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Microencapsulation of Vetiver Essential Oil using Complex Coacervation Technique

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ABSTRACT

Recently, fragrance embedded textiles are being developed in areas of sustainability, environment consciousness for healthy life style and well-being. Aromatherapy is a form of an alternative medicine in which essential oils are used to impart therapeutic effects. Vetiver essential oil was selected as the core material and gum acacia and gelatin as wall materials. For standardization of microencapsulation process, the ratios of core and wall materials, temperature and pH were optimized on the basis of size, distribution and quality of wall of formed microcapsule observed under inverted microscope. The vetiver essential oil:gum:gelatin in the ratio 1:4:4 at 45 ^{o}C temperature with initial pH 4.5 and final pH 7 were optimized for development of microcapsules as medium sized microcapsules having good uniformity in size and distribution with sharp and thick walls were obtained with these optimized process parameters. Hence, these optimized variables can be used to prepare the vetiver essential oil microcapsules for the development of fragrant textiles.

Key words: *Complex Coacervation, Control release, Gelatin, Gum acacia, Microcapsules, Vetiver oil*

Introduction

With the growing trend in enhancing beauty through healthy means, consumers demand for apparels and home textiles not only with their original basic characteristics such as warmth and comfort, but also ones that carry extra functions, including environmental protection, anti-pollution and most importantly, health and beauty care, for a more natural and healthier life. Therefore, the textile industry is currently experiencing a revolution that aims at the unique needs of the modern consumers. The integration of aromatherapy in textile application is a novel and user-friendly idea that enables an alternative means for essential substance delivery systems (Khanna *et al.,* 2015).

Essential oils are volatile, natural and complex compounds characterized by a strong odour and are extracted from various parts of aromatic plants such as flowers, leaves, stalks, fruits and roots. An oil is 'essential' in the sense that it carries a distinctive scent or essence of the plant from which it is derived. *Vetiveria zizanioides* belonging to the family Poaceae also known as *Khas* or *Khus* grass is native to India. The roots of *Vetiveria zizanioides* contain essential/volatile oil, known as vetiver or *khus* oil. It is one of the essential oils that have multi-functional values and it can be used as an eco-friendly finishing agent to impart functional properties to textiles. The essential oil of vetiver is one of the most important raw materials in perfumery both as a fixative and fragrance ingredient. The main action of vetiver oil is on the nervous system and it is both sedative and strengthening in effect. It has been traditionally used in aromatherapy from a long period of time for relieving stress, anxiety, depression, nervous tension and insomnia (Snigdha *et al.,* 2013 and Krishnaveni, 2016).

Infusion of textiles with aromatic essential oils make them of immense value enriched for the aesthetic dominated fashion consumers to attain therapeutic and medicinal benefits and also make the wearer afresh and relaxed by the unique aroma of oils. However, longevity of aroma on the textile with passage of time and subsequent launderings is a major concern for researchers and consumers too. Therefore, microencapsulation is found to be a solution to this problem as it is a rapidly expanding technology of applying relatively thin coatings to small particles of solids or droplets of liquids and dispersions. It provides the means of converting liquids to solids, providing environmental protection and controlling the release characteristics or availability of coated materials. Hence, the present study was conducted to standardize the microencapsulation process for the development of vetiver essential oil microcapsules using complex coacervation technique in order to protect and maintain its stability for effective application in various textile products (Kumar *et al.,* 2013 and Sailaja and Jyothika, 2015).

Materials and Methods

Selection of essential oil and wall materials

For preparation of microcapsules, vetiver essential oil was selected on the basis of aromatic and therapeutic properties, ease of availability and cost effectiveness. Gum acacia and gelatin were used as wall materials to encapsulate the core material. Vetiver essential oil was provided by Emmbros Overseas Lifestyle Pvt. Ltd., Haryana, India. Wall materials, i.e. gum acacia and gelatin were purchased from chemical suppliers of Haryana, India. Acetic acid, sodium hydroxide and formalin were also used in the study.

Selection of microencapsulation technique

There are many physical and chemical techniques which can be used to prepare microcapsules such as solvent evaporation, polymerization, spray drying, pan coating, phase separation- complex coacervation, centrifugal extrusion etc. Out of all these, phase separation- complex coacervation technique was selected for the present study on the basis of review and the suitability of the process to be carried out in the laboratory.

Standardization of microencapsulation process

Experiments were conducted for preparation of standardized microcapsule gel of vetiver essential oil with different ranges of variables of microencapsulation process i.e. ratios of oil, gum and gelatin, temperature and pH for optimization on review basis. The modified recipe standardized by (Kumari, 2015) was followed for preparing microcapsules. 16 g of gelatin was weighed and dissolved in 25 ml warm water and stirred using a high speed stirrer for 10 minutes. 4 g of vetiver oil was added to the solution at 45 ºC. 16 g of gum acacia was weighed and dissolved in 25 ml warm water separately. The gum acacia solution was added to the gelatin solution and the temperature of the solution was maintained at 45 ºC. The pH of the solution was decreased to 4.5 by adding dilute acetic acid and stirred at high speed for 20 minutes. The pH of the solution was increased to 8.5 using sodium hydroxide solution to form microcapsule gel. For stabilization, 1 ml of 17 percent alcoholic formalin was added to the formed capsules. After each step of microencapsulation process, the resultant precipitate obtained was observed under inverted microscope to ensure the formation of microcapsules and images were captured. The combinations of the ratios of oil, gum and gelatin which produced the desired results were selected for further optimization of other variables. At a time, the ratio of only one variable was varied and other variables were kept constant.

Optimization of essential oil ratio*:* For determining the optimum ratio of essential oil, six different ratios of oil i.e. 0.5, 1.0, 1.5, 2.0, 2.5 and 3.0 were taken while all other variables, i.e. gum, gelatin, temperature and pH were kept constant to carry out microencapsulation process. Microcapsules of medium size with good uniformity in size and distribution having sharp and thick walls were considered as best and appropriate. The ratio at which microcapsules formed of the best quality was selected as optimized ratio of essential oil for further process.

Optimization of gum ratio: To optimize the ratio of gum, six different ratios of gum i.e. 1, 2, 3, 4, 5 and 6 were taken with optimized ratio of essential oil whereas ratio of gelatin was kept constant along with all other variables. Process was carried out to prepare microcapsule gel and ratio of gum was optimized on the basis of microscopic assessment of microcapsules.

Optimization of gelatin ratio: For determination of optimum ratio of gelatin, six different ratios of gelatin, i.e. 1, 2, 3, 4, 5 and 6 were taken with optimized ratio of oil and gum and all other variables were kept constant. Process was carried out to prepare microcapsule gel and ratio of gelatin was optimized on the basis of assessment of microcapsules under inverted microscope.

Optimization of temperature: For determining the optimum temperature for microencapsulation, the process was carried out at different temperatures i.e. 30, 35, 40, 45, 50, 55 and 60 °C with optimized ratios of oil, gum and gelatin and values of initial and final pH were kept constant. Temperature was optimized on the basis of microscopic assessment of microcapsules.

Optimization of initial and final pH: The pH plays important role in microencapsulation as it is responsible for phase separation which leads to capsule formation. For optimization of pH, the optimized ratios of essential oil, gum and gelatin at optimized temperature was set to initial pH 4.0, 4.5, 5.0, 5.5, 6.0, 6.5 and 7.0. The microencapsulation process was carried out till gel formation took place and then final pH was maintained at 7.0, 7.5, 8.0, 8.5, 9.0, 9.5 and 10.0 with each initial pH. The initial and final pH were optimized on the basis of assessment of microcapsules under inverted microscope.

Results and Discussion

The data shown in Table 1 and visual assessment of microcapsule gel (Image 1) reveal that vetiver oil microcapsules were formed in five ratios of oil, gum and gelatin while in 3:4:4 ratio no microcapsules were formed. Three ratios, i.e. 0.5:4:4, 1:4:4 and 1.5:4:4 displayed good uniformity in size and distribution with thick wall of microcapsules. However, at 1:4:4 ratio of oil, gum and gelatin, best capsules were formed as they were medium sized with good uniformity in size having thick and sharp walls. Therefore, for further optimization, the essential oil in 1 ratio to wall materials was selected to achieve the desired results.

It is evident from data in the Table 2 and microscopic images of microcapsule that uniform microcapsules of vetiver essential oil were formed in four ratios of oil, gum and gelatin, i.e. 1:1:4, 1:2:4, 1:3:4 and 1:4:4. However, the best microcapsules were formed in the ratio of 1:4:4 hence, it was used for further development of microcapsules (Image 2). At higher ratios of gum acacia i.e. 1:5:4 and 1:6:4, larger microcapsules were formed which was not considered as an appropriate size for microcapsules.

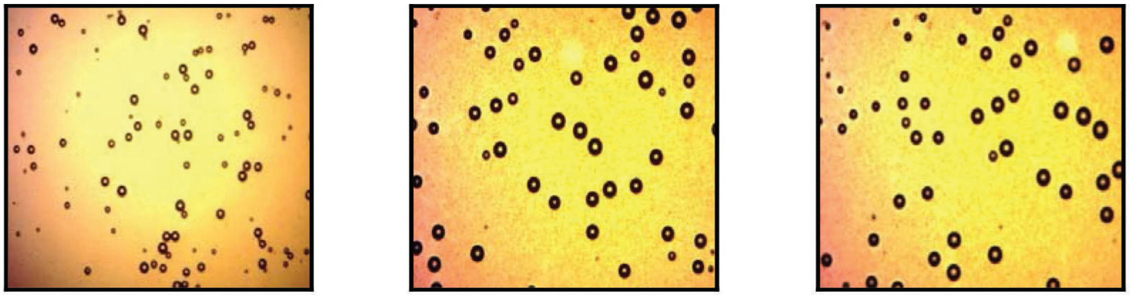
The perusal of Table 3 and Image 3 indicate that good vetiver essential oil microcapsules were formed in the four ratios of oil, gum and gelatin, i.e. 1:4:1, 1:4:2, 1:4:3 and 1:4:4. But the microcapsules formed with 1:4:4 ratio were medium sized, having good uniformity in size and distribution and the walls were also sharp and thick as compared to the capsules formed at other ratios. Thus, it was selected for further optimization.

Also, when the amount of oil, gum and gelatin was increased the size of microcapsule became larger but the walls of the capsules started rupturing and lumps were formed due to disproportionate ratio of oil, gum and gelatin. These results were in conformity with the findings of Bhatt and Singh (2018) and Senem *et al.* (2018) that with the increase in amount of oil and wall material the size of microcapsules and their oil content increased but they tended to agglomerate. After a certain concentration, no capsules were formed.

The data in the Table 4 and microscopic analysis of the microcapsule gel (Image 4) show that the microcapsules of vetiver essential oil formed at 45 °C

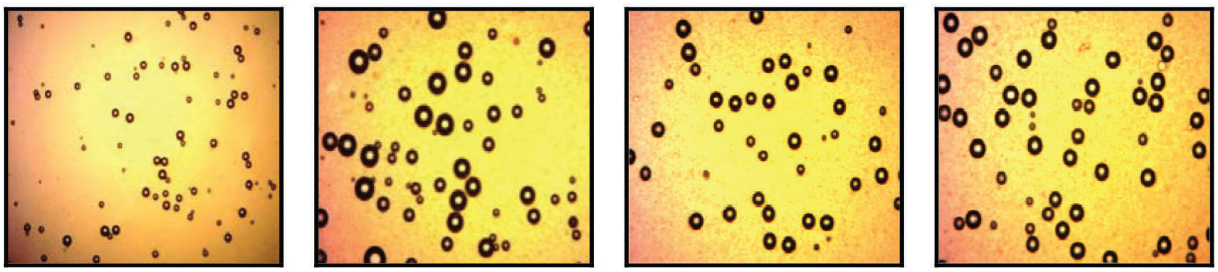
Table 1. Optimization of vetiver essential oil ratio in microcapsule gel

Ratio of oil: gum: gelatin	Size of microcapsules	Uniformity in size and distribution	Wall of microcapsules	Ranks
0.5:4:4	Very small + Small	Average	Very thick	III
1.0:4:4	**Medium**	**Good**	**Sharp + Thick**	**I**
1.5:4:4	Small + Medium	Good	Thick	II
2.0:4:4	Large	Poor	Very thick	IV
2.5:4:4	Very large	Poor	Very thick + Ruptured	V
3.0:4:4	Not formed	Not formed	Not formed	-



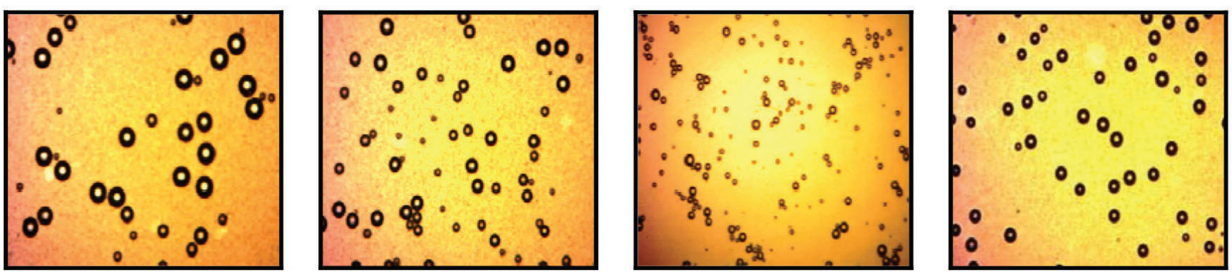
0.5:4:4 (Rank-III) 1:4:4 (Rank-I) 1.5:4:4 (Rank-II)

Image 1. Inverted microscopic images of microcapsules at different ratios of vetiver essential oil



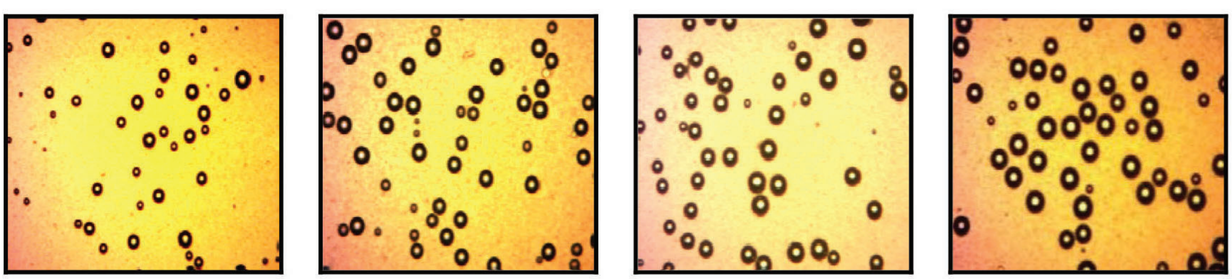
1:1:4 (Rank-IV) 1:2:4 (Rank-III) 1:3:4 (Rank-II) 1:4:4 (Rank-I)

Image 2. Inverted microscopic images of microcapsules at different ratios of gum acacia



1:4:1 (Rank-IV) 1:4:2 (Rank-II) 1:4:3 (Rank-III) 1:4:4 (Rank-I)

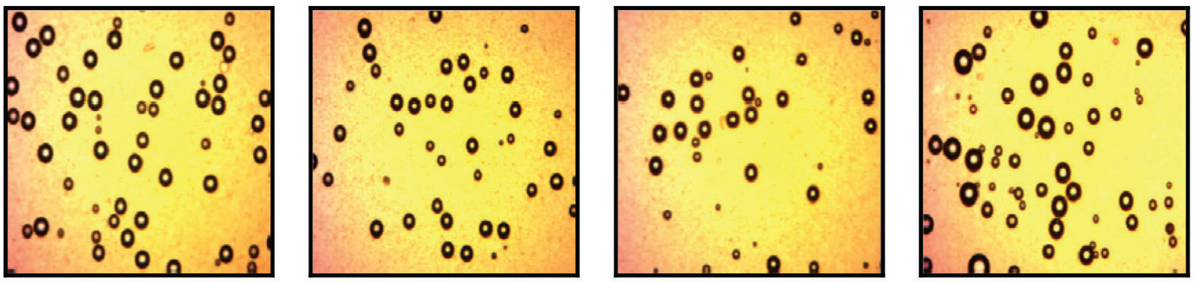
Image 3. Inverted microscopic images of microcapsules at different ratios of gelatin



40°C (Rank-II) 45°C (Rank-I) 50°C (Rank-III) 55°C (Rank-IV)

Image 4. Inverted microscopic images of microcapsules at different temperatures temperature were medium sized, had good uniformity in size and distribution and the walls of capsules were also sharp and thick as compared to capsules formed at other temperatures. Therefore, 40 °C temperature was chosen as the optimum temperature for preparation of microcapsules.

Essential oils are volatile in nature and they evaporate very easily, therefore with the increase in temperature, size of the microcapsules also noticed to be increased but they ruptured immediately. Higher temperatures than 60 °C were also tried but no microcapsules were formed on those temperatures. Similar observations were reported by Zhao *et al.* (2016) and Sannapapamma *et al.* (2018) that at



4.0/8.0 (Rank-II) 4.5/7.0 (Rank-I) 5.0/7.0 (Rank-IV) 5.0/7.5 (Rank-III)

Image 5. Inverted microscopic images of microcapsules at different initial and final pH

Table 2. Optimization of gum acacia ratio in microcapsule gel

Ratio of oil: gum: gelatin	Size of microcapsules	Uniformity in size and distribution	Wall of microcapsules	Ranks
1:1:4	Very small +Small	Average	Thick	IV
1:2:4	Small + Large	Average	Very thick	III
1:3:4	Medium + Large	Good	Thick	II
1:4:4	**Medium**	**Good**	**Sharp + Thick**	**I**
1:5:4	Large +Very large	Poor	Very thick	V
1:6:4	Very large	Very poor	Thick +Ruptured	VI

Table 3. Optimization of gelatin ratio in microcapsule gel

Ratio of oil: gum: gelatin	Size of microcapsules	Uniformity in size and distribution	Wall of microcapsules	Ranks
1:4:1	Large	Average	Very thick	IV
1:4:2	Small +Medium	Good	Thick	II
1:4:3	Very small +Small	Average	Thick	III
1:4:4	Medium	Good	Sharp + Thick	I
1:4:5	Large	Average	Thick+Ruptured	V
1:4:6	Very large	Poor	Very thick+Ruptured	VI

Table 4. Optimization of temperature for microencapsulation process

Temperature (°C)	Size of microcapsules	Uniformity in size and distribution	Wall of microcapsules	Ranks
30	Very small	Poor	Thick	VI
35	Small	Average	Very thick	V
40	Small +Medium	Good	Thick	II
45	Medium	Good	Sharp + Thick	I
50	Large	Average	Very thick	III
55	Medium +Large	Average	Very thick	IV
60	Large	Poor	Thick+Ruptured	VII

Table 5. Optimization of pH for microencapsulation process

pHinitial/ final	Size of microcapsules	Uniformity in size and distribution	Wall of microcapsules	Ranks
4.0/7.0	Very small	Poor	Very thick	IX
4.0/7.5	Small	Average	Thick	VI
4.0/8.0	Medium + Large	Good	Sharp +Thick	II
4.0/8.5	Very large	Poor	Very thick+Ruptured	XI
4.5/7.0	Medium	Good	Sharp + Thick	I
4.5/7.5	Medium	Average	Thick	V
4.5/8.0	Very small +Small	Average	Thick	VII
4.5/8.5	Large	Poor	Thick+Ruptured	X
5.0/7.0	Medium	Average	Thick	IV
5.0/7.5	Small +Large	Good	Sharp +Thick	III
5.0/8.0	Medium +Large	Average	Thick+Ruptured	VIII
5.0/8.5	Very large	Poor	Ruptured	XII
5.5/7.0	Medium+ Large	Average	Very thick	XIII
5.5/7.5	Large	Average	Thick+Ruptured	XIV
5.5/8.0	Very large	Poor	Ruptured	XV
5.5/8.5	Large + Very large	Very poor	Very thick + Ruptured	XVI

higher temperature walls of microcapsules got ruptured.

The microcapsules formed at different ranges of initial and final pH were observed to determine the optimum pH values. It was found that microcapsules were formed only when the initial pH ranged from 4.0 to 5.5 and final pH ranged from 7.0 to 8.5. It can be inferred that for phase separation and microcapsule gel formation of the vetiver essential oil, the other pH ranges were not suitable. It is obvious from Table 5 and visual assessment of microcapsule gel that microcapsules of the vetiver essential oil formed at initial pH 4.5 and final pH 7.0 were medium sized with uniform distribution having thick and sharp walls (Image 5). Hence, these pH values were considered suitable for the development of vetiver oil microcapsules. The results were in corroboration with Kumari *et al.* (2017) and Pakzad and Alemzadeh (2018).

Conclusion

The 1:4:4 ratio of essential oil, gum and gelatin at temperature 45 ^{o}C with initial pH 4.5 and final pH 7 were optimized as the medium sized microcapsules having good uniformity in size and distribution with sharp and thick walls were obtained at these optimized ratios and conditions. The best microcapsules were formed when the ratio of vetiver essential oil was lower as compared to wall materials. The microencapsulated vetiver essential oil treated fabric can be used for various applications like apparel, home, medical and healthcare textiles as vetiver essential oil have many reported aromatic and therapeutic properties. Complex coacervation technique of microencapsulation is very effective for preparation of vetiver essential oil microcapsules to drive long term sustained benefits as it provide long-lasting finish by controlling the release rate of vetiver essential oil.

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