



Phase Behaviour Study of Pitaya Seed Oil: Jojoba Oil with Non-Ionic Surfactants in Emulsion System

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Pitaya seed contains good antioxidant capacity while jojoba contains many different varieties of tocopherols which make up vitamin E to promote healthy and clear skin. The phase behaviour of systems has been investigated by constructing ternary phase diagrams consisting of pitaya seed oil: jojoba oil/non-ionic surfactant/water. Different HLB value of non-ionic surfactants exhibit different ternary diagram characteristics. A lower HLB shows a more oil-soluble and a more water-soluble surfactant (larger homogeneous and isotropic region in ternary phase diagrams) whereas high value of HLB shows the reverse of that result. The results showed that the Tween85 gave better solubility in water to produce larger isotropic and homogeneous regions for jojoba oil, pitaya seed oil and pitaya seed oil: jojoba oil. The presence of optimal HLB value of Tween85 for the stabilization of o/w emulsions contributes to the enlargement of the single phase regions.

Keywords: Pitaya seed oil, Jojoba oil, Non-ionic surfactant, Phase diagram, Emulsion system.

INTRODUCTION

As unique as its appearance, pitaya contains the characteristic of health properties. Essential fatty acids, namely, linoleic acid and linolenic acid form a significant percentage of the unsaturated fatty acids of the seed oil extract. Essential fatty acids (EFA) are important acids that are necessary in diet. However, two essential fatty acids, linolenic and linoleic acid cannot be synthesized in the body and must be obtained from food. Previous studies on red and white Malaysian *Hylocereus* seeds, after thermal degradation of fruit cells for seed separation, revealed high contents of linoleic acid and nutritionally favourable low ratios of saturated fatty acids^{1,2}. Due to its high contents of unsaturated fatty acids, like linoleic acid and the presence of vitamin E, pitaya seed oil may represent an interesting source for the food and cosmetic industries. Due to its high levels of linoleic acid, pitaya seed oil may help in relieving rough skin and maintaining health of the body's protective barrier¹.

Other than pitaya seed oil, jojoba oil is one of the natural products used in the emulsion system. Jojoba oil is extracted from the seed of Jojoba plant with binomial name of *Simmondsia chinensis*. Jojoba is a desert shrub that grows wild in southern Arizona, north-western Mexico and neighbouring areas. Jojoba is grown commercially for its oil, a liquid wax ester that used widely in cosmetic, pharmaceutical, textile and

high-grade lubricant field. Jojoba can be considered as unique oil due to its extremely long structure (C36-C46) straight-chain wax ester and not a triglyceride. Since it is composed of wax esters, it is an extremely stable substance and does not easily deteriorate and the structure closely resembles that of your own skin sebum, making it an excellent moisturizer and ideal for all skin types. Jojoba oil is favourite oil used as a massage medium because it acts as an emulsifier with the skin's natural sebum and gently unclogs the pores and lifts grime and imbedded impurities. It contains myristic acid which also has anti-inflammatory actions and since it has similarity in composition to that of the skin's own oils, it is quickly absorbed and is excellent for dry and mature skins as well as inflamed conditions.

An emulsion is a mixture of two or more liquids that are normally immiscible which contains both a dispersed and a continuous phase, with the boundary between the phases called the "interface". Emulsion is the most common delivery system used in cosmeceutical field. Wide variety of ingredients can be prepared in the emulsion formed. Their properties enable the ingredient to be delivered and absorbed to the skin conveniently. The emulsions are made by dispersing oil in water using amphiphilic emulsifiers, which have hydrophilic and lipophilic terminals that enable the molecules to interact with both oil and water, by assembling themselves at the oil-water interface. The stable emulsions are best formulated with

emulsifiers or combinations of emulsifiers, which possess HLB values close to the required HLB of the oil phase. However, the chemical type of emulsifier also can affect the stability of the emulsions. Emulsions with ionic surfactant are stable by electrostatic repulsion between the micelles whereas for the non-ionic surfactant the stability is achieved due to the steric repulsion. Ternary phase diagram consists of three components system and formed by dispersing the mixed emulsifier in water without the addition of an oil phase and can be prepared to represent the continuous phase of the corresponding emulsion³.

Surfactant also known as surface active agent is the substance that has the tendency to concentrate at the surface or interfaces and lowering the surface tension (or interfacial tension) between two liquids or between a liquid and a solid. Surfactant molecules have either one tail or two; those with two tails are said to be double-chained. Different in structure most commonly according to polar head group that classified the surfactant into four types; which are anionic, cationic, amphoteric and non-ionic. The "tail" of most surfactants is fairly the same, consisting of a hydrocarbon chain, which can be branch, linear, or aromatic.

Non-ionic surfactants do not have an electrical charge, which makes them resistant to water hardness deactivation. They have been widely used in domestic and industrial detergents and related products⁴. These surfactants are excellent grease removers that are used in laundry products, household cleaners and hand dishwashing liquids. Most non-ionic surfactants are considered to be more effective in cleaning applications at low concentration and in removing oily soil from synthetic fabrics. Non-ionic surfactants can be classified into three categories: polyethylene oxide, poly (ethylene/propylene) oxide and polyhydric alcohol, based on their hydrophilic groups. The examples of non-ionic surfactants were polyoxyethylene(20) sorbitan tri-oleate (Tween85) and [polyoxyethylene(20) sorbitan mono-oleate (Tween80). The HLB value of Tween85 and Tween80 are 11 and 15 respectively. The efficiency of a surfactant is not determined solely by the amphiphilicity; it also depends on the HLB characteristics for this compound⁵.

The aim of this work was to study the phase behaviour of pitaya seed oil: jojoba oil with non-ionic surfactant by constructing the ternary phase diagram using five different non-ionic surfactants.

EXPERIMENTAL

Pitaya fruit was purchased from Great Sun Farm Pitaya, Teluk Panglima Garang, Selangor. The composition of pitaya seed oil are linoleic acid (49.6 %), oleic acid (21.6), palmitic acid (17.9 %) and stearic acid (5.49 %)¹ pitaya seeds were finely ground before being extracted with hexane. The fine ground seeds were soaked in hexane solution for 24 h in room temperature. Upon completion of oil extraction, hexane was discarded from the oil in the rotary evaporator. Sorbitan tri-oleate (Tween85) and sorbitan mono-oleate (Tween 80) were purchased from Fluka Chemie GmbH, USA. Hexane (HPLC grade) was obtained from Fisher Chemicals, UK. Jojoba oil was purchased from Making Cosmetics, Inc., USA with

compositions of Eicosenoic acid (80.0 %), Palmitic acid (3.0 %), oleic acid (15.0 %) and linoleic acid (5.0 %), SA Handbook.⁶ Deionized water was prepared in our laboratory.

Construction of ternary phase diagram: Pitaya seed oil: jojoba oil /non-ionic surfactant/deionized water were weighed ranging from 0:100 to 100:0 (pitaya seed oil: jojoba oil /non-ionic surfactant/ deionized water (w/w). The mixture with a total weight of 0.5 g was placed in a 10 mL screw-cap glass tube. The samples were then vortexed using vortex mixture for 5 min and then centrifuged for 15 min at 4000 rpm. The phase behaviours of the samples were examined through cross-polarized light. The experiment was repeated with the addition of deionized water according to its percentage from 0 to 100 %. The phase behaviours were determined from visual observation. Ternary phase diagrams were drawn accordingly.

Effect of phase behaviour of pitaya seed oil: Jojoba oil with respect to different non-ionic surfactants: Two non-ionic surfactants were selected to construct ternary phase diagram of pitaya seed oil: jojoba oil. They were polyoxyethylene (20) sorbitan tri-oleate (Tween85) and polyoxyethylene (20) sorbitan mono-oleate (Tween80). The HLB value of Tween85 is 11.0 and Tween80 is 15.0. Comparison among the ternary phase diagrams were made to see the changes that happened when the different HLB values of the non-ionic surfactants were used.

RESULTS AND DISCUSSION

Phase behaviour study of pitaya seed oil: Jojoba oil with respect to different non-ionic surfactants: Emulsion was produced by mixing two or more immiscible liquid which in this context referred to water and oil. The mixing of oil, water and emulsifier in different ratio can produce different types of emulsion systems. Emulsion also depends on the chemical nature, molecular structure and concentration of surfactants and other ingredients⁷. The mixtures of different surfactants have many industrial applications because they show better characteristic than their building units. Non-ionic surfactants in general Tween and Span in particular are safe agents for all biological tissue in general and for skin in specific⁸. These non-ionic emulsifiers are compatible with various ingredients used in the preparation of emulsions and are not affected by pH. They are supposed to have an enhancement effect on the skin barrier⁹. Construction of ternary phase diagrams is the best way to study all types of formulation.

In the study of phase behaviour of pitaya seed oil with jojoba oil using different non-ionic surfactants, six ternary phase diagrams were constructed. Jojoba oil, pitaya seed oil and pitaya seed oil: jojoba oil conditions were tested with different HLB values. The surfactants were Tween80 and Tween85, having HLB values of 15.0, 11.0 respectively. The changes of phase behaviour were observed in the ternary phase diagram.

Phase behaviour of T80/jojoba oil/deionized water: Fig. 1 depicts the ternary phase diagram of Tween80/jojoba oil/ deionized water. Tween80 carried the HLB value of 15.0 is higher compared to Tween85, in constructing the ternary phase diagram. Fig. 1 showed the presence of three regions

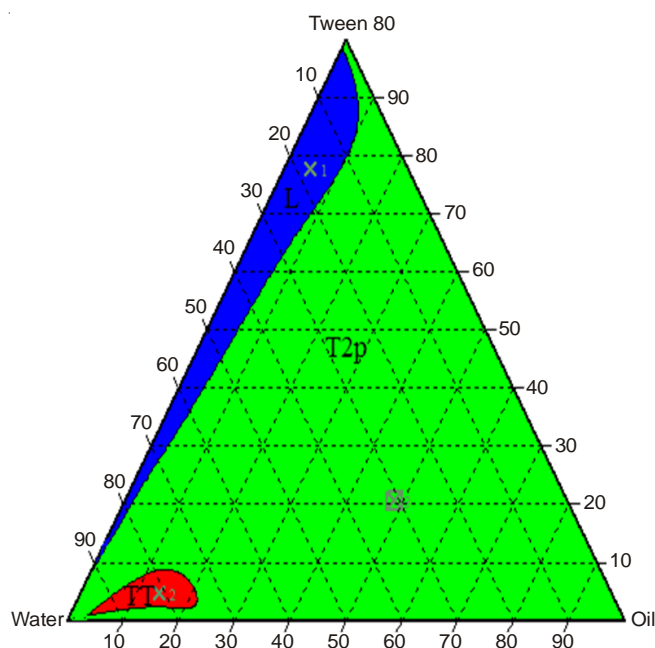


Fig. 1. Ternary phase diagram of Tween80/Jojoba oil/water. (L – Isotropic region; T2p – Two phase region; TT – Three phase region)

with different percentage of mixtures, the regions were isotropic (L), two phase (T2p) and three phase (TT) regions. The isotropic region was found along the apex line of deionized water from 2 to 90 %. The formation of one phase region suggested that the surfactant mixtures were able to lower the surface tension between the aqueous phase and oil phase, hence facilitates the formation of emulsions having a milky appearance¹⁰. Two-phase region donated as T2p domain covers most of the T80 and Jojoba oil apex line. This showed the instability and incompatibility in the emulsion system. The three phase region appeared at a water-rich corner and at low percentage of Tween80. Even though Tween80 is the best surfactant to form an o/w emulsion in the system, the formations of two and three phases are still exists.

Phase behaviour of Tween85/Jojoba oil/deionized water: Fig 2 shows the phase diagram of phase behaviour of Tween85/Jojoba oil/deionized water. The HLB value of Tween85 (11.0) is lower compared to Tween80 (15.0). Four phases were observed in this phase diagram. The homogenous region, milky emulsion system was found at the water rich corner in the system, meanwhile in Tween80/Jojoba oil/deionized water system, there is no homogenous region found. This is because for the stabilization of oil-in-water emulsions, surfactant with HLB value in the range 9-12 are optimal⁵ and suitable for emulsification. In the isotropic regions, clear emulsion system was appeared in two areas. The largest area was found along the apex line of Tween85 from 5 to 90 %. Second area was found at the middle of the ternary phase diagram. The two phase region cover along Tween85 and Jojoba oil apex line. The two phase regions domination also appeared when using Tween80 as an emulsifier. The three phase region was found at the water-rich corner in the system. This was due to the increasing in the percentage of deionized water. The area for three-phase region was rather small compared to Tween80 phase diagram.

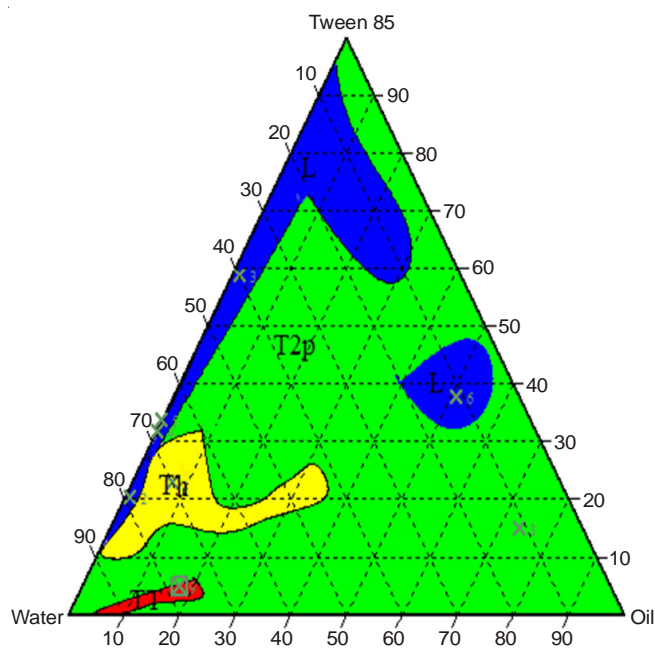


Fig. 2. Ternary phase diagram of Tween85/Jojoba oil/deionized water. (Th – Homogenous region; L – Isotropic region; T2p – Two phase region; TT – Three phase region)

Phase behaviour of Tween80/pitaya seed oil/water: The phase diagram of Tween80/pitaya seed oil/deionized water is shown in Fig. 3. A distinct one-phase region, isotropic region was observed. The isotropic region was found along the apex line of Tween80 and deionized water from 0 to 94 % and covers the most apex line of Tween80 and pitaya seed oil. The isotropic region occurred are larger compared to the ternary phase diagram of Tween80/Jojoba oil/deionized. The rest of the region consisted of two phase and three phase regions. Most of the two-phase region appeared at the middle of the system. The formation of two and three phases was due to the mixture being unable to

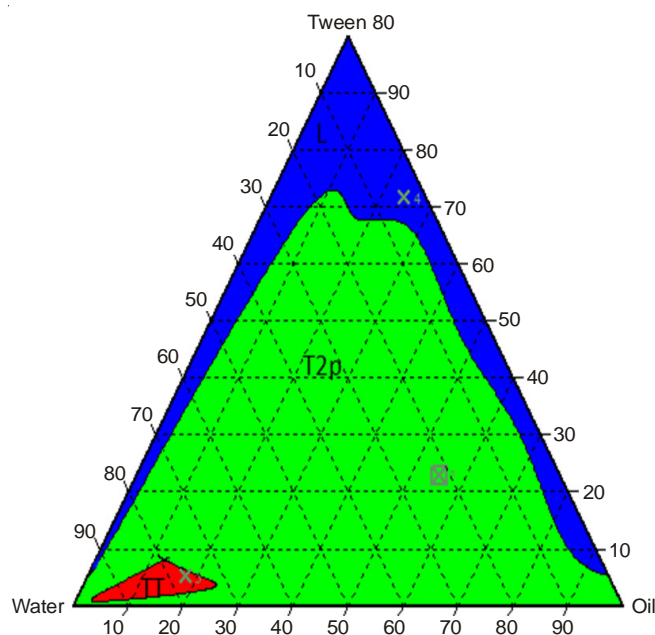


Fig. 3. Ternary phase diagram of Tween80/pitaya seed oil/deionized water. (L – Isotropic region; T2p – Two phase region; TT – Three phase region)

mix well and formed one phase mixture. The three-phase region appeared at the low percentage of Tween80 with the percentage of deionized water at 92 % and above.

Phase behaviour of Tween85/pitaya seed oil/deionized water: Fig. 4 depicts the ternary phase diagram of Tween85/pitaya seed oil/deionized water. Four phases were observed in this ternary diagram compared to Tween80. The homogenous region was appeared at the water rich corner in the system. The isotropic region was found along the apex line of Tween85 and deionized water from 4 to 70 %. The two-phase region covers along Tween85 and jojoba oil apex line. The two-phase region appeared in the middle of the system. The three-phase region was found at the water-rich corner in the system. It has a larger area compared to Tween85/jojoba oil/deionized water phase diagram. Pitaya seed oil works well with both Tween80 and Tween85. However there are more isotropic region, are formed in Tween80/pitaya seed oil/deionized water compared to Tween85/pitaya seed oil/deionized water. Whereas, the Tween85 formed both single-phase which is homogenous, the milky phase and isotropic, the clear phase.

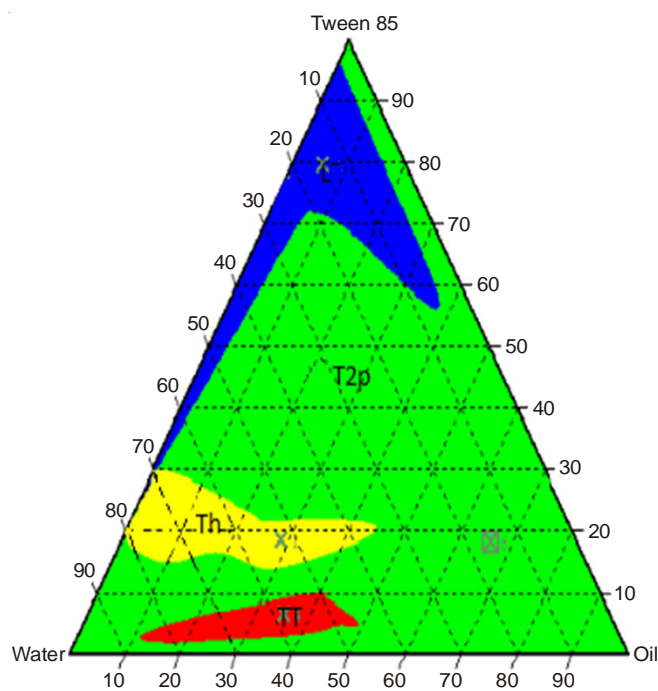


Fig. 4. Ternary phase diagram of Tween85/pitaya seed oil/deionized Water. (Th – Homogenous region; L – Isotropic region; T2p – Two phase region; TT – Three phase region)

Phase behaviour of Tween80/pitaya seed oil: jojoba oil/deionized water: Fig. 5 shows the ternary phase diagram of Tween80/pitaya seed oil: jojoba oil/deionized water. The isotropic region, single-phase was found along the apex line of Tween80 and deionized water from 6 to 85 %. The formation of a large two phase region was observed on the middle of ternary phase diagram. The reason of the inability of the surfactant to facilitate the emulsification process was probably due to no synergistic effect of the surfactant in enhancing the surface activity¹¹. Three phase region also appeared at water-rich corner in the ternary phase diagram. It has high percentage of Tween80 and low percentage of pitaya seed oil: jojoba oil.

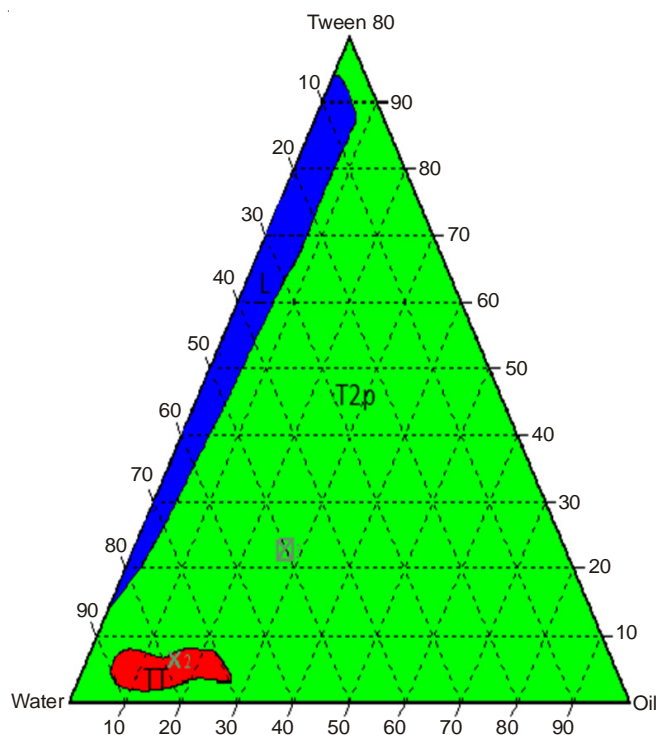


Fig. 5. Ternary phase diagram of Tween80/pitaya seed oil/de-ionized water. (L – Isotropic region; T2p – Two phase region; TT – Three phase region)

The ternary phase diagram of Tween80/pitaya seed oil: jojoba oil/deionized water is quite similar to Tween80/jojoba oil/deionized water phase diagram.

Phase behaviour of Tween85/pitaya seed oil: Jojoba oil/deionized water: Fig. 6 depicts the ternary phase diagram of Tween85/pitaya seed oil: Jojoba oil/deionized water. Similar to previous Tween85 ternary phase diagram, four phases were able to be observed. The homogenous region was appeared at water rich corner in the system having a quite large area. The emulsion formed could be classified as oil in water emulsion as the percentage of deionized water was higher than the percentage of oil. The isotropic region was found along the apex line of Tween85 and deionized water from 4 to 70 %. The two-phase region covers along Tween85 and jojoba oil apex line. The two-phase region appeared in the middle of the system. The three phase region was found at the water-rich corner in the system.

This study demonstrated the importance of selecting a surfactant with proper HLB for specific oil. The types and properties of surfactant will determine the types of emulsion formed. The HLB number could be used as a reference and guidance to determine whether the surfactant is suitable to use for several types of emulsions or not. In general, surfactants with lower HLB numbers (4-6) are mostly used as emulsifiers (water-in-oil), while those with higher HLB numbers (10-15) are detergents and surfactants with HLB numbers between 7 and 9 are suitable for wetting agents¹². However, in this study we wish to form an oil-in-water emulsion. According to Griffin's theory, to select a surfactant properly for any application, one must have the optimal HLB value and the correct chemical group. For the stabilization of oil-in-water emulsions, surfactant with HLB value in the range 9-12 are optimal⁵ and

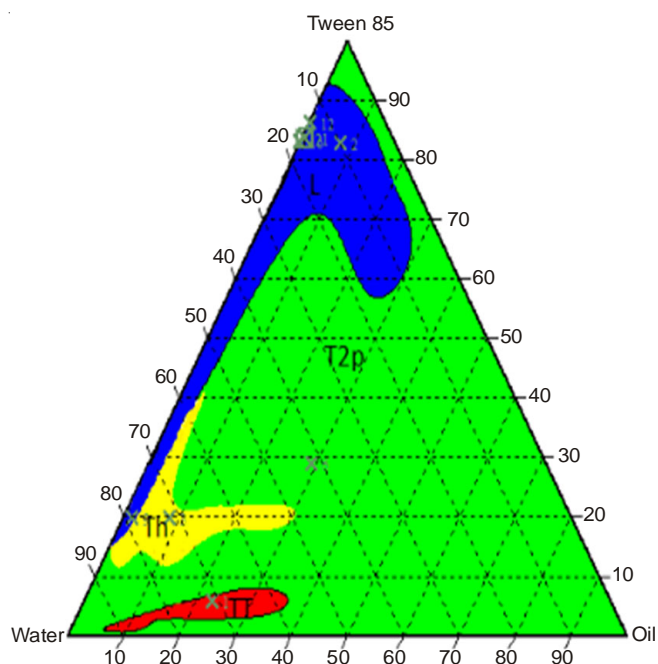


Fig. 6. Ternary phase diagram of Tween85/pitaya seed oil: Jojoba oil/ de-ionized water. (Th – Homogenous region; L – Isotropic region; T2p – Two phase region; TT – Three phase region)

suitable for emulsification. Both surfactants were suitable surfactants and formed good results which have HLB numbers (10-15), however Tween85 produced a better result and works well for all three types of oil by forming two single phases which is homogenous and isotropic phase.

Conclusion

Pitaya seed oil was successfully extracted using *n*-hexane. In this research, six ternary phase diagrams of pitaya seed oil:

jojoba oil with non-ionic surfactants was successfully constructed. The results showed that the Tween85 gave better solubility in water to produce larger isotropic and homogeneous regions for jojoba oil, pitaya seed oil and pitaya seed oil: jojoba oil. The presence of optimal HLB value of Tween85 for the stabilization of o/w emulsions contributes to the enlargement of the single phase regions.

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