(REFEREED RESEARCH)

VITAMIN E LOADED FABRICS AS COSMETOTEXTILE PRODUCTS: FORMULATION AND CHARACTERIZATION

KOZMETİK TEKSTİLLER OLARAK VİTAMİN E AKTARILMIŞ KUMAŞLARIN FORMULASYON VE KARAKTERİZASYONU

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ABSTRACT

Skin fights constantly during the day to be saved from free radicals caused by UV rays and pollution. However, skin cells repair damage and restore complexion during sleep. Enhancement of repair and restoration can be achieved more effectively by the cosmetic products such as antioxidants applied during night. In this study, functional fabrics were prepared for single-use which are impregnated with three different delivery systems containing vitamin E, the mostly known antioxidant ingredient. Comparison of vitamin E release from microcapsule, microemulsion and solid lipid nanoparticle systems embedded in polypropylene fabrics (PP) was aimed in this study. Final purpose of preparing a cosmetotextile for ocular area was to obtain prolonged activity of vitamin E. Following particle size measurement and scanning electron microscopic analyses of all delivery systems prepared, systems embedded in polypropylene nonwoven fabrics were tested for vitamin E meant to be released over time. According to the results obtained, vitamin E was found to be successfully incorporated into all three delivery systems and release of vitamin E was determined to be prolonged best by solid lipid nanoparticles.

Keywords: Vitamin E, microparticle, microemulsion, solid lipid nanoparticle, polypropylene fabric

ÖZET

Deri gün içinde UV ısınlarından kaynaklanan serbest radikallerden ve hava kirliliğinden korunmak için sürekli olarak savaşır. Fakat, deri hücreleri oluşan hasarı uyku sırasında onarır ve restore eder. Gece cilde uygulanan antioksidan kozmetik ürünler onarımın gelişmesine yardımcı olur. Bu çalışmada, antioksidan etkisi ile bilinen vitamin E içeren kompleks yapı üç farklı sistem ile oluşturulmuş ve emdirme yöntemiyle polipropilen dokusuz yüzeye aktarılarak tek kullanımlık fonksiyonel kumaşlar elde edilmiştir. Üç farklı yöntem ile üretilen vitamin E komplekslerinin partikül boyut ölçümleri yapılmış ve polipropilen kumaşa aktarılmış olan yapıların taramalı elektron mikroskobik görüntüleri, vitamin E'nin mikrokapsül sistemi, mikroemülsiyon sistemi ve katı lipit nanopartikül sisteminden salımı karşılaştırılmıştır. Test sonuçlarına göre, vitamin E kompleksleri üç farklı yöntem ile başarılı bir şekilde oluşturulabilmiş ve en uzun salımın katı lipid nanopartiküller sistemine ait olduğu belirlenmiştir.

Anahtar Kelimeler: Vitamin E, mikropartikül, mikroemulsiyon, katı lipit nanopartikül, polypropilen kumaş

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1. INTRODUCTION

Human body naturally enhances its ability to fight environmental and natural damage. Sleep reduces cortisol (stress hormone) which is responsible for thinning skin, stretch marks, and discoloration while increasing melatonin (sleep hormone) which acts like an antioxidant to fight age spots, fine lines, or the worst skin cancer. A good sleep can also increase the efficiency of special growth hormones that

repair and regenerate collagen-producing cells, which are responsible for skin's elasticity and tightness. Therefore it can said that the body is repaired and detoxified with sleep [1].

Metabolic rate slows down during sleep and therefore free radicals decrease resulting in low consumption of antioxidants. Enhancement in repair process of skin cells may be achieved by antioxidants which promote cell turnover and remove toxins during sleep. It is well known that applying cosmetic products at night is better owing to deeper penetration of active ingredients with no exposure to sunlight or pollution and no competition with other cosmetic products like make-up and also higher skin temperature[2].

Vitamin E (VE) is a reference ingredient which is a collective name for a group of fat-soluble compounds with distinctive antioxidant activities [3]. Naturally occurring VE exists in eight chemical forms (*eg* alpha-, beta-, gamma- and delta-tocotrienol) that have varying levels of biological activity. Among them, α-tocopherol is the most abundant and biologically active compound to fight free radicals [4]. Free radicals containing highly energetic unshared electrons form reactive oxygen species (ROS). Body forms ROS endogenously or by environmental exposures such as UV radiation and air pollution. VE, a potent antioxidant, is used widely in skin care industry as a topical agent in an attempt to prolong the young appearance of skin [5, 6].

Since VE is sensitive to light, high temperatures and moisture like almost all other vitamins, it has to be protected from the environmental conditions [7]. Temperature and light sensitivity of α-tocopherol is the reason why commercial products are prepared with encapsulation, embedding or complexation [8, 9]. It is also possible to apply stable VE on textile (cosmetotextile) enabling controlled release during wearing [10].

Encapsulation is a method of preparing microcapsules with a core material surrounded by a shell material. Microencapsulation technology provides controlled release of the core material and also reduction in its toxicity [9, 11, 12]. When applied on textiles, microencapsulation adds functional finishes and properties which demonstrate many advantages in terms of economy, energy saving, eco-friendliness and controlled release of ingredients in comparison to conventional processes [10-12, 13, 44,45].

Microemulsion systems encapsulating active ingredients are ternary or pseudoternary dispersed systems comprising oil, water and surfactant. They possess some unique characteristics such as optical transparency, Newtonian flow type, ease of preparation, physical stability, biocompatibility/ biodegradability, imparting long shelf-life and cost-effectiveness [14].

Solid lipid nanoparticles (SLN) represent an alternative carrier to traditional colloidal carriers such as liposomes, emulsions, and biodegradable polymeric nanoparticles. SLN are prepared by replacing the liquid lipid (oil) of an o/w emulsion with a solid lipid or a mixture of solid lipids. SLN system has many advantages such as low-cost ingredients, ease of preparation and scale up, rapid dispersion in aqueous environment, high biocompatibility/biodegradability with improved storage stabilities [15, 16].

Cosmetotextiles are high-performance textiles representing a fusion of fabric materials with cosmetic active substances [17]. Cosmetic ingredients are embedded in fabrics and when applied to the skin, active ingredient is released from the textile to the skin to meet the specific cosmetic purpose. Despite not being accepted as cosmetic products, they are able to impart skincare benefits and fight ageing [18]. Commercial cosmetotextiles claim moisturizing, cellulite reducing, perfumed, body slimming, energizing, rejuvenating, refreshing, firmness improving and elasticity enhancement, reducing fine lines and wrinkles benefits [19].

Exposure to environmental stress upsets the balance between cellular antioxidant and oxidant levels and also damages the defense mechanism of cells which leads to skin aging [20-25]. Being destitute of sebaceous glands, preorbital area is thin and dry. Preaging symptoms are first apparent in the preorbital area due to daily facial mimics with the existence of its intensive muscular tissue [26]. Prolonged antioxidant activity of VE incorporated into a functionalized textile was aimed in this study to be used as a sleep mask acting on the preorbital area. For this purpose, three different delivery systems of VE prepared, namely microcapsules (MC), microemulsions (ME) and solid lipid nanoparticles (SLN) were evaluated to determine the effect of different systems on VE release from the textile material. MC, ME and SLN prepared were applied on nonwoven polypropylene fabrics for a potential use as sleep mask.

2. EXPERIMENTAL

2.1. Materials

VE (α-tocopherol) was purchased from Fluka Chemicals, Turkey while arabic gum from Zag Chemical, Turkey, sodium sulphate, gluteraldehit and caprylic acid ester from Sigma Aldrich, Turkey, glyceryl behenate (Compritol® 888 ATO) from Gattefossé, France and polyoxyethylene-80 sorbitan monooleate (Tween® 80) and polyethylene glycol 400 (PEG 400) from Merck, Germany. Polypropylene nonwoven fabric (70 g/m^2) was obtained from Teknomelt Kahramanmaraş, Turkey.

2.2. Preparation of Formulations

In this study, three different delivery systems, MC, ME and SLN incorporating VE were prepared. Two methods, namely simple hot (SHC) and simple cold coacervation (SCC) methods were used to prepare MCs. Intact VE and all formulations prepared were each applied to fabrics.

2.3. Preparation of Microcapsule Formulation

Microencapsulation is one of the methods which provides incorporating materials for release under controlled conditions mechanically, electrically, chemically or by leaching action in a liquid environment [9, 11, 12]. Microencapsulated VE was reported to significantly increase skin moisture and elasticity and also reduce skin wrinkle and roughness [27].

Coacervation method which is among the other encapsulation methods is considered as spontaneous liquid/liquid phase separation in colloidal systems occurring through electrostatic interaction between the two oppositely charged colloids (Figure 1) [12, 28]. This method was selected for preparing microcapsules owing to its ease in application.

Following preliminary studies for determining the optimum parameters of preparation considering particle sizes, coacervation method comprised of dissolving arabic gum in distilled water at a ratio of 1:5 and subsequent addition of 4 g VE to this solution. Addition of VE to the arabic gum solution was performed at two different temperatures of 22ºC (SHC) and 10ºC (SCC) to determine the effect of temperature on preparing MCs. The mixture was stirred at 1600 rpm prior to the addition of 1 ml sodium sulphate solution (25%) to separate the shell and core materials. 1.7 g gluteraldehyde was then added under continuous stirring for solidifying the shell material. Finally, cooling was carried out at -18ºC for 12 hours. The simple coacervation method used is summarized in Figure 2.

In simple coacervation method, shell material is carried out disperse with a dissolver. After, core material is added in the dispersion and phase separation is achieved with decrease in temperature or changing of pH.

2.4. Preparation of Microemulsion (ME) Formulation

ME system (oil/water) was prepared as reported previously [29]. Briefly, VE (8 % w/w) was dissolved in the oil, *ie* caprylic acid ester. Tween® 80 as the surfactant and PEG 400 as the cosurfactant were added into the oily phase and the mixture was stirred using 1500 rpm at 70ºC for 15 min. Distilled water was then added dropwise into the oily solution. The lightly blurred microemulsion was agitated using ultraturrax (Janke&Kunkel IKA, Germany) at 8000 rpm for 2 min and a transparent microemulsion was obtained. Preparation of the ME system is summarized in Figure 3.

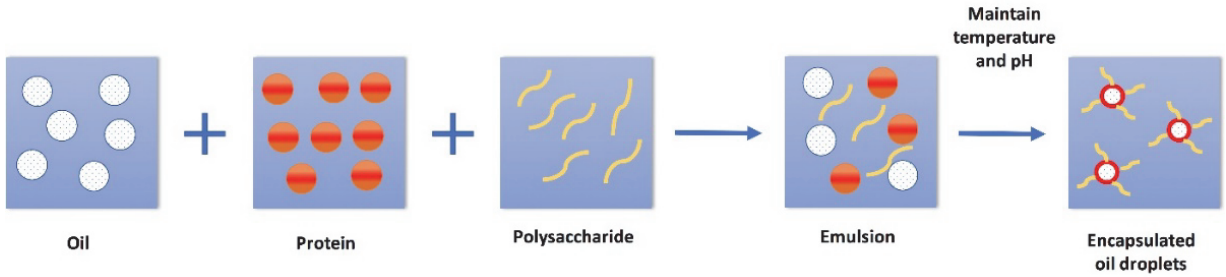


Figure 1. Coacervation method of microencapsulation [12].

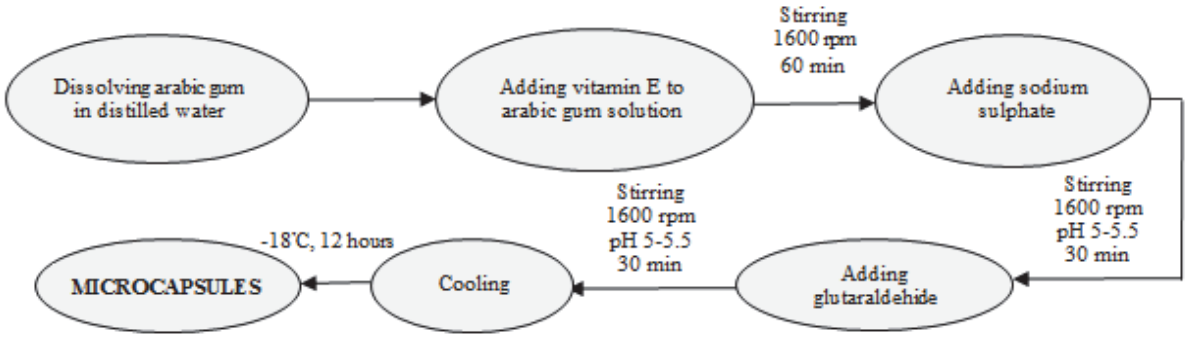


Figure 2. Simple coacervation method for MC preparation.

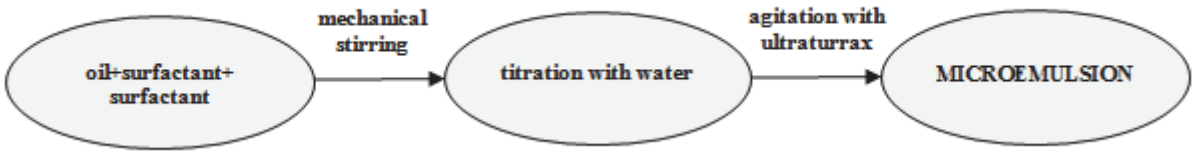


Figure 3. Microemulsion preparation.

2.5. Preparation of Solid Lipid Nanoparticle (SLN) Formulation

Hot homogenization technique was used for the preparation of SLN system which is suitable for lipophilic ingredients. SLN system developed and reported previously by our group was repeated for this study [29]. The solid lipid, Compritol® 888 ATO, was heated 5-10 ºC over its melting point (70° C) and VE was dissolved in this melt. Aqueous phase containing the surfactant Tween® 80 (0.8 % w/w) was also heated to the same temperature followed by mixing with the melt using ultraturrax to obtain a pre-emulsion. The pre-emulsion was homogenized further with ultrraturrax at 11000 rpm for 10 min. The resulting mixture was then cooled down to room temperature and SLNs were collected after crystallization of lipids (Figure 4).

2.6. Application to Fabric

Untreated polyproplene nonwoven fabric which had hydrophilic pre-treatment before the applications, was conditioned at 20ºC±2ºC and 65 %±2 humidity for 24 hours. Polyproplyene fabric was selected because it is one of the mostly used fibre type intended for a single use. After the conditioning process, VE aqueous solution, MCs prepared both at 22ºC and 10ºC, ME and SLN dispersion were applied to fabrics by pad-dry method with 70 % wet pick-up ratio obtaining VEF (VE treated fabric), SHCF (fabric treated with microcapsule synthesized at 22°C) and SCCF (fabric treated with microcapsule synthesized at 10°C), MEF (microemulsion applied fabric) and SLNF (solid lipid nanoparticle applied fabric). After the dipping process, the samples were dried at room temperature for 8 hours. The wet pick-up (wpu) was measured at 60 % for all applications and was calculated by the following equation,

$$wpu\ \% = \frac{E_2 - E_1}{E_1} x100 \quad (1)$$

where wpu is the amount of solution applied to the fabric, E_2 is the fabric weight after dipping and E_1 is the fabric dry weight. After the drying process, the weight of samples was measured and the capsules pick-up was calculated at 40 %.

2.7. Particle/Droplet Size Analysis

Particle/droplet mean size and size distribution (PDI) of formulations were measured using Malvern Zetasizer-Nano ZS (Malvern Instruments Limited, Worcestershire, UK) with a dynamic light scattering method at 25°C.

2.8. Scanning Electron Microscope Analysis

Surface morphology of polypropylene fabric samples were scanned by an electron microscope (ZEISS/EVO 40, Germany) at 2000 magnification and 10 kV under a high vacuum after being coated with gold-palladium at a thickness of 40-50 nm using BAL-TEC SCD 005 coating device.

2.9. High-Performance Liquid Chromatography Method (HPLC)

Quantification of VE was needed during the release study. Even though some other methods were used for the quantification of VE, HPLC method is more rapid, precise and accurate compared to other methods [30]. Therefore, HPLC method validated previously was used for determining the release of VE from different fabrics prepared [27]. In this method, 300 $\mu g.mL^{-1}$ stock solution was diluted to obtain the concentrations in the range of 30-150 $\mu g.mL^{-1}$. The mobile phase was methanol-acetonitrile (95:5) with a flow rate of 1 $mL.min^{-1}$; temperature was 30ºC and the injection volume was 20 µL. The size of the column used was 4.6 mm x 250 mm packed with 5 µm C18. The method was validated for linearity, precision and accuracy with reference to the ICH guidelines [31].

2.10. *In Vitro* Release from Fabrics

Franz diffusion cells were used for *in vitro* release studies [29, 32]. The diffusion cells were thermoregulated with a water jacket at 32ºC. 1 cm^2 of each fabric sample was cut and placed as a donor phase and receptor phase was selected as 50 mL ethanol under continuous stirring at 100 rpm with magnetic stirring for 12 hours. The receptor fluid was kept at a constant temperature of 32 ºC. After 5, 15, 30, 45, 60 and 90 min and every 60 min after 1 hour, 1 mL of samples collected with *Pasteur* pipette was put into vials. Equivalent amount of ethanol was added to the receptor solution to replace the amount withdrawn. Generally, the receptor phase is either isotonic solution or another aqueous medium. However, ethanol was used as the receptor phase in this study due to the extremely low solubility of VE in aqueous medium.

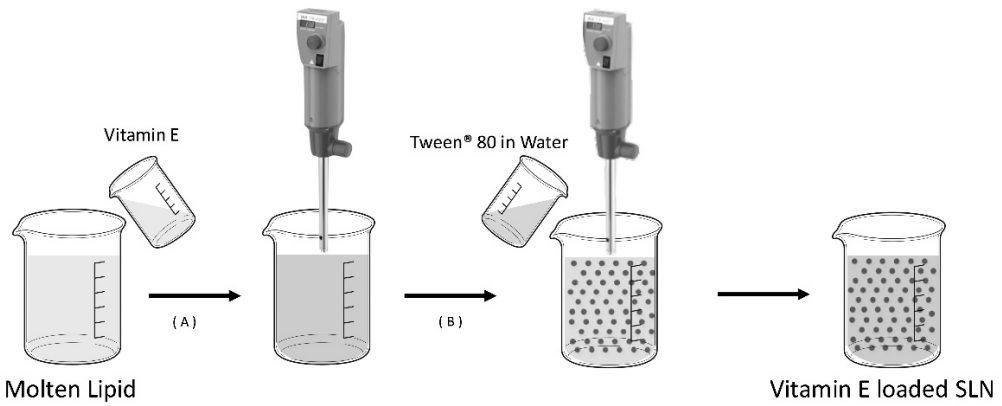


Figure 4. SLN preparation.

3. RESULTS and DISCUSSION

3.1. Particle/Droplet Size Analysis

Particle/droplet sizes and size distributions of MCs synthesized at two different temperatures, ME and SLN are given in Table 1. Sizes of ME, SLN, SHC and SCC formulations were determined to be in the nanometer range. Size distributions were relatively monodisperse in all formulations with the PDI values of ≤ 0.9.

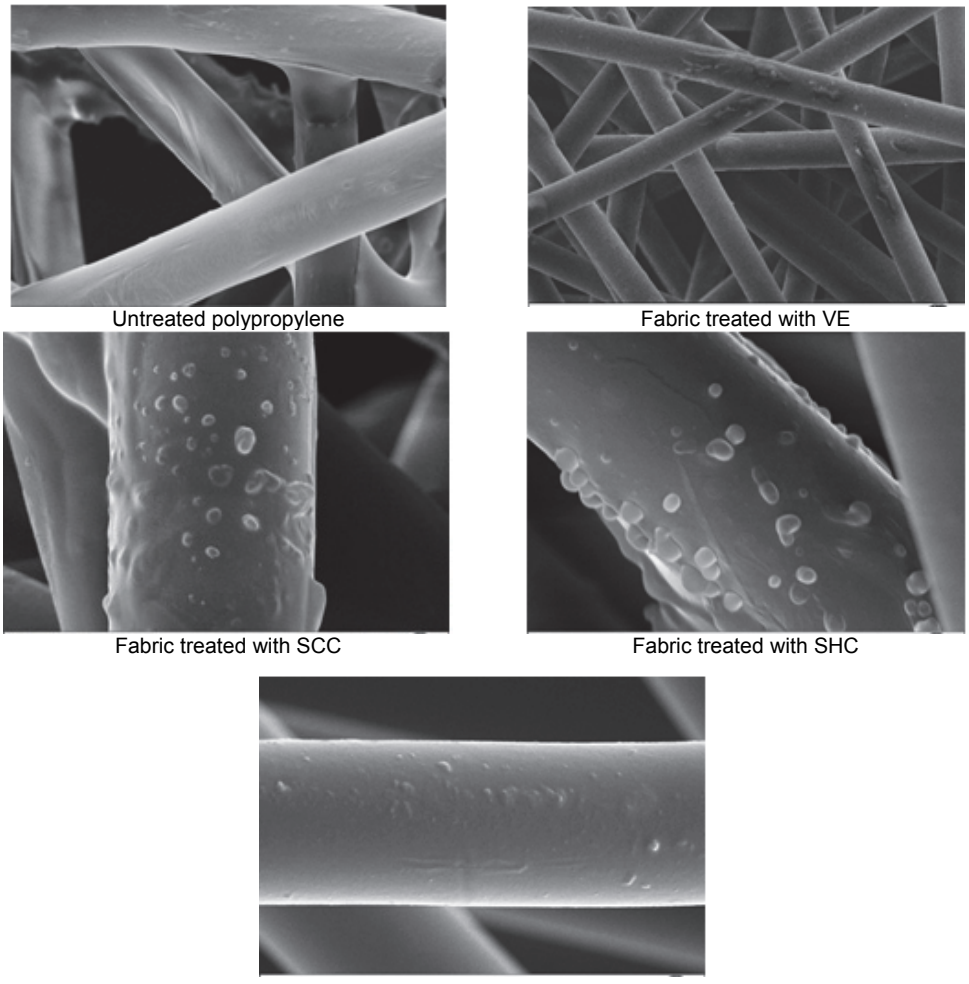
Particle size distribution and the size of particles play a key role in their adhesion and interaction with biological cells [33, 34]. According to Table 1, the mean particle/droplet size of VE loaded formulations ranged from 124.39± 0.78 nm to 368.00±1.06 nm with a relatively monodisperse distribution. It is well known that smaller particle/droplet size helps in targeting and increasing the penetration of the active ingredient through biological membranes [35].

3.2. Scanning Electron Microscope (SEM)

SEM images of untreated fabric and those treated with MC and SLN are shown in Figure 5. While untreated polypropylene fabric showed a smooth surface with no superficial particle/droplet, particles/droplets deposited on the textile fibers were observed on the fabrics treated with SCC, SHC and SLN. In the SEM image of fabric treated with VE, chemical residues were indicated on the surface of the fiber since vitamin E was applied directly on the fabric without using any delivery system in order to make a detailed comparison between treated samples. While the droplets of fabrics treated with SCC and SHC were observed clearly on the surface of the material and they were so similar to each other because of being a part of same delivery system (MC), droplets/particles of fabric treated with SLN were observed as more penetrated into the fabric. This could be because of particles/droplets that were partially molten or fused during the deposition process of SLN [36].

Table 1. Particle/droplet size and size distribution of formulations.

Formulation	Particle/ Droplet size (nm) ± SD	PDI ± SD
SHC	297.64 ± 0.96	0.599 ± 0.022
SCC	124.39 ± 0.78	0.832 ± 0.028
ME	325.00 ± 0.63	0.211 ± 0.030
SLN	368.00 ± 1.06	0.154 ± 0.160



Untreated polypropylene

Fabric treated with VE

Fabric treated with SCC

Fabric treated with SHC

Fabric treated with SLN

Figure 5. SEM images of fabrics.

3.3. High-Performance Liquid Chromatography (HPLC)

Retention time of VE was 11 min according to HPLC method used. Linear equation for VE was determined to be y = 608.4211x – 144.678 regarding peak area (y) and injected amount (x, µg). Limit of quantification value which is defined as the lowest concentration of VE which can be detected with acceptable precision was found to be 0.1038 µg/mL while limit of detection value defined as the lowest detection limit was 0.013 µg/mL.

3.4. *In Vitro* Release of VE from Fabrics

VE is a novel active agent investigated by cosmetic scientists. In a study investigated it was shown that chitosan nanoparticles were promising encapsulation and delivery system for prolonged release [37] of VE. Also in another study it was entrapped in poly (D,L-lactic-co-glycolide) nanoparticles [38] and also VE incorporated melamine-formaldehyde microcapsules were embedded in cotton fabrics [10, 39]. Apart from these, another research about pharmaceutical textiles imprinted with lipid microparticles of Econazole nitrate (ECN) was carried out in order to improve patient compliance while maintaining drug activity. Comparison of cosmetotextiles containing particles prepared and a commercial formulation showed different skin distributions where the particles prepared were mainly distributed in the *Stratum corneum* penetrating less in epidermis and dermis while commercial formulation was mainly found in dermis [40]. This difference was attributed to the difference in particle sizes of both formulations. As a result of this study, functionalized textiles such as bandages and socks were suggested for targeted delivery of active ingredients [40]. However, application onto textiles were poorly characterized and examined in the mentioned study.

In vitro release of intact VE and VE from all fabrics prepared by embedding MC, ME and SLN are shown in Figure 6. Rapid dissolution of intact VE and SHCF was determined within the first 45 minutes. Following a burst release from SCCF and MEF, total release from SCCF was seen in around 80 min while MEF was released totally within 2 hours.

Release profile of SLNF was observed to give prolonged pattern around 300 min (over 4 hours). It is clear that a significant improvement in VE release was achieved with MEF and SLNF. However, the best extended release was obtained with SLNF. For SLNF, VE slowly diffused to the exterior medium since it was entrapped in the solid lipid matrix [41]. In a previous study, thermo-sensitive behavior of textiles was confirmed by drug release profiles obtained at room temperature and at 32ºC with Franz diffusion cells [42]. This suggested the reservoir property of textiles as was determined for SLNF.

Release of active substances from nanocarriers is usually dependent on several factors: penetration of the release medium into particles/droplets, diffusion of the active ingredient through the matrix, degradation of the particle/droplets and the release/transfer of particles/droplets to the release medium [43]. The burst release can be attributed to the amount of VE adsorbed onto the microparticle/nano-droplet surface. Affinity of VE to the release medium can also contribute to its rapid diffusion to the medium.

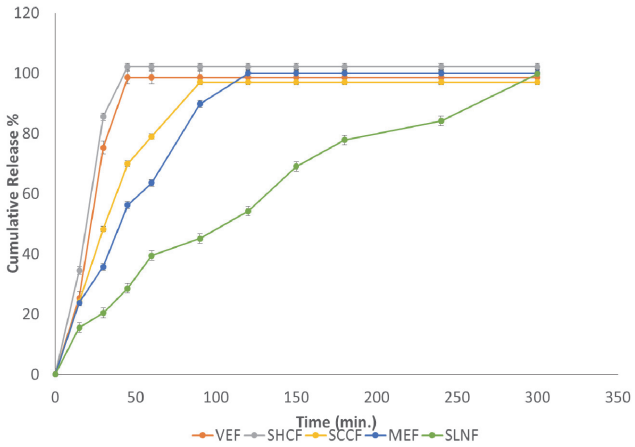


Figure 6. *In vitro* release of VE from fabrics (n=3).

4. CONCLUSION

In this study, polypropylene fabrics were functionalized using three different vitamin E delivery systems for potential preparation of cosmetotextiles. Particles/droplets prepared were applied on hydrophilic polypropylene nonwoven fabric by the pad-dry method. VE (α-tocopherol) release from the fabrics impregnated with microcapsule, microemulsion and solid lipid nanoparticle systems was compared. SEM images of functionalized fabrics showed loading of VE into the fabrics. SLNF presented the best prolonged release of VE in comparison to other delivery systems. Cosmetic application as a sleep mask was determined to be promising with particles/droplets developed in this study where VE can impart vital benefits such as skin protection, antiaging or moisturizer. Conclusively, it was shown that controlled release formulations can be loaded to fabrics with promising functional and innovative cosmetic properties. However, further *in vivo* studies are needed to verify the cosmetic benefits of cosmetotextiles on skin.

5. ACKNOWLEDGEMENT

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