Another Multiparametric Way In Planning Of Experiments For O/W Emulsion Design

NT_621

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Introduction:

Making appropriate choices to optimize the stability of a particular emulsion represents a challenge with mid-to-long-term consequences. It is therefore of paramount importance.

Main parameters having an impact on cosmetic emulsions :

- Raw material choice: nature & origin (Surfactants, oils, etc.)
- Production conditions of emulsions (physicochemical properties)
- External parameters (packaging, storage conditions, etc.)

[1]
- Aqueous phase (W)
- Fatty phase (O)
- Surfactants (S)

Different methods exist to better understand (if not optimize) the emulsions, but with associated constraints [2]:

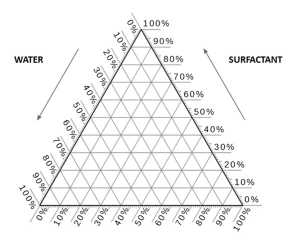
Existing methods:
- Multidimensional analysis based on dichotomic approach ➔ Trial and error approach
- Design of Experiments (DOE) ➔ Need of specific software

Quick and easy screening methods [3 – 5]:
- ***Fact Screening Method (FSM)*** **completed with Principle Component Analysis (PCA)**

These methods can be applied for preliminary systemic studies such as HLD-WOR, Winsor, etc [6 – 7].

Materials & Methods:

The Fast-Screening Method (FSM) uses an evenly distributed scanning of the raw material proportions (composing the three SOW phases) to easily define stability zones.



5% : minimum difference between two distinct phase proportions
➔ 171 combinations of S/O/W
➔ Values ∈ [5% ; 95%] for each phase

Materials

Raw material category	INCI name
Water	Demineralized Water
Gelling agent	Carbomer
Oil	Isopropyl palmitate (IPP)
	Sunflower Seed Oil (SO)
Surfactant	Polysorbate 80 (P80)
	Oleth-2
	Glyceryl stearate (GMS)

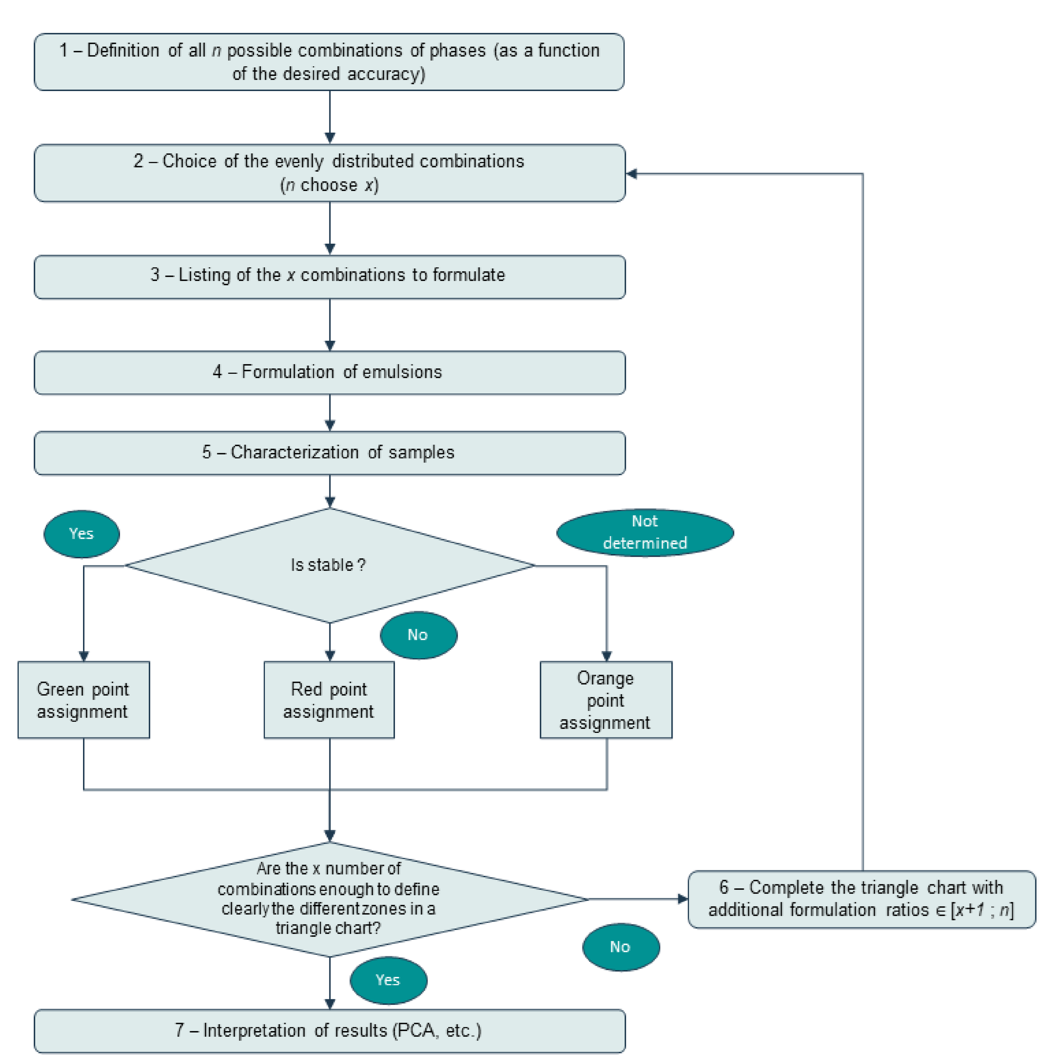
4 sets of composition formulated:

A: P80 + GMS / IPP / Water + Carbomer
B: P80 + Oleth-2 / IPP / Water + Carbomer
C: P80 + GMS / SO / Water + Carbomer
D: P80 + Oleth-2 / SO / Water + Carbomer

Protocol

Both surfactants and aqueous phase are heated up to 75°C before being added to the oil at room temperature. This mixing is emulsified with a vortex 1 minute. Once back at room temperature, the pH is then adjusted at 5.5, by adding 10% triethanolamine (TEA) solution (TEA/Water = 2.26) to thicken the emulsion thanks to the carbomer. Each sample is stored at 25°C for 24 ± 2 hours before the organoleptic analyses.
➔ Organoleptic study made on a 10 g test tube.

Fast Screening Method (FSM)



Example with a 20% minimum difference

Step 1: generation of the combinations with a program

Step 2: assignment of the pseudo random numbers with a spreadsheet

Step 3: ranking of the pseudo random numbers

Step 4: list of the x first combinations to formulate (n choose x)

Combination number	Phase 1	Phase 2	Phase 3	Generation of pseudo random numbers	Pseudo random numbers associated rank
1	20	20	60	0.0020	1
2	20	40	40	0.5160	4
3	20	60	20	0.0998	2
4	40	20	40	0.5740	5
5	40	40	20	0.2850	3
6	60	20	20	0.8330	6

For only 3 combination formulations, the 3 first rank appearing are chosen:

Combination 1 : 20/20/60
Combination 4: 40/20/40
Combination 2: 20/40/40

In this example, n = 6

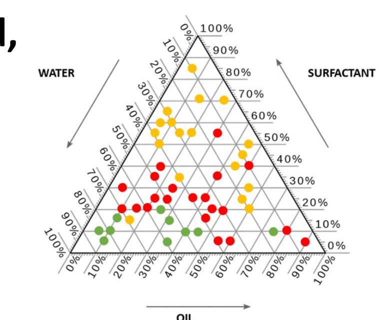
In this example, x = 3

Results & Discussion:

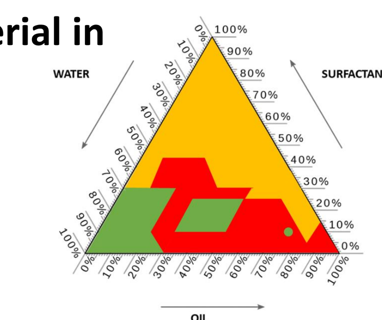
50 samples per set are achieved: 49 trials of formulation and a 50th one randomly duplicated to verify the reproducibility of the process. The triangle chart aims to easily detect the stability zones (green for stable O/W emulsions, red for unstable emulsions (several phase behavior) and orange for stable emulsions except O/W), for emulsified and non-emulsified mixing. The PCA shows the predominant parameters for the emulsions including the optimal cream parameters for a O/W cream (white; creamy; opaque; without granules).

Set of composition A

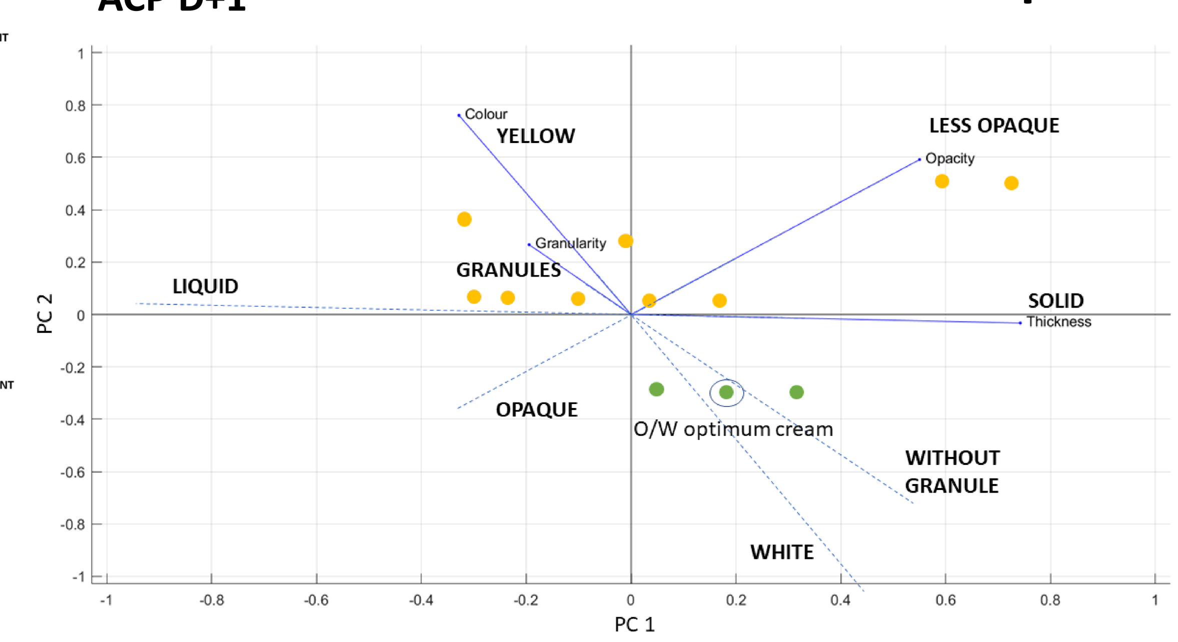
Emulsified, D+1



Raw material in contact, D+180



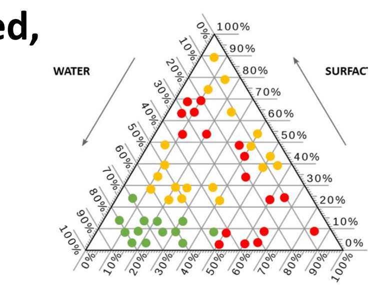
ACP D+1



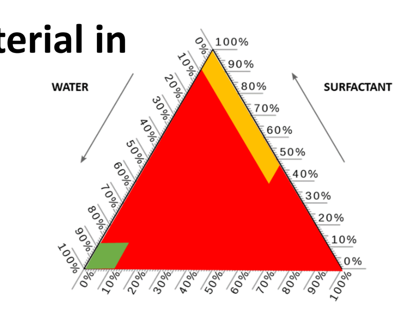
- Position of the O/W optimum emulsion ➔ driven by surfactant quantity
- The opacity descriptor ➔ linked with the ratio Surfactant/Aqueous phase
- The granularity ➔ depends on the oil quantity (a lack of oil = aggregation of the surfactant)

Set of composition B

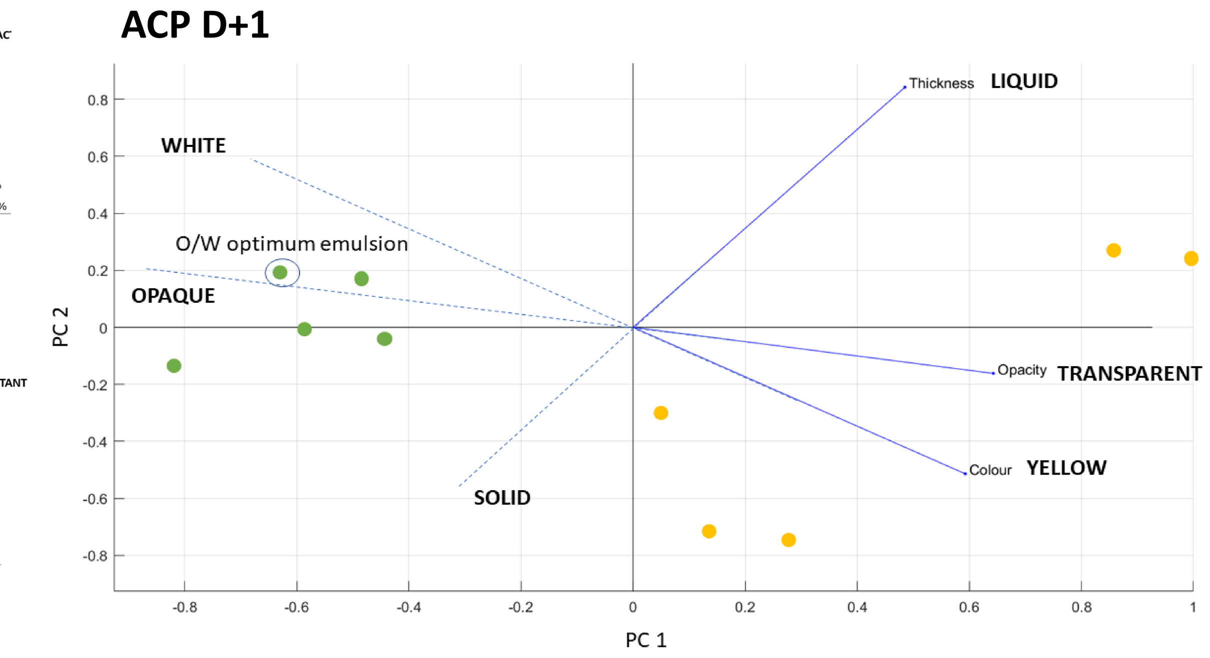
Emulsified, D+1



Raw material in contact, D+180



ACP D+1



The change of the solid surfactant for a liquid one allows to maintain a smooth texture without granularity. The behavior of the emulsion and the zone of stability are still comparable through time.

The change of oil from a chemical one to a natural one highlights a better definition of the different zones. However, the sets of composition C and D contain sunflower oil with a complex fatty acid mix, leading to at least two stability zones, separated with an unstable one; this can have an impact during the scale up.

One can see that the observations after a long duration without emulsification are comparable with the emulsified ones thanks to our FSM.
The method focuses on organoleptic properties to show its easy implementation BUT can be completed with other characterization technics.

Conclusions:

Opacity, thickness and granularity ➔ Most relevant descriptors to outline the emulsion stability state.

FSM:
- Easy methodology using pseudo-random screening uniformly distributed and PCA
- Screening quickly the map of formulation before further analysis

➔

Allowing a fast preliminary determination of the stability zone to develop new emulsion formulations

Determining preventively the zone of interest for an accurate longer-term study

References:

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