# INFLUENCE OF THE SURFACTANT CONCENTRATION ON THE ROSE OIL FILLED UREA-FORMALDEHYDE MICROCAPSULES

Stanislav G. Bayryamov

*Department of Repair, Reliability, Mechanisms, Machines, Logistic and Chemical Technologies*
*Agrarian and Industrial Faculty, "Angel Kanchev" Univesity of Ruse*
*8 Studentska St., Ruse 7017, Bulgaria*
*sbayryamov@uni-ruse.bg (S.B.)*

*Received 02 December 2024*
*Accepted 12 May 2025* *DOI: 10.59957/jctm.v60.i5.2025.8*

## ABSTRACT

*The microencapsulation process proceeds much more efficiently, with a higher yield and quality of the resulting microcapsules in the presence of an emulsifier. Its influence is due, on the one hand, to the reduction in the diameter of the microcapsules and on the other hand - to an increase in the thickness, density and quality of the capsule wall. However, the type and amount of emulsifier must be precisely selected to obtain satisfactory results, while avoiding the adverse effect of capsule agglomeration and increasing the cost of the process. In the presented work, SDS was used in different concentrations: 0 %, 0.5 %, 1 %, 2 %, 3 %, 4 %, 5 % for encapsulation of rose oil by in situ polymerization between urea and formaldehyde. Experimental results showed that the efficiency of the microencapsulation process increased with raising surfactant concentration. Nevertheless, the presence of a large amount of surfactant (over 3 %) was undesirable, due to an increase in the viscosity of the reaction medium, followed by an increase in the size of the microcapsules because of their sticking together. In addition, the presence of a large amount of surfactant is economically unprofitable, which is the reason for realizing these studies with the aim of selecting the optimal amount of emulsifier.*

*Keywords: poly(urea - formaldehyde), microdroplet, sodium dodecyl sulfate (SDS), surfactant, microencapsulation, in situ polymerization, rose oil.*

## INTRODUCTION

Rose oil occupies an important place in the Bulgarian industry, which is known for producing some of the highest quality rose oils in the world, which are used in the pharmaceutical, cosmetic, perfumery and laundry industries, as well as many other areas. This makes us look for ways to preserve the longevity of rose oil. One of these ways is the application of the microencapsulation process.

Due to the importance and worldwide relevance of the field of science and industry (problem) related to the microencapsulation of substances, there is a huge variety of methods, techniques and related protocols for obtaining microcapsules [1 - 4]. These methods depend on both the type of core substance to be encapsulated and its intended purpose, i.e. from the goal and field of its application [1 - 4].

The chemical method of microencapsulation by *in situ* polymerization is one of the widespread methods used for encapsulation of various types of substances [5]. The process depends largely on the conditions of the emulsification step, which produces an emulsion of the encapsulated substance in a solution of the particles making up the capsule shell [6]. The emulsification step results in two types of liquid - in - liquid dispersed systems, i.e. emulsions, depending on the encapsulated substance forming the capsule core

(dispersed phase) and the particles forming the capsule wall (their solution acting as the dispersion medium). When the core substance is hydrophobic (or dissolved in a hydrophobic solvent) and the shell substance is hydrophilic, most often in aqueous solution, the emulsion is oil - in - water. Conversely, in the case where the core substance is hydrophilic (dissolved in water) and the coating substance forming the capsule shell is hydrophobic (or dissolved in a hydrophobic solvent), the resulting emulsion is water - in - oil.

It has been found that the size of the capsules depends on the micro-droplet size produced during the emulsification step [5, 6]. At this step, three factors in the conditions affect the droplet size: stirring speed, temperature and process time [5 - 7]. It turns out that a fourth factor also affects the encapsulation efficiency. This is the presence of a substance that helps form an emulsion, i.e. an emulsifying agent, called an emulsifier or surfactant [8]. In general, the emulsification step is much more difficult in the absence of an emulsifier. Both the type of surfactant and its concentration affect the size of the capsules. In addition, the type of surfactant and its concentration also affect other capsule characteristics, such as yield, thickness, density and therefore microcapsule wall quality. For this reason, our research was aimed at studying the influence of surfactant concentration in the process of obtaining rose oil microcapsules. The rose oil was encapsulated through the process of in situ polymerization of mono methylol urea on the surface of the pre-formed microdroplets. Here we focused on the influence of SDS concentration in increasing the efficiency of the rose oil microencapsulation process.

Preliminary results showed that when the reaction was carried out in the absence of an emulsifier, despite high homogenization rates (stirring speeds), the size of the microcapsules was large (about 400 - 500 μm). In the presence of surfactant, however, this size decreased sharply [8]. Furthermore, SDS affected both the size, yield, and quality of the resulting microcapsules. With an increase in the surfactant concentration from 0.5 % to 3 %, the average size of the microcapsules decreases from 260 - 200 μm to 30 - 20 μm, after which a subsequent increase in this concentration adversely affects the quality of the obtained microcapsules, including the fact that their size increases, probably due to agglomeration of the microcapsules [8]. For example, increasing the SDS concentration from 4 % to 5 % leads to an increase in their average size from 40 - 60 μm to 90 - 130 μm. In turn, the encapsulation efficiency increases from 44.3 % - at a SDS concentration of 0.5 %, to 84.3 % at a SDS concentration of 5 %; the yield of microcapsules also increases from 11.7 % (0.5 % SDS) to 68.1 % (5 % SDS). The content of encapsulated substance (E % core) decreased from 59.8 % (0.5 % SDS) to 36.3 % (5 % SDS), indicating that the mass of encapsulated substance from the insoluble shell increased, and consequently that the density of the shell material (shell quality) increased. The studies are consistent with those done by other authors. The preliminary results obtained in this way directed our efforts towards carrying out more extensive studies on the effect of the weight percentage concentration of surfactant (SDS) on the process. The aim of the present work is to clarify this influence by carrying out a study on the microencapsulation process of rose oil using different concentrations (w/w) of SDS - 0 %, 0.5 %, 1 %, 2 %, 3 %, 4 %, 5 %. The author hopes that the experimental data will contribute to optimizing the technological conditions for obtaining rose oil microcapsules, as an important product for the Bulgarian economy.

## EXPERIMENTAL

### Methods and materials

The technical urea was recrystallized from ethyl alcohol. Formalin as a 37 % formaldehyde solution, and the rose oil was purchased from licensed Bulgarian producers. The pre - polymer was obtained in an alkaline media as a solution of mono methylol urea at a specified concentration. Freshly prepared 10 % sodium hydroxide solution, 1N solution of sodium hydroxide, and 10 % citric acid solution were used to adjust the pH throughout the process.

For the control of the reaction mixture (pH) a professional benchtop pH - meter: BANTE Instruments, Model 920 - UK with a combinative pH electrode with BNC coupling was used. The pH - meter included temperature compensation in the temperature range of 0$^{o}$C to 100$^{o}$C. Operating conditions: from 0oC to 50$^{o}$C with relative humidity up to 95 %. Division of pH = 0.001 pH units, range: from pH = - 2.000 to pH = 20.000, accuracy (at 20$^{o}$C) pH ± 0.002.

For agitation of the reaction mixture and for control of the stirring speed from 0 - 1000 rpm, an electromagnetic stirrer with heating was used with an included temperature probe to control the actual temperature, brand DIAB, Model MS7 - H550 - S with a temperature range of + 30 - 550°C, stirring speed 0 - 1000 rpm, Power 1030 W. For agitation above 1000 rpm a homogenizer for solid and liquid media Velp Scientifica, Model OV5 with a stirring speed of 1000 - 22000 rpm was used.

Weight analyses including microcapsule yield (%), encapsulation efficiency (EE, %), % sample (% encapsulated compound, core content, E% core), resin efficiency (RE, %) and encapsulation factor (EF) were performed by weighting various components on an analytical and precise balance with internal calibration "KERN" model ABJ 120 - 4NM, range 120 g, accuracy 0.0001 g, plate diameter: d = 91 mm; as well as using an analytical and precise balance with internal calibration - "KERN" model ABJ 220 - 4NM, range 220 g, accuracy 0.0001 g, plate diameter: d = 91 mm.

The shape, morphology and approximate size of the microcapsules were analyzed with a light microscope CARL ZEISS JENA, model 30 - G0020a, with magnifications of 12.5 x, 25 x, 40 x and 100 x, as well as a reflective optical metallographic microscope Nikon, included in the equipment of CSEM Scratch tester and digitized with a 14 - megapixel camera. The size of the microcapsules as well as their size distribution were determined using a laser diffraction apparatus brand MICROTRACK MRB model SYNC, with a working range of 0.01 μm - 4mm.

FT-IR analyses of rose oil microcapsules were carried out on PerkinElmer Spectrum™ 3 FT-IR apparatus (21 CFR Part 11 Compatible) operating at the wavelenght range between 7800 $cm^{-1}$ - 225 $cm^{-1}$. The spectra of the prepared microcapsules were obtained after their freeze drying using KBr pellets or NaCl crystals.

**Preparation of microcapsules**

***Pre - polymer synthesis step. General procedure***

To a 500 mL three - necked round bottom flask fitted with a thermometer, reflux condenser and electromagnetic stirrer, 60 g of urea (Mm = 60.06 g $mol^{-1}$; 1 mol) were added. After that, 120 mL of 37 % formalin solution (44.4 g formaldehyde, Mm = 30.03 g $mol^{-1}$; 1.48 mol) were added with vigorous stirring, as the pH of the mixture was controlled via adjusting to pH 8 - 8.3 by slowly adding drop wise of 10 % sodium hydroxide solution. The reaction mixture was heated in a water bath for 1 h, all the while ensuring that the temperature of the medium did not exceed 70°C. The heating at this temperature was continued, then the water bath was removed, and the flask was refluxed at room temperature. After cooling, the reaction mixture was diluted with distilled water to 250 mL of pre - polymer solution. As the pH of the reaction mixture decreased during the reaction, therefore it was necessary to maintain the alkalinity in the range of 8 - 8.3 by drop wise addition of dilute sodium hydroxide solution. This decrease in the pH of the reaction mixture is not desirable due to the creation of conditions for the formation of insoluble undesirable side by - products. This lowering of the pH of the reaction mixture was not desirable due to the creation of conditions for the formation of insoluble unwanted by - products. For this purpose, various alkaline salts, such as ammonium carbonate, sodium acetate and its mixture with citric acid were used. Moreover, various bases and their salts such as urotropin, melamine, TRIS. HCl or TRIS - base, triethanolamine, ammonium chloride and others can be used instead of sodium hydroxide solution.

***Emulsification step, varying the surfactant concentration. General procedure***

The emulsification step was also the same as in our previous article [9], excluding that the variety percentage concentration of surfactant was used i.e.: 0 %, 0.5 %, 1 %, 2 %, 3 %, 4 %, 5 % of SDS at constant stirring of 1500 rpm. After transferring of the pre - polymer and surfactant solution to a 500 mL round - bottom flask, equipped with a thermometer, reflux condenser and mechanical or electromagnetic stirrer, the flask was placed on a hot plate and the reaction mixture was heated up at a temperature of 70oC. In a beaker, after preparing a 5 mL solution of rose oil in medical paraffin (rose oil : medical paraffin - 1 : 49, 0.1 mL : 4.9 mL) it is added to the solution of pre-polymer and surfactant (SDS). Then, the same stirring speed and temperature of 70°C were maintained for about 3 h until a milky white emulsion was obtained. After that, the temperature of the reaction mixture was lowered to 45°C. Finally, the reaction was allowed to proceed for 2 h.

***Microencapsulation (polymerization) step***

The microencapsulation step was implemented according to the procedure described in the article, studying the effect of time in the emulsification step on the microencapsulation of rose oil through the *in situ* polymerization process [9].

**Product analysis**

***Weight analysis***

The yield (%), the core content (E % core) and the encapsulation efficiency (EE %) were calculated using the equations presented in [9]. The resin efficiency (%) and the encapsulation factor were calculated using the Eq. (1) and Eq. (2) from [9].

***Particle size analysis***

The mean particle diameter, size distribution and standard deviation were determined by the laser diffraction apparatus MICROTRACK MRB model SYNC, range of detection: 0.01μm - 4mm. Standard deviation was calculated using the equation (3) from the article [9].

***FT-IR spectroscopic analysis***

The spectra of the prepared microcapsules were obtained after freeze-drying the whole microcapsules (without breaking them by grinding in a porcelain mortar or by sonication) and the capsule wall was analysed by FT-IR spectrometry. The spectra of the obtained microcapsules were at 3470 $cm^{-1}$ and 3360 $cm^{-1}$, 2850 $cm^{-1}$ and 2680 $cm^{-1}$, 1640 $cm^{-1}$ and 1430 $cm^{-1}$, 1170 $cm^{-1}$ and 1060 $cm^{-1}$ which characterizes C-H, N-H, C-N and C=O vibrations, respectively as well as the N-H of the amine are at 3350 $cm^{-1}$ and 3200 $cm^{-1}$, respectively (Fig. 1).

**RESULTS AND DISCUSSION**

Since the *in situ* polymerization process of the UF pre - polymer occurs on the surface of the droplets, the average diameter of the droplets determines the diameter of the finally prepared microcapsules. The influence of the surfactant, together with other conditions such as stirring speed and temperature, is due on the one hand to the reduction of the diameter of the droplets obtained during first sub - step (stage A) of the emulsification step, which allows the synthesis of smaller microcapsules in the polymerization step. On the other hand, since the *in situ* polymerization process of the UF pre - polymer takes place on the surface of the droplets, the emulsifier influences the adsorption of the pre - polymer during the second sub - step (stage B) of the emulsification step, reducing the surface tension

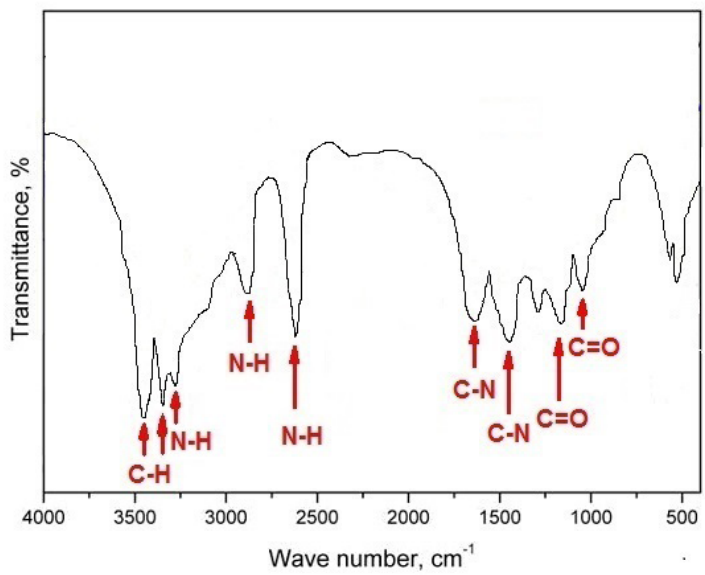


Fig. 1. FT - IR spectrum of poly(urea - formaldehyde) shell of the microcapsules filled with rose oil.

of the surface of the resulting microdroplets and thus having a positive effect on the thickness, density and quality of the capsule wall. Surfactant plays a key role in the microencapsulation process, and in its absence, this process proceeds “slowly”, with low intensity, which leads to low yields, the formation of large-sized capsules, with poor quality and low density of the microcapsule shell. In the microencapsulation procedure, SDS is used as a surfactant in various concentrations.

As can be seen from the data in Table 1, Fig. 2 in the absence of surfactant, the yields of microcapsules do not exceed 9 % (8.68 %), the encapsulation efficiency is also low and is 35.6 %, the content of the encapsulated substance (E% core), respectively, is the highest: 68.2 %. Other characteristics such as resin efficiency (RE) (7.42%) and encapsulation factor (EF) (0.21) also have low values. On the other hand, the sizes of the microcapsules are very large and vary between average values of 350 - 240 μm (Table 1).

With increasing surfactant concentration from 0 % to 0.5 %, the yield of microcapsules increases to 12.7 % and the encapsulation efficiency increases to 46.3 %. The content of the encapsulated substance (E% core) accordingly decreases to 60.7 %, which provides information that the mass of the encapsulating substance from the insoluble shell increases, and hence the quality and density of the microcapsule wall increase. This in turn has a direct impact on the efficiency characteristic of the resin, which increases from 7.42 % at an SDS concentration of 0 %, to 10.8 % at an SDS concentration of 0.5 %. Regarding the encapsulation factor, it also increases from 0.21 at 0 % SDS concentration, to 0.32 at 0.5 % SDS concentration. In turn, the size of the microcapsules decreases to values ranging between 240 - 200 μm.

With increasing surfactant concentration from 0.5 % to 3 %, a decrease in the average size of rose oil microcapsules was observed from 240 - 200 μm to 25 - 15 μm, after which, with further increase in SDS concentration, their size increased, probably due to agglomeration of microcapsules (Fig. 3). For example,

Table 1. Effect of surfactant weight percentage concentration (SDS) on the characteristics of the obtained rose oil microcapsules.

| № | SDS, % w/w | Yield, % | EE, % | $E\%_{core}$ | RE, % | EF | Size, μm |
|---|---|---|---|---|---|---|---|
| 1 | 0 | 8.68 | 35.6 | 68.2 | 7.42 | 0.21 | 350 - 240 |
| 2 | 0.5 | 12.7 | 46.3 | 60.7 | 10.8 | 0.32 | 240 - 200 |
| 3 | 1 | 24.5 | 62.4 | 47.4 | 22.1 | 0.59 | 140 - 60 |
| 4 | 2 | 53.6 | 80.7 | 36.9 | 45.1 | 1.38 | 40 - 20 |
| 5 | 3 | 67.8 | 83.9 | 36.5 | 59.1 | 1.83 | 25 - 15 |
| 6 | 4 | 71.6 | 82.5 | 39.2 | 48.9 | 1.89 | 60 - 40 |
| 7 | 5 | 67.3 | 80.3 | 43.3 | 35.5 | 1.86 | 120 - 80 |

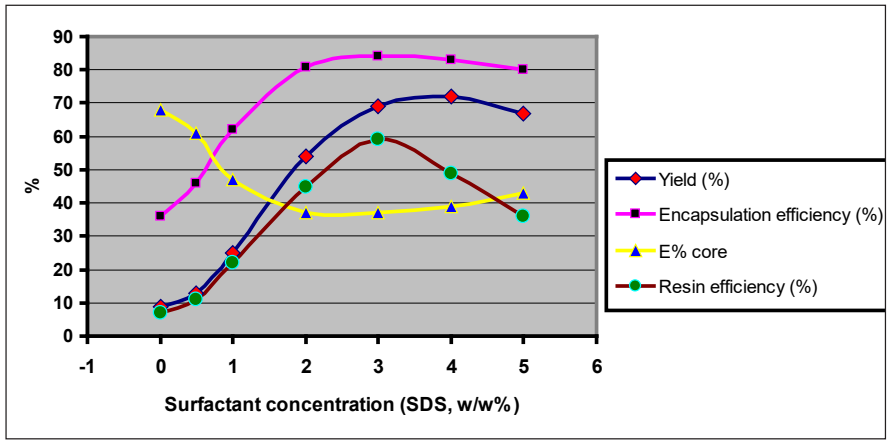


Fig. 2. Effect of the surfactant weight percentage concentration on the characteristics of the obtained rose oil microcapsules.

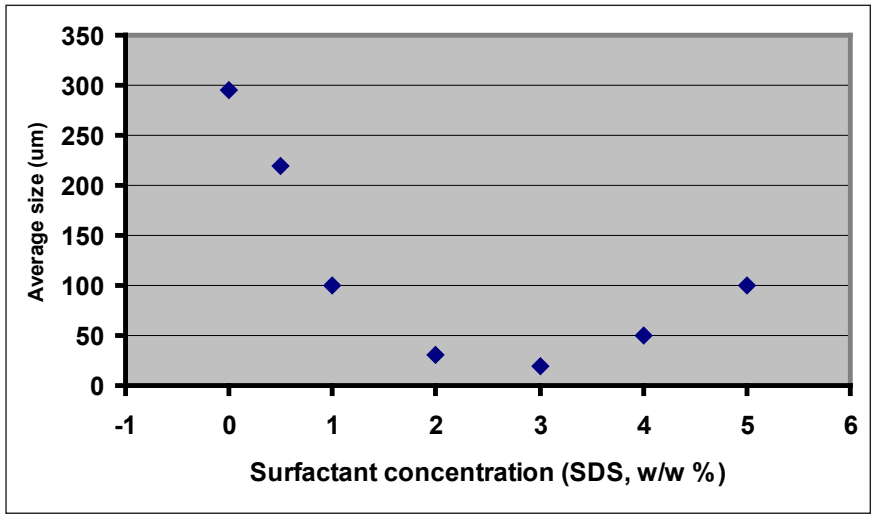


Fig. 3. Effect of the surfactant weight percentage concentration on the size of the obtained rose oil microcapsules, represented by the mean value of the diameter, in μm.

increasing the SDS concentration from 4 % to 5 % leads to an increase in the average size of the resulting microcapsules from 60 - 40 μm to 120 - 80 μm. On the other hand, when the SDS concentration increased from 0.5 % to 3 %, the encapsulation efficiency increased from 46.3 % to 83.9 %, while at higher concentration, the encapsulation efficiency slightly decreased. For example, at SDS concentration of 4 % it is 82.5 % and at SDS concentration of 5 % it is 80.3 %. The yield of capsules also increased sharply from 12.7 % (0.5 % SDS) to 67.8 % (3 % SDS). Then, as the percentage concentration of SDS increases, it slightly increases (71.6 % at 4 % SDS), after which a slight decrease in the yield is observed (67.3 % at 5 % SDS), i.e. it can be summarized that it remains relatively constant. In terms of the content of the encapsulated substance (E% core), with an increase in the SDS concentration from 0.5 % to 3 %, E% core decreases from 60.7 % to 36.5 %, indicating that the density of the material making up the shell (and therefore the quality of the shell) increases. The concentration of 3 % SDS seems optimal also with respect to this characteristic, since, as can be seen from the data in Table 1 (6th and 7th rows), further increase in SDS concentration leads to an increase in E% core from 39.2 % at 4 % SDS to 43.3 % at 5 % SDS. This in turn indicates that the capsule shell quality and density are decreasing. The dependence of the change in the values of E% core, in turn, directly reflects another characteristic such as resin efficiency (RE), whose values move inversely proportional to the content of the encapsulated substance, i.e. when the SDS concentration increases from 0.5 % to 3 %, the RE values increase from 10.8 % (0.5 % SDS) to 59.1 % (3 % SDS). The increase in RE values confirms the claims of increased capsule wall quality when the SDS concentration is increased from 0.5 % to 3 %. Furthermore, increasing the concentration of SDS leads to a decrease in the values of this characteristic (Table 1, 6th and 7th rows). Similar dependencies are also observed for the EF characteristic. As the surfactant concentration increases from 0.5 % to 3 %, the EF values increase from 0.32 to 1.83. This relationship indicates that most of the pre - polymer was used to build the microcapsule wall, leading to an increase in the thickness and density, and hence the quality of the capsule wall. With subsequent increases in emulsifier concentration, the values remain relatively constant, at a weight percentage concentration of SDS of 4 %, the EF value slightly increases to 1.89, and at 5 % SDS it slightly decreases to 1.86.

This indicates that increasing the emulsifier concentration above 3 % (w/w) has no beneficial effect on the efficiency of the encapsulation process and the characteristics of the resulting microcapsules. When the surfactant concentration increases above 3 %, some of the characteristics change slightly, others do not

change, and still others deteriorate. For example, the yield increases slightly, the encapsulation efficiency, encapsulation factor, and resin efficiency decrease, and the E% core and microcapsule size increase.

The influence of the weight percent concentration of the surfactant on the microencapsulation process, yield and quality of the obtained microcapsules follows a similar dependence (i.e. trend) observed for the other parameters in the reaction conditions, such as stirring speed, temperature and time. The difference between the influence of other conditions and the influence of the emulsifier is that, for example, increasing the stirring speed after a certain optimal speed, as well as the time specifically during the emulsification step, has a negligible effect on the characteristics of the resulting microcapsules, i.e. increasing the values of these parameters in the conditions after a certain value does not have a significant impact on the process. Unlike the stirring speed and time during the emulsification step, the increase in temperature beyond a certain optimum value during the emulsification step, as well as the concentration of the emulsifier, also beyond a certain optimum value, negatively affects the process and the characteristics of the resulting microcapsules. For example, increasing the temperature above its optimum value increases the desorption of pre-polymer particles (mono methylol urea) from the surface of the microdroplets formed during the first stage (stage A) of the emulsification step, and this leads to thinning of the capsule shell and deterioration of its thickness, density and quality. The same negative impact is also caused by the very high concentration of surfactant, since high values in the weight percent concentration of SDS cause agglomeration of the microdroplets, followed by an increase in the size of the microcapsules and a similar deterioration of the quality of the capsule wall. This deterioration in the density, thickness and quality of the capsule wall is caused by the reduced amount of prepolymer adsorbed on the surface of the microdroplets, observed at very high SDS concentration.

For this reason, based on the experimental results obtained, the optimal surfactant concentration was determined to be 3 %, since a lower concentration leads to low yields, low capsule wall quality, and a high concentration causes droplet agglomeration and larger capsule size, reducing yields and capsule wall quality.

Other conditions: emulsification step - stirring speed: 1500 rpm, 3.5 h at temperature: 70°C, 2.5 h at temperature 45°C; polymerization step - stirring speed: 750 rpm, stirring time: 3 h, temperature: 45°C.

## CONCLUSIONS

Most methods for obtaining microcapsules require the presence of an emulsifier - a surfactant. Microencapsulation of substances can also occur without its participation but is more difficult due to the sticking together (agglomeration) of the microdroplets, which leads to a larger size of the resulting microcapsules. This work investigates the effect of surfactant concentration (SDS) on the microencapsulation process of rose oil in a solvent using the chemical method of *in situ* polymerization, with the formation of a urea-formaldehyde shell forming the capsule wall. The author found that increasing the concentration leads to an increase in encapsulation efficiency, an improvement in the quality of the resulting microcapsules, as well as a reduction in their size. After a certain concentration, however, further increase in surfactant concentration leads to a decrease in process efficiency due to an increase in the viscosity of the reaction mixture. Thus, the present work helps to select the optimal concentration of the emulsifier, on the one hand, to carry out an efficient process for obtaining microcapsules with desired small sizes and high-quality dense shell forming the capsule wall, and on the other hand - to prevent agglomeration of microdroplets and unprofitability of the process due to increased costs. In this way, the author hopes to contribute to optimizing the process of preserving rose oil for a long period of time through its encapsulation - an important area of global practice and industry.

***Acknowledgements***

*This research was funded by Research Science Fund of University of Ruse (Project 2024-AIF-02)*

***Authors' contributions**: The entire layout of the theoretical part of the article, as well as all experimental results: the synthesis of the pre-polymer, the preparation of the microcapsules and their analysis were performed by the author.*

## REFERENCES


1. N. Choudhury, M. Meghwal, K. Das, Microencapsulation: An overview on concepts, methods, properties and applications in foods, Food frontiers, 2, 4, 2021, 426-442, https://doi.org/10.1002/fft2.94.
2. B. Srinivasaraonaik, L. P. Singh, I. Tyagi, A. Rawat, S. Sinha, Microencapsulation of a eutectic PCM using in situ polymerization technique for thermal energy storage, 2020, Int. J. Energ. Res., Wiley online library, https://doi.org/10.1002/er.5182.
3. P. Arce, C. Castellón, A. Castell, L.F. Cabeza Use of microencapsulated PCM in buildings and the effect of adding awnings. Energy Build, 44, 2012, 88-93. doi:10.1016/j.enbuild.2011.10.028.
4. B. Zalba, J.M. Marín, L.F. Cabeza, H. Mehling, Review on thermal energy storage with phase change: materials, heat transfer analysis and applications, Appl. Therm. Eng., 23, 2003, 251-283. doi:10.1016/S1359-4311(02)00192-8.
5. Microencapsulation via In Situ Polymerization, Handbook of Encapsulation and Controlled Release, Edited by Munmaya Mishra, 1st Edition, CRC Press, 2015, eBook ISBN: 9780429183478.
6. O. Nguon, F. Lagugné-Labarthet, F. Brandys, J. Li, E.R. Gillies, Microencapsulation by in situ polymerization of amino resins, 2018, Chemistry Publications, 110. https://ir.lib.uwo.ca/chempub/110.
7. S.G. Bayryamov, M.P. Nikolova, Preparation of urea-formaldehyde microcapsules filled with rose oil by *in situ* polymerization method. Influence of the stirring rate, stirring (homogenization) time, and reaction temperature of the stirring (homogenization) process, Proceedings of the University of Ruse "Angel Kanchev", Chemical technologies, 58, 10.1, 2019, 77-86, SAT-CR-P-2-CT(R)-12, (in Bulgarian).
8. S.G. Bayryamov, M.P. Nikolova, Preparation of urea-formaldehyde microcapsules filled with rose oil by *in situ* polymerization method. Influence of the surfactant concentration, Proceedings of the University of Ruse "Angel Kanchev", Chemical technologies, 58, 10.1, 2019, 44-50, SAT-CR-P-2-CT(R)-07, (in Bulgarian).
9. S.G. Bayryamov, Effect of time in the emulsification step on the yield and quality of urea-formaldehyde rose oil microcapsules obtained by interfacial *in situ* polymerization method, J. Chem. Technol. Metall., 60, 3, 2025, 383-392. ISSN 1314-7471.
10. S.G. Bayryamov, A comparative study on the synthesis of rose oil containing microcapsules by complex coacervation, using different solvents, J. Chem. Technol. Metall., 58, 2, 2023, 318-326. ISSN 1314-7471.