS} \times \mathrm{DA} \times \mathrm{SA} \times 10^6 \quad (3.7)$$

For the analysis, OMES (Overall Mean Erythema Scores) was used only for irritation test. For patch test (figure 3.13), protocol for standard predictive annoyance was used. The test describes utilisation of 4 cm^2 of SAPs/mSAPs coated sanitary napkins section and dampened with 0.2-0.4 mL of physiological brine. The prepared samples were utilised at the back of arm daily for 21, 5, 4 and 1 days, and the skin reactions were examining visually after removing the applied product. Visual assessment performed under 100-W incandescent bulb, and standardized grading scale used as 0 – 4 (figure

3.14). Where, 0 suggests apparent cutaneous presence is nil, and 4 suggests moderate-to-severe spreading erythema (Botham, 2004; Richard & James, 1982; Woeller & Hochwalt, 2015).

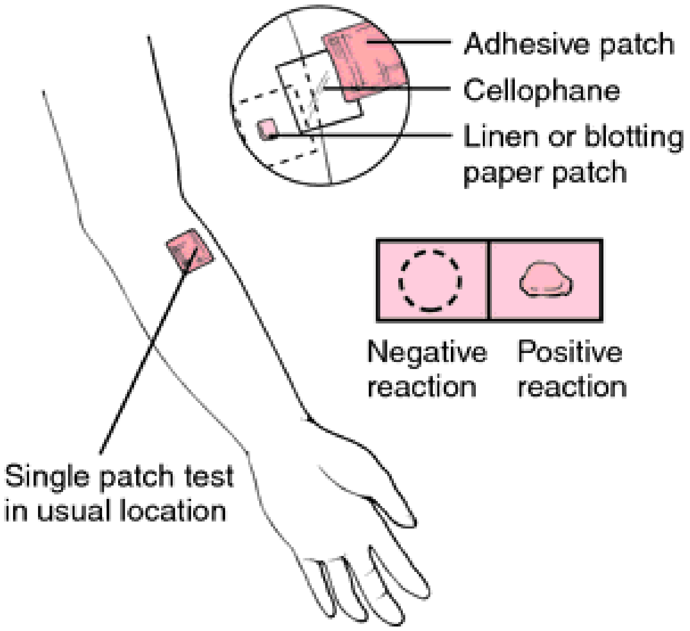

Figure 3.13: Patch test

Figure 3.14: Negative and Positive reaction grading for patch test © *2021 Elsevier Inc(Garg et al., 2021)*

The standard Shelanski type Human Repeated Insult Patch Test (HRPIT) was used to analyse the irritation for nine 24 hours plosive squares at exterior facet of upper arm (single site) through 24 hours rest between. Before utilising the prepared products at single site, the site was graded, and then the grading was performed after 24- to 48-hourse of removal the products from the site. Visual assessment was done with 0 – 3 scaling, where, 0 denotes visible reaction as nil, and 3 indicates erythema (Marzulli & Maibach, 2019).

3.24 Effluent Analysis

Water sample (SAPs/mSAPs) was prepared by dipping the used sanitary napkin (by menstruating women) in 1000 ml of water. Also, sanitary napkins available in markets (CA1 to CA4) were analysed by dipping them in 1000 ml water.

3.24.1 Hardness (mg/l)

Hardness of the water was also measured for the water in which prepared napkins and market available napkins were dipped. The procedure was followed as per the (IS 3025 (Part 21), 2009) method.

3.24.2 Alkalinity and Chloride (mg/l)

Above prepared water (1000 ml) in which market available used napkins of different companies dipped and also napkins prepared in present work were dipped analysed for alkalinity and chloride by ISO 9963-1:1994 and ISO 9297:2000 respectively (ISO 9297, 2000; ISO 9963-1, 1994).

3.24.3 COD and BOD

Chemical Oxygen Demand (COD) and Biological Oxygen Demand (BOD) of the above prepared samples and market available samples measured by applying IS 3025 (Part 58): 2006 and IS 3025 (Part 44): 1993 test method respectively (IS 3025 (Part 44), 1993; IS 3025 (Part 58), 2006)

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