

A SYNOPSIS
OF
THE THESIS ENTITLED

**“Design and Fabrication of pH Responsive Microencapsulated
Phase Change Materials for Textile Hygiene Applications”**

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Submitted by

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SYNOPSIS

Many women do not realize the vagina is an ecosystem that requires a balanced environment to remain healthy; and events like menstruation, sex and hormonal changes can throw off that balance. Healthy pH and beneficial lactobacillus are keys to reducing the risk of vaginal issues. pH refers to the vagina's acidity level. A vaginal pH of 3.5 to 4.5 indicates that the vagina is populated by good bacteria (lactobacilli) without overgrowth of the bad bacteria that can cause odor and irritation. An elevated pH means that the healthy flora of the vagina may struggle to survive, while harmful bacteria thrive. Women experience many of the triggers for elevated pH on a regular basis are as follows:

1. Period : The pH of blood is 7.4, which is much more alkaline than normal vaginal pH (3.5 to 4.5), which can lead to elevated vaginal pH
2. Sex: Semen has a pH of 7.1 to 8 and can increase vaginal pH
3. Hormones: Pregnancy, menopause and your normal monthly cycle can cause hormone changes that alter vaginal pH

Taking control of pH means taking control of the natural vaginal environment, putting an end to unpleasant odor, discomfort and itching.

Diaper: A piece of towelling or other absorbent material wrapped round a feminine vaginal and baby's bottom; and between its legs to absorb and retain urine and faeces; a nappy.

Vaginal pH: A normal vaginal pH is between 3.8 and 4.5. A pH level within this range can help to keep bacterial and fungal infections at bay. Lactobacilli bacteria live in the vagina and secrete lactic acid and hydrogen peroxide, which give the vagina its acidic pH level. Vaginal pH can change throughout a person's lifetime.

pH responsive material: pH responsive polymers are materials which will respond to the changes in the pH of the surrounding medium by varying their dimensions. Materials may swell, collapse, or change depending on the pH of their environment.

Microencapsulation: Microencapsulation is a process in which tiny particles or droplets are surrounded by a coating to give small capsules, with useful properties. In general, it is used to incorporate food ingredients, enzymes, cells or other materials on a micro metric scale.

Phase change material: A phase change material is a substance which releases/absorbs sufficient energy at phase transition to provide useful heat/cooling. Generally the transition will be from one of the first two fundamental states of matter - solid and liquid - to the other.

Polymerisation: Polymerisation, is a process of reacting monomer molecules together in a chemical reaction to form polymer chains or three-dimensional networks. There are many forms of polymerization and different systems exist to categorize them.

Hygiene Textile: Cloths or other fabric items used during food preparation, or for cleaning the toilet or cleaning up material such as feces or vomit are a particular risk.

Feminine hygiene and baby care materials are diapers, skin protective towelettes for children, make-up remover towels, covet wipes, baby wipes, nappy (Diaper) liners, dry wipes (antimicrobial), disposable baby bibs, baby pillow and changing mats, baby blanket, dermo-protective childrens' towels, etc. The basic fundamental structure is not changed since many years. So, this one is the new technological development tend to synthesis the pH responsive polymer, which can be applied on hygiene textile materials.

In literature review, the thermo-responsive phase change polymers are mostly studied. The work has been carried out on cotton and nonwoven or the study of effect of the application of hydrogels, thrho-responsive polymers and somewhat about pH-responsive polymers also. Generally, pH-responsive polymers are applied in drug, gene delivery carriers and biosensors. pH-sensitive hydrogels are basically polyelectrolytes of either charge and

operate by widening of the mesh sizes of their network resulting from the repulsive forces developed due to ionization or protonation of the constituent polymer chains. Whereas cationic polyelectrolyte like chitosan work well in the low pH environment of the stomach.

In the case of feminine vaginal or the bottom of the baby, the diapers available in the market having more absorption, but still they don't passage the pH-related properties. Though some are thermo-responsive also. Therefore, uneasiness, rashes, unbalanced pH happen to the female and baby.

The investigation on the design and fragmentation of pH-responsive polymer, with microencapsulation technique, applied to the textiles may be the solution of such problems. Further experimental procedure and characterization of the pH-responsive materials must be necessary for the individual study.

From the literature observed that polymers or hydrogels utilized for the textile application are only thermo-responsive materials. Also, pH-responsive polymers are specifically used for the drug delivery or gene delivery, not for the surface application on textiles. Specifically for the feminine and baby hygiene products.

The Purpose of this research is to prepare a pH-responsive polymer can be microencapsulated for hydrogel and can be applied to the textiles, which are utilized in the preparation of hygiene textiles for female and baby. Also, to analyze the prepared product with conventional one running in market. It is also investigated the effect of such material on nearest skin area to the vaginal

The thesis entitled "Design and Fabrication of pH Responsive Microencapsulated Phase Change Materials for Textile Hygiene Applications" comprises five chapters.

1. Introduction

Chapter 1 History and background of the present investigation and distribution of the thesis.

2. Review of Literature

Chapter 2 reviews, the literature available in the related area, i.e. pH-responsive polymers, micro-encapsulations and its application on textile hygiene.

3. Materials and Methods

This chapter is dedicated for synthesizing of pH responsive polymer by suitable polymerisation method, and then it is characterised via different analytical procedures. The design of the assembly for polymerisation was set. Also, hydrogel can be produced with pH responsive material, which absorb more compare to conventional one.

The five naked round bottom flask is the main accessory for the polymerisation. Produced polymer is applied with padding action on the textile materials. Processed textile materials are quality checked for the absorption, pH stabilization, flexibility, stretching etc. The material must balance the pH.

The synthetic route consisting the addition of deionized water taken in 1000 ml five neck round bottom flask. MAA, DAM, BP, n-eicosane was added in the flask by maintaining the temperature 55°C with constant stirring. ADS was added in above mixture after 15 minutes, and then the mixture is allowed to stand at 55°C for 2.5 hours with the purge of nitrogen. As the stable O/W emulsion is produced the temperature gradually increased to 75°C, so free radical polymerization took place under the presence of nitrogen gas, and the mixture is allowed to stand at the same temperature for 3 hours with constant agitation. After 3 h, mixture of MAA, ACR, ACN and $K_2S_2O_8$ introduced in above system. After mixing the flask was heated at 82°C for 2.5 h with constant stirring to accomplish interfacial polymerization. At the end of reaction, yield was washed with deionized water and white powders collected by filtration, which are pH-responsive polymer microcapsules. They are dried at room temperature. Series of experiments was performed for achieving desirable quality of pH responsive polymer and its encapsulation.

JSM 7001F scanning electron microscope (SEM) was used for study the morphology of pH-stimuli microcapsules. Nano Measure™ software was utilized for measurement for particle size distribution and mean particle size of pH-responsive microcapsule. For FTIR (Fourier-transform infrared spectroscopy), PerkinElmer Spectrum™ 3 FT-IR spectrometer was used which can analyze the chemical structure and composition of pH-responsive microcapsules.

The amount of distilled water that is absorbed by the sample during its freely swollen condition is measured by this test result (free swell capacity). In terms of convenience and speed, the tea-bag method is the best option for samples with limited quantities. AUL (absorption under load) determines the capacity of SAP samples to absorb fluid at certain pressures. The filter screen was filled with an SAP sample of 0.9 grams to create an even bed. On the bed, a piston made of aluminium was placed. pH of the saline solution was measured using pH-meter. 0.5 g of sample was added gradually to 100 ml of a saline solution with stirring. The transportation speed of fluid travelling from the surface on which it absorbs to the inner zone of pad is measured with strike through test. Higher value of strike through indicates the top layer of pad is quickly absorbing the fluid, and so the skin feels dry. A 5 ml drop of 0.9% saline solution is used to conduct the test, and the penetration time is recorded.

The absorbent capacity of a superabsorbent polymer (SAP) was assessed by measuring its absorbency and absorption speed according to the EAS 96:2008-Annex C standard test procedure. Rewet test, the resistance of the SAP-coated sanitary napkin to transferring liquid that floods the coverstock to the skin is measured. Five milliliters of 0.9% saline solution were placed in the middle of a SAP-coated sanitary napkin for this test, and the solution was allowed to settle for one minute.

Polymer-coated samples were analyzed for antimicrobial activity against *E.coli* (gram negative) by spreading on a nutrient agar plate and incubated at 37 °C for 24 to 48 hours. The diameter of the inhibiting zone was measured for studying the activity against bacteria.

IS 7702 test was used for analyzing the thickness of various menstrual pads (coated with prepared polymer and commercially available) with the MAG-C1001 thickness tester possessing a range of 0.010–10.00 mm.

4. Result and Discussion

Fourth chapter responses with the results received by the application and its characterization of polymer on sanitary napkin. SEM micrographs suggests the particle size distribution of prepared microcapsule is between 1 to 20 μm , and the all-present microcapsules are showing diameter between 8 μm to 15 μm . FTIR was also performed and analyzed for the prepared product, which was utilized for coating on sanitary napkin.

Free swell capacity (FSC) and absorption under load (AUL) of all the samples treated with pure n-eicosane, poly(MAA-co-ACR-co-ACN) and pH-responsive microcapsules in tap water and saline solution are shown in table. The highest FSC value 94.23 g/g is attributed to sample prepared with S-3, While in saline water, the FSC value is comparatively lower than the value obtained with tap water. 55.87 g/g attributed to sample SAP3, while other samples are between 32.42 to 41.78 g/g.

The measurement of the wet back capacity of the sanitary napkins is done because it can prevent fluid from the napkin absorbed on the coverstock from getting to the skin. The performance analysis of samples coated with SAPs and controls is clearly suggesting the values of 0.22 to 0.24 g, which is clearly supported with the products manufactured during the study, and is comparable with conventionally available napkins which have the wet back capacity between 0.18 g to 0.22 g.

The fluid's transit speed from the absorbent surface to the inner pad zone is determined using a strike-through test. The skin feels dry when the strike-through value is higher because it means the pad's top layer is absorbing the fluid more quickly. The fluid moves from the surface to the interior in less time—2.62 seconds, according to figure, because the CA3 has a faster rate of transportation. The fluid transportation in the napkin, where SAP3 was applied to the top layer, required slightly fewer seconds (2.46). The times needed to pass the fluid for CA1, CA2, and CA4 are 2.51, 2.48, and 2.46, in that order. Consequently, the amount of time needed to strike through was the same for both SAP3 and CA4.

As the SAP was applied to a non-woven cotton web, the thickness of the SAP-coated napkins improved, as shown in figure 8. CA1 shows the lowest thickness among all the samples. CA2 and CA3 have hydrophilic fiber and cotton, so thickness is higher than CA1. SAP-coated napkins (SAP1 to SAP3) were thinner, except CA1, and the thickness of Sap1 to SAP3 was between 1.7 and 4.2 mm due to the presence of adsorbent sheets. SAP3 shows the lowest thickness value of 1.7 mm, which is near CA1.

5. Conclusions

The chapter contains conclusion and future scope for the study.

Paper published and presented

Published Papers

- Madhu, C., Patel, B., **Designing pH-responsive SAP-coated menstrual pads and comparing them to marketable products through quality analysis**, *Bulletin of National Research Center*, **48**, 41, 1-12 (2024). (Springer Open)
<https://doi.org/10.1186/s42269-024-01196-0>
- Madhu, C., Patel, B., **pH-Sensitive Polymers with their Important Applications (A Review)**, *Current Physical Chemistry*, **14**, 2, 93-115 (2024). (UGC CARE listed)
<https://doi.org/10.2174/0118779468296209240427102827>
- Madhu, C., & Patel, B., **Preparation of microencapsulated phase change materials for use in feminine hygiene napkins based on pH response**, *Essential Chem*, **1**, 1, 1–9(2024). (Taylor & Francis)
<https://doi.org/10.1080/28378083.2024.2357332>
- Madhu, C., & Patel, B., **Enhancing the efficacy of feminine hygiene sanitary napkins by developing a super absorbent polymer based on MMA and MAA**, *Journal of Applied Polymer Science*, (2024). (Wiley-Scopus indexed)
<https://doi.org/10.1002/app.56038>

Presented Papers

- Paper presented in **international conference** (“INDIA@100; building ‘Atmanirbhar’ Bharat from Farm to Fashion to Future, NIFT, Gandhinagar) **23,24 February 2023**
Synthesis and Evaluation of AA, ACN-Based SAP and its Application for Hygiene Textile
- Poster presented in **National conference** (Condensed Matter Physics and Material Science (CMPMS-2023)) **04 March 2023**
Application of Microencapsulation in Textile Wet Processing: A Review
- Paper Presented in **international conference** (Advances in Water Treatment and Management (ICAWTM-23)) **11,12 March 2023**
Reducing the Effluent Load by Coating Hygiene Textile with Microencapsulated AA, ACN Based SAP
Receive best presentation award in one of the panels
- Paper presented in **international conference** (Recent Advances in Applied Sciences and Engineering (RAISE 2023)) **12,13 April 2023**
Synthesis and Evaluation of MAA, ACR, AN-Based SAP for Application for Hygiene Textile

Papers (Under Correspondence)

- Madhu, C., Patel, B., **Formulation and Characterization of Superabsorbent Copolymer AA, ACN Grafted Sodium Acrylate for Feminine Napkin**, *The Journal of The Textile Institute*, Revision submitted on 10/01/2024. **(Taylor & Francis)**
- Madhu, C., Patel, B., **Developing pH-responsive MAA and MMA based (SAP)-coated menstrual pads and compare their performance with commercially available products**, *Research Journal of Textile and Apparel*, Submitted on 09/09/2024. **(Emerald, Scopus Indexed)**

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