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RESEARCH ARTICLE OPEN ACCESS

Adaptability of polyurea microcapsules loaded with octyl salicylate for sunscreen application: influence of shell thickness of microfluidic-calibrated capsules on UV absorption efficiency

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Abstract – The common chemical ultraviolet (UV) filters such as octyl salicylate that are used in sunscreen cream may cause irritation and other unknown medical conditions. One possible route to avoid the direct contact of UV filters with human skin is to enclose those filters in microcapsules. In this study, microcapsules with a size calibrated at 78 μm via microfluidics but with different shell thicknesses (0.4 to 2.5 μm) are used for the first time, to study the influence of shell thickness of microcapsules on the UV absorption efficiency. The results show that the shell thickness of microcapsules has no obvious influence on the UV absorption efficiency. Besides, a rough model based on the Beer–Lambert law is used to compare the experimental results.

Keywords: UV filter, Microcapsules, Microfluidics, Shell thickness, UV absorbance

Introduction

The exposure to ultraviolet (UV) radiation in sunlight could lead to undesirable health issues related to human skins. This may cause damage to the skin molecules, structures and genes [1–3], mainly caused by UV exposure. This in turn can lead to sunburn, pigmentary, skin ageing and in the worst case nonmelanoma skin cancer [4]. Physical coverage by clothing and glasses can be useful against UV. However, it is not always applicable in summer and especially when people go to the beach. Therefore, sunscreens containing mineral or chemical filters (or a mixture of both) are often used to protect the skin from the sun's UV rays.

The mineral sunscreens are generally formulated with zinc oxide and titanium oxide. These oxide microparticles sit on the surface of the skin and work as a shield so most UV photons in sunlight are reflected and scattered. Though they are regarded to be safe and effective UV filters [5] by the Food and Drug Administration (FDA), there are several downsides: low water resistance, hard spreading and noticeable white trace on the skin after application. These substances can be reduced to nanoparticles (less than 100 nm) using nanotechnology, which become easier to apply and invisible on the skin [6]. However, they may be more bioreactive and more easily enter the skin and other organs after micronization, bringing questions concerning their safety [7].

The chemical sunscreens contain different organic molecules which can absorb high-energy UV rays. The received energy is converted into heat energy or safe low-energy radiation and then released and consumed, which avoids damage to the skin. Compared to mineral microparticles, the smaller organic molecules are more likely to penetrate the skin [8]. Some studies review the health concern of using chemical UV filters on kidney and liver and of their neurotoxicity [9–11]. Most of the common chemical UV filters are not recognized as safe and effective drugs by the FDA because of their insufficient safety data, so further works on this topic are demanding for both industry and academia [5]. Nevertheless, using sunscreens is the most efficient way to avoid skin diseases caused by UV in sunlight. Therefore, the FDA recommends that consumers continue to use either chemical or mineral sunscreens, although their risks remain unknown [12].

Considering the necessity of using chemical sunscreens as well as their unknown toxicity, an expedient solution is to avoid the direct contact between the chemical filters and human skin by a physical barrier which can also prevent the penetration of those organic molecules through

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the skin. Polyurea microcapsules with hard and dense shells can be used to protect core materials and prevent the core mass transfer to the outside. For example, polyurea microcapsules loaded with different phase-change materials are fabricated for thermal insulation in architecture or in fireproof clothing [13–15].

However, to our best knowledge, there is no study about using polyurea microcapsules for enclosing UV filters and avoiding their direct contact with human skin. The scientific problems are: can common chemical UV filters be successfully encapsulated by polyurea (chemical compatibility of UV filters with polyurea microcapsules)? Does the shell thickness of microcapsules affect the UV absorption efficiency of UV filters? For the first problem, our previous study [16] explores the optimal conditions for successful microencapsulation of octyl salicylate by polyurea shells. To respond to the latter one, a microfluidic approach is used to calibrate the size of microcapsules [17–22], and thus we just need to use different concentrations of reactants for varying the shell thicknesses of microcapsules.

Herein, we first reproduce the microcapsules with a calibrated size at 78 μm but with different shell thicknesses from 0.4 to 2.5 μm, the synthetic conditions and characterization information for those microcapsules can be found in our previous study [16]. Then UV absorbance for the measuring plates and for the suspension medium of microcapsules are tested for the aim of calibration. In the end, the absorbances of microcapsules of different shell thicknesses are measured.

Materials and methods

Materials

The hexamethylene diisocyanate biuret (HDB-LV, Vencorex Chemicals, free isocyanate group in a molecule: 23.5 ± 1.0 wt%) is pucharsed from Vencorex. Sodium dodecyl sulfate (Across Organics, grade pure); Ethylenediamine (Sigma Aldrich, ≥99%); octyl salicylate (or 2-Ethylhexyl salicylate, Sigma Aldrich, ≥99%); and 2-Propanol (Sigma Aldrich, ≥99.9%) are used without additional purification. Distilled water is produced by mono-distillate 2008, GFL. All liquids are filtered by syringe filter (JVLAB, PTFE with 0.45 μm pore size), before being supplied into the microchannel. Glycerol (Ph.Eur., wasserfrei, ≥98%) is used to disperse the microcapsules on the PMMA plates. The Polymethyl methacrylate (PMMA) plates (5 cm × 5 cm) with a roughness of 4.5–5.5 μm are friendly provided by Helioscience.

Production of monodispersed polyurea microcapsules

For the formation of polyurea microcapsules, octyl salicylate (OS) containing isocyanate (reagent 1) is emulsified by an aqueous phase with a surfactant (SDS) to form an oil-in-water (O/W) emulsion. Then, ethylenediamine (reagent 2) is added to this emulsion. This last step triggers an interfacial polymerization between these two reagents, which forms a solid shell around the drops. In this study, microfluidics is used to calibrate the capsule size at 78 μm. To vary the shell thicknesses of microcapsules, different isocyanate concentrations (c_{HDB}) are used in droplet phases. More detailed information on the experimental protocol can be found in our previous studies [16, 22].

UV absorbance of suspension medium

In order to avoid the agglomeration of capsules during UV absorbance measurements, the capsules are dispersed in a 1 wt% SDS (surfactant) solution. Thus it is important to control any possible UV absorption of this suspension medium. The UV absorbance from 290 nm to 390 nm for the 1 wt% SDS solution is measured by a standard UV Spectrophotometer (model: Cary 60).

UV absorbance of microcapsules deposed on a PMMA plate

Experimental apparatus

The measurements for the UV absorbance of the capsules need a special UV spectrophotometer. In fact, the light beam can be scattered by the capsules and may not reach the detector of a standard UV Spectrophotometer, which yields absorbance measurements much higher than the "real" value. In this case, the Bio-Tek Kontron UVIKON 933 Double Beam UV Spectrophotometer (Fig. 1a) which is equipped with an integrating sphere inside measuring chamber to collect the scattered light, is used to characterize the efficiency of UV adsorption of the capsules.

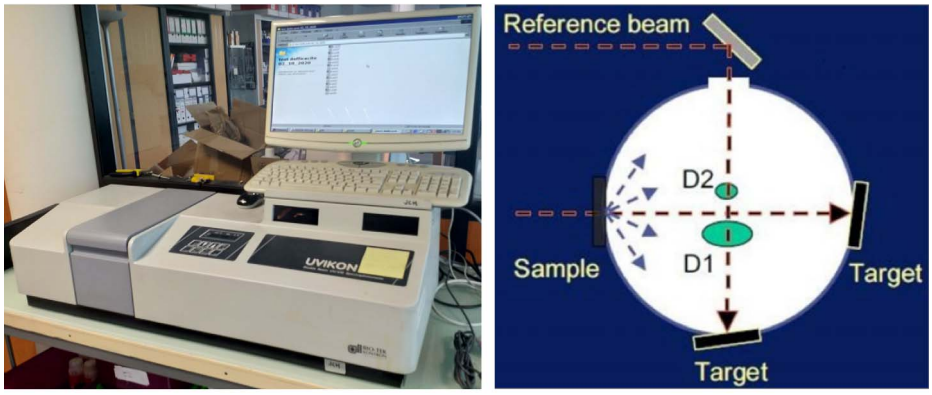
The design of a typical double beam integrating sphere is illustrated in Figure 1b. It is a hollow ball made by highly reflective material. For a double beam sphere, there is a light entrance port (dashed line in Fig. 1b) for both the reference and sample respectively. The green components (D1 and D2 in Fig. 1b) are photosensitive detectors for measuring the reflected and scattered UV light inside the sphere. The two black targets which are placed with a certain angle in Figure 1b, are used to reflect the axial lights (brown dotted line) to the spherical surface rather than reflect them back to the entrance ports.

Protocol of measurement

During the measurements, the samples (PMMA plates) are placed vertically inside the measuring chamber. The capsules should well stick to the plates and be spread out evenly. If they fall from the plates or have some flowability which leads to an uneven dispersion of capsules on the plates, the measuring errors can be very large. To avoid these problems, the capsule suspensions are first concentrated as much as possible and then glycerol is added to this suspension to further reduce its flowability on the plates.

1. Concentrating the microcapsule suspensions

All the fabricated polyurea microcapsules have density values larger than that of the continuous phase. Therefore



(a) (b)

Figure 1. (a). The UV spectrophotometer that is used in this study. Model: Bio-Tek Kontron UVIKON 933. (b). Illustration of a typical double beam integrating sphere.

the capsules can settle naturally to the bottom of the beaker. To concentrate the capsule suspensions, they just need to be transferred into a 1 mL container with a small cross-section. Once the capsules settle to the bottom again, the supernatant is removed by a pipette. In general, the concentration of capsules in suspension can arrive at 50 wt% (150 mg of capsules suspended in 150 mg continuous phase).

2. Spreading out the microcapsules on PMMA plates

For a better dispersion of the microcapsules on PMMA plates, 100 mg of glycerol is added with capsules suspensions onto PMMA plates. Then this mixture is spread out evenly by a finger from the center to the border of plates. The mass of this mixture before and after spreading-out is controlled by an analytical balance. The mass measurements show that approximately 10 wt% of the total mixture remains on the finger in the process of spreading.

Results

UV absorbance calibration for suspension medium and PMMA plate

UV absorbance of suspension medium

The UV absorbance from 290 nm to 360 nm for the 1 wt% SDS solution is shown in Figure 2a. This suspension medium has almost no UV absorption (less than 0.01) for the range of interest of wavelength, which is consistent well with the results in the literature [23]. Thus the UV absorbance of capsules can be measured directly with the presence of suspension medium because the the influence of surfactant is completely negligible on the UV absorbance capsules.

UV absorbance of PMMA plate

For the measurements of UV absorbance of microcapsules, they are first mixed with 100 mg of glycerol for reducing their flowability and then deposed on a PMMA plate. Thus it is also important to know if glycerol and PMMA plates can absorb UV at around the wavelength of 300 nm. Their UV absorbance are measured and shown in the Figure 2b. It can be seen that the PMMA plate have an UV absorption value around 0.16 from 290 nm to 360 nm whereas glycerol does not contribute to the UV adsorption apparently. Therefore, for the results in the next about UV absorption of microcapsules or pure OS, the UV absorption of a PMMA plate with 100 mg glycerol is removed.

UV absorbance of pure OS and microcapsules

UV absorbance of pure OS

To have an idea about the UV absorption efficiency of the microcapsules, the UV absorbance of pure OS on a PMMA plate is measured firstly. Three grams of pure OS liquid are added to one hundred milliliters of 2-propanol. Then one milliliter of this solution is added on a PMMA plate. Since 2-propanol wets the PMMA plate, the solution can spontaneously spread on the PMMA plate. After that, this PMMA plate is left to dry for one day. Finally, the mass of OS on the PMMA plate is measured by an analytical balance to make sure that there is 30 mg of OS deposed. The reason for applying 30 mg of OS on a PMMA plate (5 cm × 5 cm), is to follow the commonly used method (1.2 mg sunscreen per square centimetre) for sunscreen tests in vitro [24]. The absorbance for 30 mg of pure OS on a PMMA plate is shown in Figure 3. The range of wavelength for the measurement is from 290 nm to 360 nm. OS is a good absorber for the range of UV from 290 nm to 330 nm with an absorbance of more than 1.0. The maximum absorbance of 1.25 is found at the wavelength of 320 nm.

We must point out that the absorbance of pure OS is measured under an ideal situation where the OS forms a homogeneous film on the PMMA plate. While in a real sunscreen cream, the UV filters are generally in form of an emulsion. Therefore, for the same quantity of OS, the

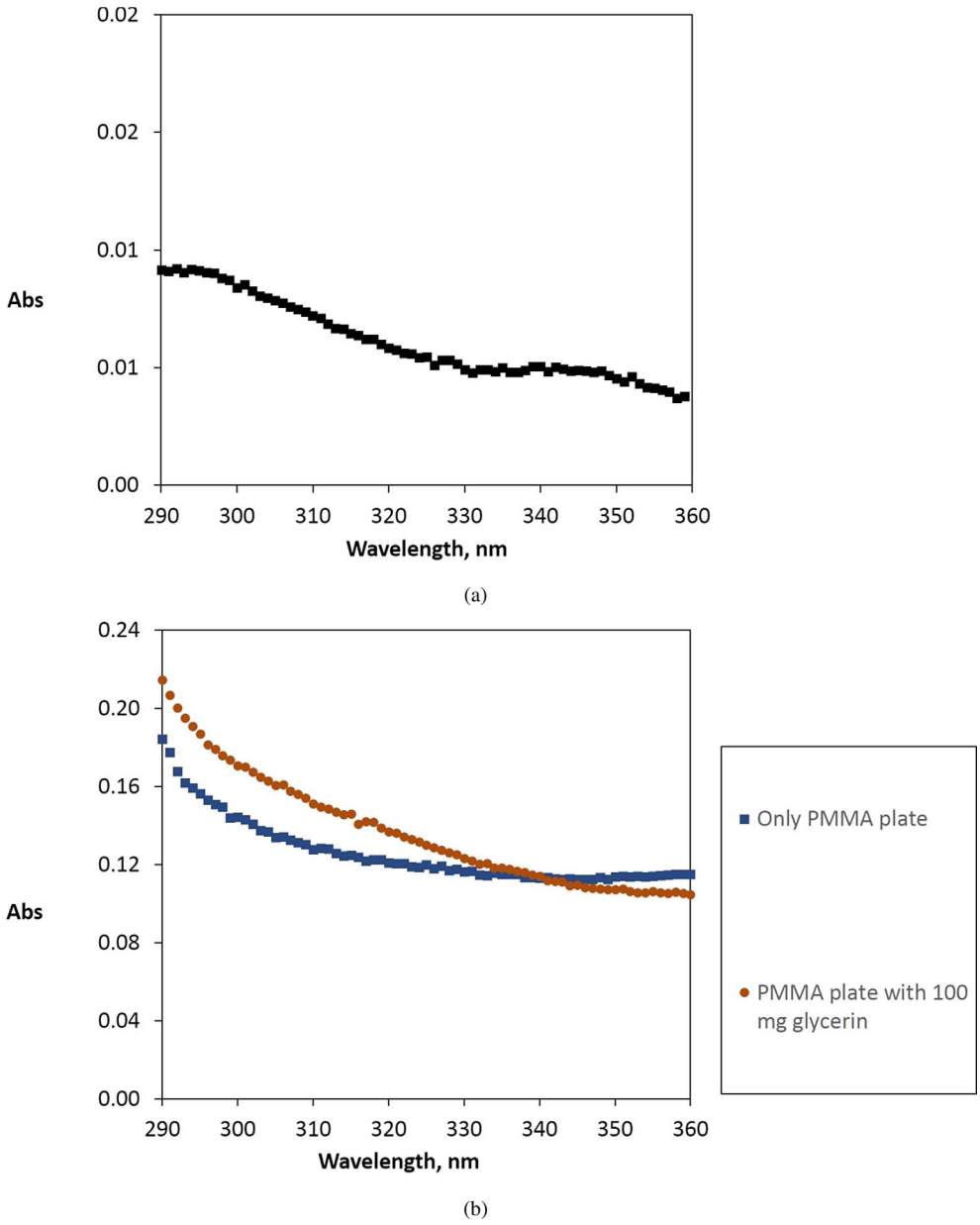


Figure 2. (a) UV absorbance of the 1 wt% SDS solution. (b) UV absorbance of a PMMA plate with and without glycerol.

actual UV absorbance can be less than the value measured here.

UV absorbance of microcapsules

In this section, the total applied OS (loaded in the polyurea microcapsules) on the PMMA plates are controlled at 33 ± 3 mg. The chemical systems for characterizing the UV absorbance of microcapsules are summarized in Table 1. To simplify calculations, we use directly the concentration of the encapsulated OS rather than the concentration of the microcapsules in capsule suspension. For different c_{HDB} (or shell thickness), the UV absorbance of microcapsules is given in Figure 4. Note that the UV absorbance contributed by the blank PMMA plate is subtracted.

The average absorbance at the wavelength of 320 nm is 0.123 for the thinnest microcapsule (c_{HDB} = 5 wt%) and 0.154 for the thickest one (c_{HDB} = 30 wt%), which slightly increases with the c_{HDB} (or shell thickness). This observation seems to be contrary to our first expectance that higher UV absorption should have been obtained with a thinner shell thickness of microcapsules. Overall, the UV absorption efficiency for all the microcapsules in this study is not as high as the pure OS. For the microcapsules with a calibrated size of 78 μm, the maximum UV absorption efficiency at the wavelength of 320 nm is calculated as,

$$\frac{0.154 \times 100\%}{1.25} = 12.3\%. \quad (1)$$

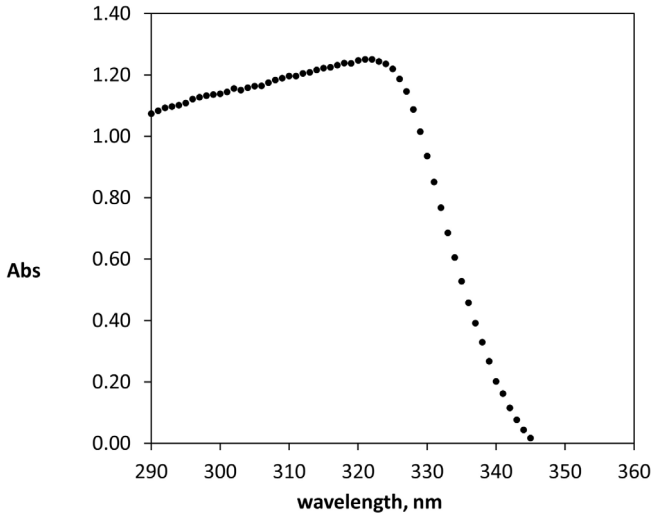


Figure 3. UV absorbance of 30 mg of pure OS on a PMMA plate.

Table 1. The chemical systems for measuring the UV absorbance of the microcapsules on PMMA plates.

Sample name	c_{HDB}, wt%	Mass of capsule suspension, mg	Concentration of OS in suspension, mg/mg	Δm, mg
S1-1	30	122	2.94/10	33
S1-2	30	115	3.26/10	35
S2-1	20	90	4.00/10	33
S2-2	20	85	4.05/10	31
S3-1	10	95	3.80/10	33
S3-2	10	75	4.61/10	31
S4-1	5	89	3.89/10	31
S4-2	5	86	3.89/10	30

Discussion

To understand why the microcapsules have much lower UV absorption efficiency than that of pure OS, the principle for the measurement of UV absorbance should be clarified first. According to the Beer–Lambert law, the UV absorbance (Abs) for pure OS or microcapsules on PMMA plates can be expressed as,

$$\mathrm{Abs} = \log_{10}\left(\frac{1}{T_{\mathrm{UV}}}\right) = \log_{10}\left(\frac{1}{T_{\mathrm{UV}c} f_c + 1 \times (1 - f_c)}\right), \quad (2)$$

where $T_{\mathrm{UV}c}$ is the transmittance for the area that is covered by OS or microcapsules on a PMMA plate. The uncovered part $(1 - f_c)$ of the plate gives a transmittance of one (no absorption). f_c is the coverage fraction of the plate by pure OS or microcapsules.

For a plate that is covered completely ($f_c = 1$) by a homogeneous film of pure OS, the transmittance for the whole plate is the same. In fact, the transmittance for the wavelength of 320 nm can be calculated through equation (2),

$$\mathrm{Abs} = \log_{10}\left(\frac{1}{T_{\mathrm{UV}}}\right) = 1.25, \quad (3)$$

so T_{UV} is 0.056. It indicates that most UV rays in the range of 290–320 nm are absorbed by that pure OS film.

For the microcapsules loaded with OS, the f_c of the PMMA plate can be calculated as,

$$f_c = \frac{S_c}{2.5 \times 10^{-3}}, \quad (4)$$

where 2.5×10^{-3} (m^2) is the total surface area of the plate. S_c is the surface area covered by microcapsules, which can be calculated as,

$$S_c = N_m \pi r_{\mathrm{OS}}^2, \quad (5)$$

where N_m is the number of capsules that are deposed on the plate. r_{OS} (m) is the radii of the OS core inside a microcapsule. Because the total mass of encapsulated OS on a plate is fixed at Δm (kg), the numbers (N_m) of microcapsules can be calculated as,

$$N_m = \frac{\Delta m}{\frac{4}{3}\pi r_{\mathrm{OS}}^3 \rho_{\mathrm{OS}}}, \quad (6)$$

where $\left(\frac{4}{3}\pi r_{\mathrm{OS}}^3\right)$ is the volume (m^3) of the OS core in one microcapsule and ρ_{OS} (1012 $\mathrm{kg/m}^3$) is the density of OS at room temperature. The radii (r_{OS}, m) of the OS core inside the microcapsule can be calculated by the mass balance as,

$$r_{\mathrm{OS}} = r_d \sqrt[3]{\frac{\rho}{\rho_{\mathrm{OS}}}(1 - c_{\mathrm{HDB}})}, \quad (7)$$

where ρ ($\mathrm{kg/m}^3$) is the density of the droplet phase and r_d is the radii of the capsule, which can be found in our previous study [2]. Thus, the total covered surface (S_c) as well as the coverage fraction (f_c) of the plate are summarized in Table 2.

For the microcapsules with different shell thickness, their f_c values are not so varied. According to equation (2), the UV absorbance for the capsules with different shell thicknesses should also be close. Indeed, our experiment results in Figure 4 about UV absorbance of microcapsules confirmed this theoretical estimation.

Let us consider that the covered part of the plate has a transmittance of 0, the UV absorbance for the microcapsules produced with 30 wt% of c_{HDB} can be roughly estimated as,

$$\mathrm{Abs} = \log_{10}\left(\frac{1}{0 \times 0.29 + 1 \times (1 - 0.29)}\right) = 0.149, \quad (8)$$

where the measured absorbance is 0.154. The experimental value is slightly higher than the estimated one. This difference can be caused by several reasons. Firstly, there is only chemical UV absorption in our estimation but scattering effects may also contribute during the measurement and thus give a higher apparent UV absorbance. Besides, there may be a few capsules that are broken during the spreading-out process by fingers. The released OS can cover more space on the PMMA plate and thus also give a higher UV absorbance.

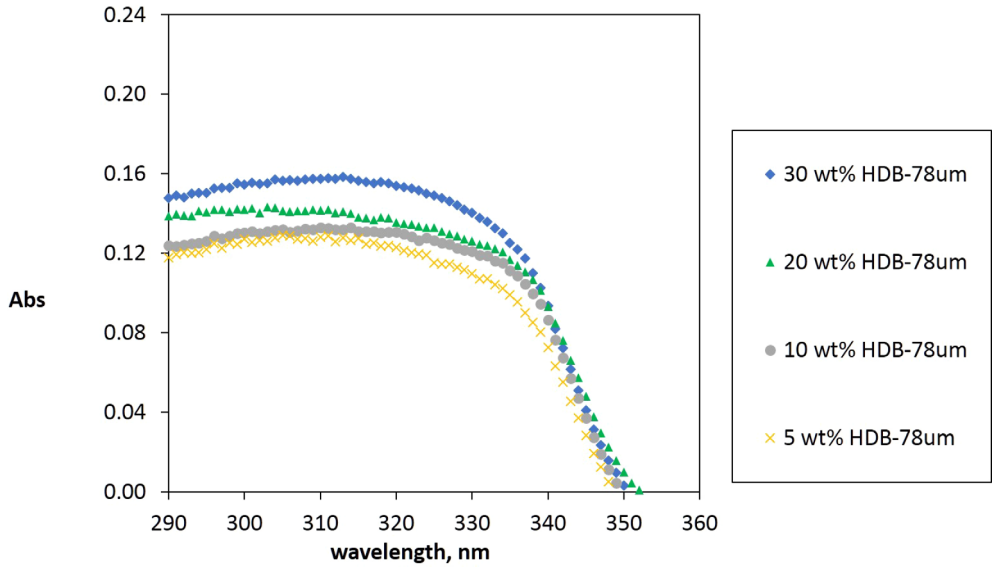


Figure 4. UV absorbance of the microcapsules synthesized with different c_{HDB} on PMMA plates.

Table 2. Calculated r_{OS}, S_c and f_c for the microcapsules synthesized at different conditions.

c_{HDB}, wt%	r_d, m	r_{OS}, m	Δm, kg	S_c, m^2	f_c
30	3.9×10^{-5}	3.5×10^{-5}	3.4×10^{-5}	7.2×10^{-4}	0.29
20	3.9×10^{-5}	3.6×10^{-5}	3.2×10^{-5}	6.5×10^{-4}	0.26
10	3.9×10^{-5}	3.8×10^{-5}	3.2×10^{-5}	6.3×10^{-4}	0.25
5	3.9×10^{-5}	3.8×10^{-5}	3.1×10^{-5}	5.9×10^{-4}	0.24

Though the currently used microcapsules give a relatively low UV absorption efficiency compared with pure OS film, at least it is confirmed that shell thicknesses of microcapsules have no obvious influence on the UV absorbance. However, the coverage fraction of PMMA plates does affect UV absorbance significantly. To achieve a high UV absorption efficiency, either we can apply more microcapsules on a PMMA plate, or we can increase the surface coverage fraction of a PMMA plate by decreasing the size of microcapsules.

Conclusion

In this study, an entire UV measurement protocol for the characterization of microcapsules is proposed and validated, which includes the calibration tests for the PMMA plates and suspension medium. The microcapsules loaded with octyl salicylate were tested for their UV absorbance on PMMA plates. The UV absorption efficiency of current microcapsules with the size of 78 μm is lower than the pure OS film. And no obvious influence of shell thicknesses of microcapsules on the UV absorbance of microcapsules is observed.

Besides, we find that the UV absorbance of microcapsules is linked to the surface coverage of PMMA plates. Therefore, we target to produce smaller capsules than the current ones in the next work. And we will explore the relationship between sizes of microcapsules and the UV absorbance of microcapsules. Except that, a more precise model needs to be established for estimating the UV absorbance of microcapsules of different sizes.

Conflict of interest

All the authors certify that they have no financial conflict of interest (e.g., consultancies, stock ownership, equity interest, patent/licensing arrangements, etc.) in connection with this article.

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