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Research Paper

Development of palmarosa oil microcapsules

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■ **ABSTRACT :** Natural fragrant microcapsule with gelatin and gum as a wall material palmarosa as a core material were prepared by complex cocervation technique. Different variables, including ratio of essential oil, gum, gelatin, temperature and pH were optimized on the basis of comparative analysis through visual assessment under inverted microscope. Result showed that medium sized microcapsules having sharp and thick wall and good uniformity in size and distribution were formed at ratio of 1:2:4 of oil, gum and gelatin, at a temperature of 50°C with initial and final pH 4.5 and 9.0, respectively. Hence, these optimized ratios were selected for preparation of microcapsules.

■ **KEY WORDS:** Cocervation technique, Gelatin, Microcapsule, Palmarosa oil

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In today's era of modernization of the textile industry, we are going through advancements of technology in every field of this industry. The world where this would lead us would be astonishingly hitech and materialistic. To ensure our security and safety from the future hazards, we need to equally develop the technology for our protection (Kumaravel *et al.*, 2009). Value addition in clothing has changed the global textile scenario. A novel and holistic approach of the 21st century has been the use of microencapsulation in textile finishing. Creative designers of the 21st century want to diversify their vision from visual aesthetics to performance value like sense of smell, colour change technology, phase change materials and bactericides. Today textiles can be treated so that they protect one from all kinds of adverse conditions yet at the same time are comfortable (Karolia and Mendapara, 2005). Microencapsulation is actually a micropackaging technique involving the production of microcapsules which act as barrier walls of solids or liquids. The microcapsules are produced by depositing a thin polymer coating on small solid particles or liquid droplets, or on dispersions of solid in liquids. The core contents are released under the controlled conditions to suit a specific purpose. Particles in micro-size prepared by the microencapsulation technique are called microcapsules or microparticles. The release mechanism of the core contents vary depending on the selection of wall materials and more importantly, its specific end uses. The core content may be released by friction, pressure, change of temperature, diffusion through the polymer wall, dissolution of the polymer wall coating or by biodegradation. Hence, present study was undertaken to optimize the process of different variable for preparation of palmarosa oil microcapsules.

■ RESEARCH METHODS

Palmarosa essential oil was procured from Delhi and it was selected on the basis of aroma and therapeutic effect. Complex cocervation technique was used for preparation of microcapsule. Various concentrations of raw material and conditions *i.e.* essential oil, gum, gelatin, temperature and pH were optimized. Comparative analysis on the various aspects such as size of microcapsule, uniformity in distribution and wall of the microcapsules was done on the basis of visual analysis by inverted microscope in the laboratory. Microencapsulation was done at specific ratios at specific conditions.

Standardization of palmarosa oil microencapsulation process :

For preparation of microcapsules of palmarosa oil different variables of microencapsulation process *i.e.* ratio of oil, gum and gelatin, temperature and pH were optimized one by one. The basic recipe was modified by incorporating different amount of oil, gum and gelatin subjected to different ranges of temperature and pH and the resultant precipitate obtained after each process was analyzed under inverted microscope to ensure the formation of microcapsules and images were captured. For optimization comparative analysis of the images were done for various aspects. The combinations of concentrations of oil, gum and gelatin which produced the desired results were further subjected to optimization of the other variables. At a time the concentration of only one variable was varied and other variables were kept constant.

Microcapsule gel was prepared using the standardized recipe. Optimized ratio of gelatin was dissolved in warm water and was stirred at high speed for 10 min. Optimized ratio of essential oil (core material) was added to the solution at optimized temperature and pH of the solution was set at optimized initial pH. After that optimized ratio of gum acacia was dissolved in water and mixed with above solution. The whole solution was stirred at high speed for 20 min and temperature was lowered to 5ºC for gel formation. The pH of the gel was set at optimized final pH. 1 ml of alcoholic formalin (17 %) was added to the formed capsules.

■ RESEARCH FINDINGS AND DISCUSSION

The findings of the present study as well as relevant discussion have been presented under following heads :

Standardization of preparation of microcapsules :

Optimization of ratio of palmarosa oil in microcapsule gel :

Palmarosa oil forms the core material of the microcapsule and is basically responsible for the both *i.e.* fragrance and therapeutic effect. The data presented in Table 1 and visual assessment of microcapsule gel indicate that though microcapsules were formed in four ratio of oil, gum and gelatin *i.e.* 1:4:4, 1.5:4: 4, 2:4:4 and 2.5:4:4, respectively (Fig. 1). The microcapsules formed in the ratio of 1:4:4 were medium sized, had good uniformity in size and the wall was also sharp and thick as compared to the capsules formed at other three ratios. Therefore, the ratio 1 of palmarosa oil was used for further optimization. No gel formation occurred if more amount of essential oil were used because gum and gelatin were not able to form wall around it. Danfeng *et al.* (2012) prepared olive oil microcapsules by complex cocervation and reported that microcapsules formed at core to wall ratio 1:3 as ratio more than this formed large size of capsules and wall started rupturing.

Optimization of ratio of gum acacia in microcapsule gel :

Gum acacia was used as a wall material. Different

Table 1 : Optimization of ratio of palmarosa oil in microcapsule gel

Ratio of oil: gum: gelatin	Formation of microcapsules	Parameters			Rank
		Size of microcapsules	Uniformity in size and distribution	Wall of microcapsules	
0.5:4:4	No	-	-	-	-
1:4:4	Yes	Medium	Average	Sharp and thick	I
1.5:4:4	Yes	Small	Good	Very thin	IV
2:4:4	Yes	Medium + small	Poor	Thick	II
2.5:4:4	Yes	Very large	Good	Thick	III
3:4:4	No	-	-	-	

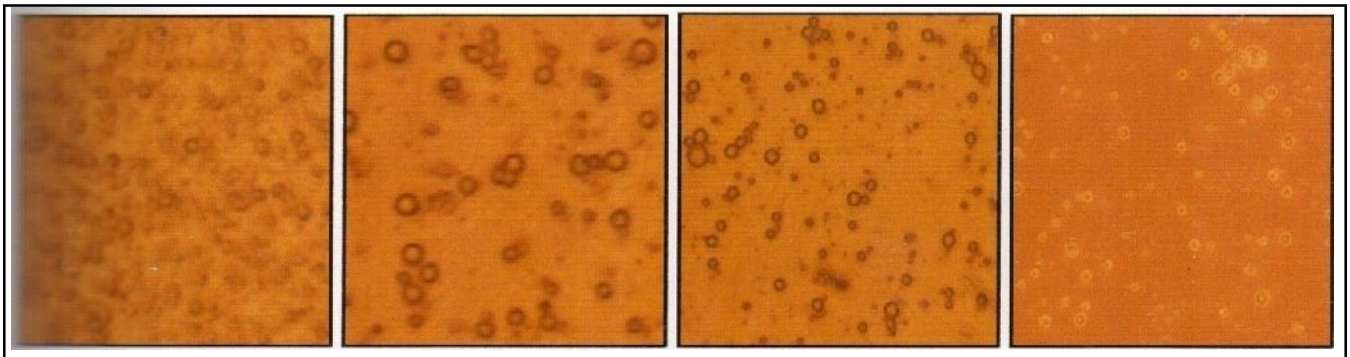


Fig. 1 : Microcapsules at different ratio of oil

Table 2 : Optimization of ratio of gum acacia in microcapsule gel

Ratio of oil: gum: gelatin	Formation of microcapsules	Parameters			Rank
		Size of microcapsules	Uniformity in size and distribution	Wall of microcapsules	
1:1:4	No	-	-	-	-
1:2:4	Yes	Medium	Good	Thick	I
1:3:4	Yes	Small	Poor	Thin	III
1:4:4	Yes	Small	Average	Thin	IV
1:5:4	Yes	Medium	Very poor	Sharp and thick	II
1:6:4	No	-	-	-	-

Oil ratio: 1

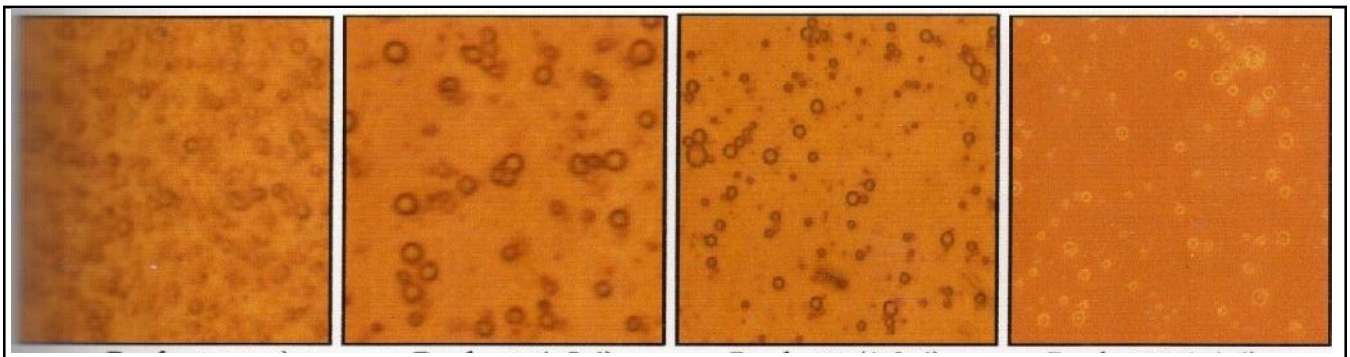


Fig. 2 : Microcapsules at different ratio of gum acacia

ratio of gum acacia was taken from 1 to 6. From the Table 2 and visual assessment of microcapsule gel (Fig. 2) indicates that microcapsules were formed in four ratios of oil, gum and gelatin *i.e.* 1:2:4, 1:3:4, 1:4:4 and 1:5:4. The microcapsules formed in the ratio of 1:2:4 were medium with good uniformity in size and distribution and the wall was also thick as compared to the capsules in the other three ratios. As the ratio of gum was increases the size of capsules was increased and lumps were formed due to disproportionate ratio of oil and gum. Therefore, the ratio of 2 of gum acacia was used for further optimization. Zivdar and Najafi (2004) recommended core to wall ratio as 1:1 and 1:2 while gelatin to gum arabic content to be 2:1.

Optimization of ratio of gelatin in microcapsule gel:

Different ratio of gelatin was used *i.e.* 1, 2, 3, 4, 5, and 6 for preparation of microcapsule gel. The data presented in Table 3 and visual assessment of microcapsule gel indicates that microcapsules were formed in the ratio of oil, gum and gelatin *i.e.* 1:2:2, 1:2:3, 1:2:4 and 1:2:5 (Fig. 3). The microcapsules formed in

the ratio of 1:2:4 were medium in size, had good uniformity in size and distribution and the wall was thick as compared to the capsules formed in the ratio of 1:2:2 and 1:2:3 which were small and medium in size, average and very poor uniformity, thick walls, respectively and large in size with good uniformity, thick and sharp wall of capsules in case of 1:2:5. Therefore, the ratio 4 was optimized for gelatin.

Optimization of temperature for microencapsulation :

Different temperatures were taken *i.e.* 30, 40, 50, 60, 70 and 80°C for preparation of microcapsule. The data presented in Table 4 and visual evaluation (Fig. 4) reveals that the microcapsules formed at 50°C were medium sized, had good uniformity in size and distribution and the wall was sharp and thick as compared to microcapsules formed at 40, 60 and 70°C. Hence, 50°C temperature was optimized for microencapsulation. Agarwal and Goel (2010) reported that higher temperature led to increase in evaporation of essential oils resulting in rupturing of walls. It was also revealed that smaller microcapsules have longer life as compared to larger microcapsules.

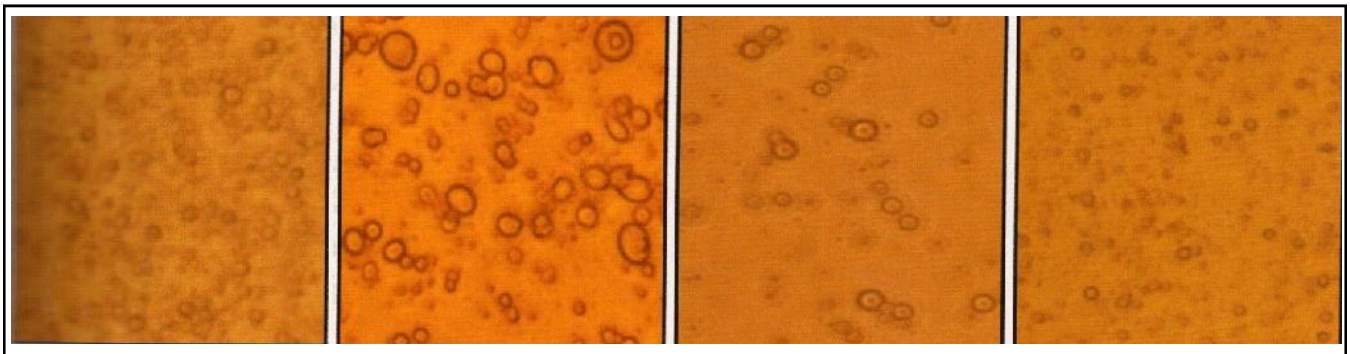
Optimization of pH for microencapsulation :

Microencapsulation was carried out with optimized ratio of oil: gum: gelatin, temperature and pH. pH play

Table 3 : Optimization of ratio of gelatin in microcapsule gel

Ratio of oil: gum: gelatin	Formation of microcapsules	Parameters			Rank
		Size of microcapsules	Uniformity in size and distribution	Wall of microcapsules	
1:2:1	No	-	-	-	-
1:2:2	Yes	Small	Average	Thick	IV
1:2:3	Yes	Medium	Very poor	Thick	III
1:2:4	Yes	Medium	Good	Thick	I
1:2:5	Yes	Large	Good	Thick and sharp	II
1:2:6	No	-	-	-	-

Oil: gum- 1:2



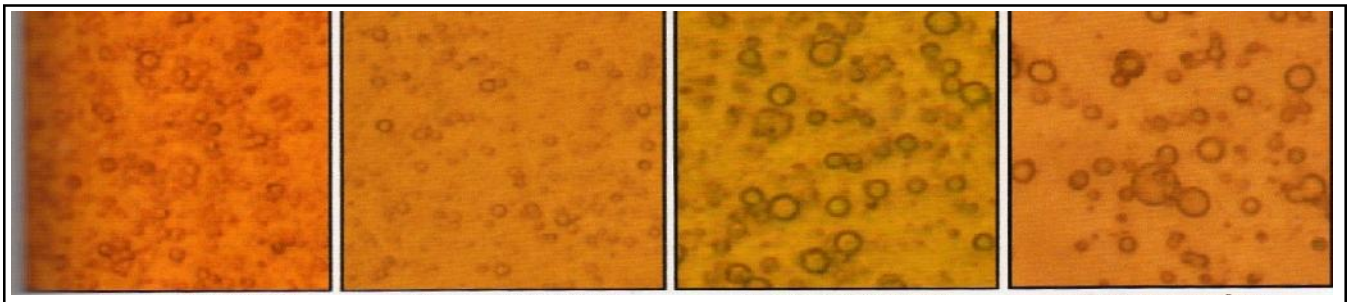
Rank-I (1:2:4) Rank-II (1:2:5) Rank-III (1:2:3) Rank-IV (1:2:2)

Fig. 3 : Microcapsules at different ratio of gelatin

Table 4 : Optimization of temperature for microencapsulation

Temperature (°C)	Formation of microcapsules	Parameters			Rank
		Size of microcapsules	Uniformity in size and distribution	Wall of microcapsules	
30	No	-	-	-	-
40	Yes	Small	Poor	Sharp and thick	II
50	Yes	Medium	Good	Sharp and thick	I
60	Yes	Large	Good	Thick	III
70	Yes	Medium +large	Poor	Thick	IV
80	No	-	-	-	-

Oil: gum: gelatin- 1:2:4



Rank-I (50^{0}C) Rank-II (40^{0}C) Rank-III (60^{0}C) Rank-IV (70^{0}C)

Fig. 4 : Microcapsules at different temperature

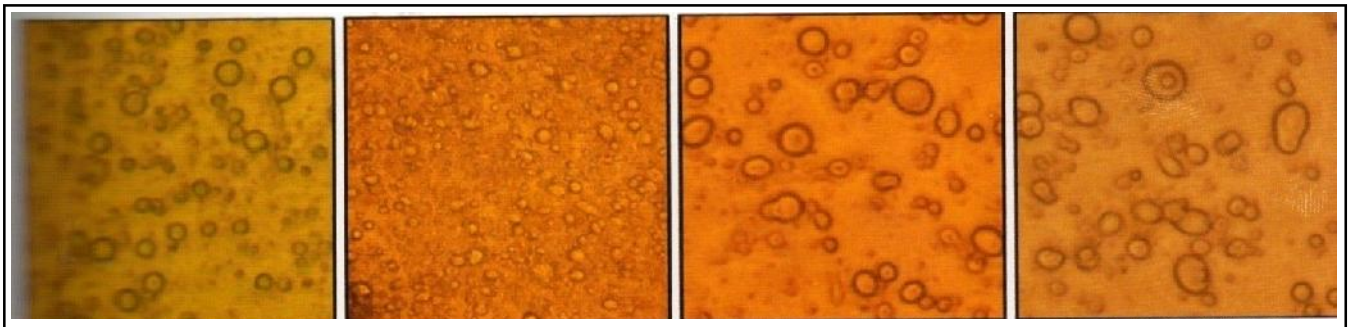
Table 5 : Formation of microcapsules at different pH

Initial pH	4.0	4.5	5.0	5.5	6.0	6.5	7.0
Final pH	Formation of microcapsules						
7.0	x	x	x	x	x	x	x
7.5	x	x	x	x	x	x	x
8.0	x	x	x	x	x	x	x
8.5	x	x	x	x	x	x	x
9.0	Formed	Formed	Formed	x	x	x	x
9.5	Formed	Formed	Formed	x	x	x	x
10.0	Formed	Formed	Formed	x	x	x	x

Oil: gum: gelatin-1:2:4, temperature- 50ºC

Table 6 : Optimization of pH for microencapsulation

pH initial/final	Formation of microcapsules	Parameters			Rank
		Size of microcapsules	Uniformity in size and distribution	Wall of microcapsules	
4.0/9.0	Yes	Small	Average	Thin	VI
4.0/9.5	Yes	Small +Medium	Average	Sharp and thick	V
4.0/10.0	Yes	Small	Good	Thick	II
4.5/9.0	Yes	Medium	Good	Sharp and thick	I
4.5/9.5	Yes	Medium	Average	Sharp and thick	III
4.5/10.0	Yes	Medium + large	Average	Sharp and thick	IV
5.0/9.0	Yes	Large	Average	Ruptured	VII
5.0/9.5	Yes	Very large	Poor	Ruptured	VIII
5.0/10.0	Yes	Very large	Poor	Ruptured	IX



Rank-I (4.5/9.0) Rank-II (4.0/10.0) Rank-III (4.5/9.5) Rank-IV (4.5/10.0)

Fig. 5 : Microcapsules at different pH

important in microencapsulation as it is responsible for phase separation that leads to capsule formation. To optimize initial pH and the final pH, microcapsule gel was initially set at pH 4.0, 4.5, 5.0, 5.5, 6.0, 6.5 and 7.0. After the completion of microencapsulation process and gel formation the final pH of the gel was set at 7.0, 7.5, 8.0, 8.5, 9.0, 9.5 and 10.

It is clear from the Table 5 that microcapsules were formed only when the initial pH ranged from 4 to 5 and final pH ranged from 9 to 10. The pH range other than this was not suitable for phase separation and microcapsule formation with oil.

As apparent from Table 6 and visual analysis of microcapsule gel indicates that the microcapsules formed at initial pH 4.5 and final pH 9.0 were medium in size with good uniformity in size and distribution and thick and sharp walls (Fig. 6). Therefore, these pH were selected for preparation of microcapsules.

Hence, it can be concluded that best results were obtained when the ratio of oil: gum: gelatin was 1:2:4, temperature 50ºC, initial pH 4.5 and final pH 9.0. Microcapsules formed with these optimized conditions were medium sized, had good uniformity in size and distribution with sharp and thick wall of capsules. The findings of Sukumar and Lakshmikantha (2010) support the results as they reported that the microcapsules were formed at initial pH ranged from 4-4.5 and final pH to be 9.

Conclusion :

Natural fragrant microcapsules have been prepared using palmarosa essential oils as a core material and gum and gelatin as wall material by complex cocervation technique. The microcapsules prepared with these optimized proportion of essential oil, gum and gelatin in the ratio 1:2:4 at temperature 50°C with initial pH 4.5 and final pH 9.0 were medium sized, had good uniformity in size and distribution and sharp and thick walls of capsules. Medium sized microcapsules with sharp and thick walls and good uniformity in size and distribution was obtained at temperature 50°C. Microcapsules were formed only when the initial pH was set at acidic pH between 4-5 as phase separation takes place at this pH only. Hence, it can be concluded that these optimized variables can be used to prepare the palmarosa oil microcapsules for application on fabrics for the development of fragrant textiles.

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