Experimental design – approach for the formulations of oral care products containing *Vitis vinifera L.* extracts

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Background

▪The development of a cosmetic product using an experimental design represents a systematic approach that allows to set the best ranges for the formulation factors with a minimal number of experimental runs.

▪ This new approach in the field of cosmetic formulation provides novel inp formulators in order to choose the appropriate ingredients in an accurate rat obtain the product to better-fit to the consumers expectancy.

Aim

▪ The study investigate the use of experimental design to study the impact of the formulation factors on the final product performance in order to obtain toothpastes and mouthwashes with optimal characteristics

Material and methods

▪ Two factorial experimental design with three factors and two levels (Modde 12.1 software, Sartorius Stedim Data Analytics AB, Umea, Sweden) were developed.

A - Toothpaste formulation
→ 14 experimental runs

Variable inputs:
- % silicon dioxide– X1 (15-25%)
- % xanthan gum– X2 (0,5-1,5%)
- % sodium carboxymethylcellulose– X3 (0-1%)

Dependent variables :
- Firmness - Y1
- Consistency -Y2
- Rigidity -Y3
- Adhesive force -Y4
- Cohesiveness -Y5
- Stringiness length -Y6
- Stringiness work -Y7
- Viscosity Y8 – Brookfield cone-plate CAP2000 Viscometer

CT3 Texture Analyzer (Brookfield Engineering Laboratories)

B- Mouthwash formulation
→ 11 experimental runs

Variable inputs:
- % xanthan gum– X1 (0-0,05%)
- % ethanol– X2 (0-20%)
- stirring rate during the preparation process– X3 (100-1100 rpm)

Dependent variables :
- Consistency - Y1
- Firmness - Y2
- Cohesiveness - Y3
- Resilience - Y4
- Viscosity measurements -Y5- Brookfield DV-III Ultra LV

- Quality-of-fit assessment, coefficient calculation and statistic parameters evaluation were performed with Modde 12.1 software.

Results and discussion

A - Toothpaste formulation

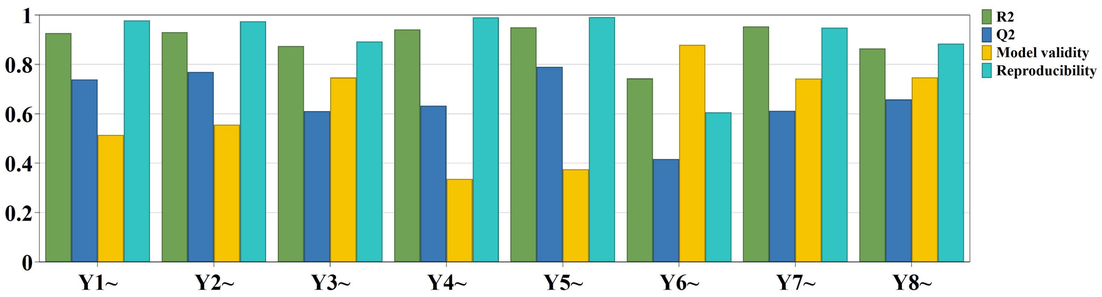

Figure 1. Summary of fit- toothpaste formulation

▪ Firmness (Y1) values ranged from 37.70 g to 1059.00 g and Consistency (Y2) values ranged from 5.22 mJ to 152.10 mJ. The values of the firmness and consistency of the toothpaste depend mainly on the variation of the percentage of powders and thickening agents. In addition, the content of powders influences the level of abrasiveness and the extrudability the cosmetic product. Thus, it was observed that an increase in the percentage of silicon dioxide (X1) and sodium carboxymethylcellulose (X3) caused an increase of the values of consistency and firmness.

▪High ratios of silicon dioxide (X1) and sodium carboxymethylcellulose (X3) led to an increase of the Rigidity values (Y3) between 10.50 g and 32.20 g, the values of the Adhesive force (Y4) between 15.00 g and 807.70 g and the Cohesiveness values (Y5) between 3.43 mJ and 118.50 mJ. These parameters give information about the structural deformation of the toothpaste and the behavior during the extrusion process.

▪ Stringiness length (Y6) was negatively influenced by the increasing ratio of silicon dioxide (X1) and also by the combination X1-X2.

▪ Stringiness work (Y7) was positively influenced by the increase of the concentration of these two compounds, but the combination of silicon dioxide with xanthan gum(X2) or sodium carboxymethylcellulose led to the decreasing of this parameter.

▪The values recorded for viscosity ranged from 345.67 to 1753.33 P. Thus, the viscosity was positively influenced by the powders ratio and the thickening agents increase.

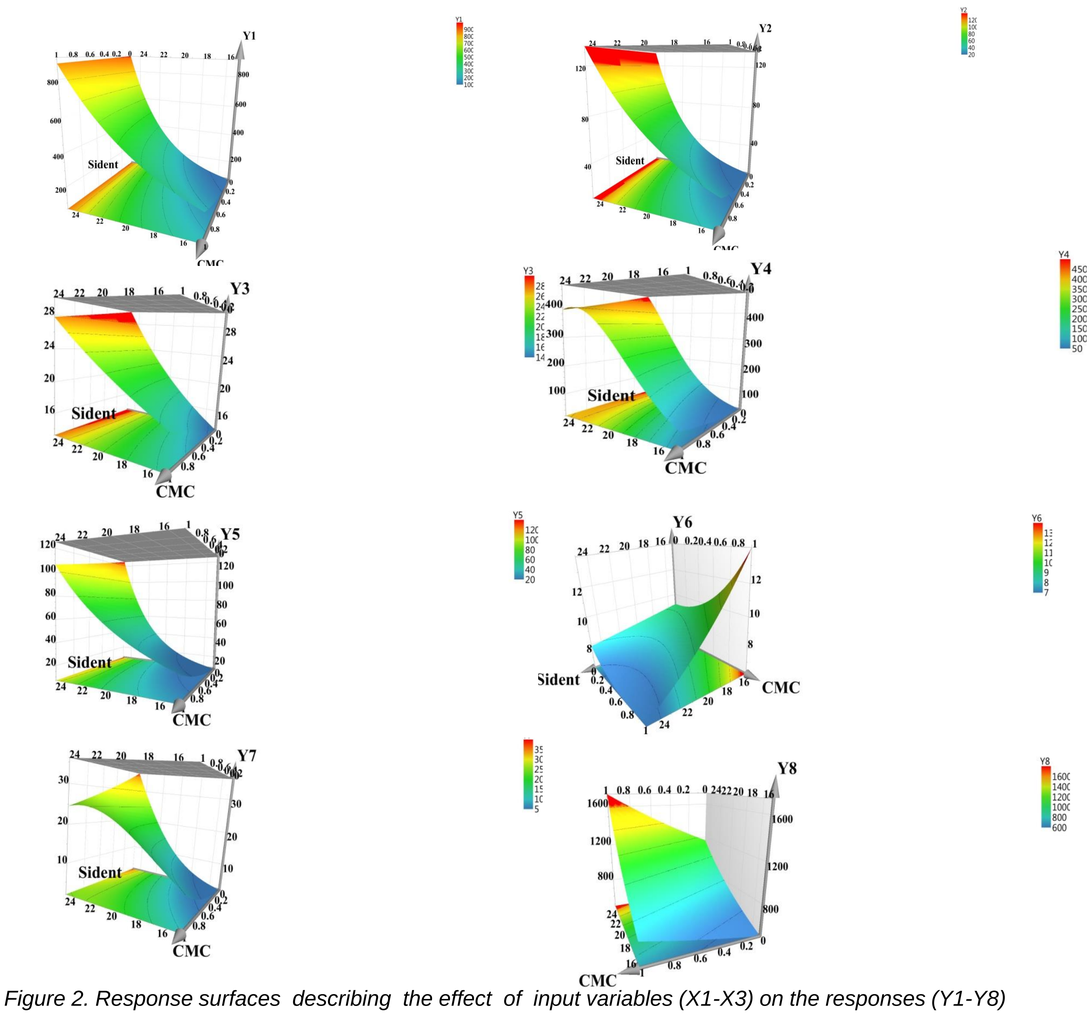

Figure 2. Response surfaces describing the effect of input variables (X1-X3) on the responses (Y1-Y8)

B – Mouthwash formulation

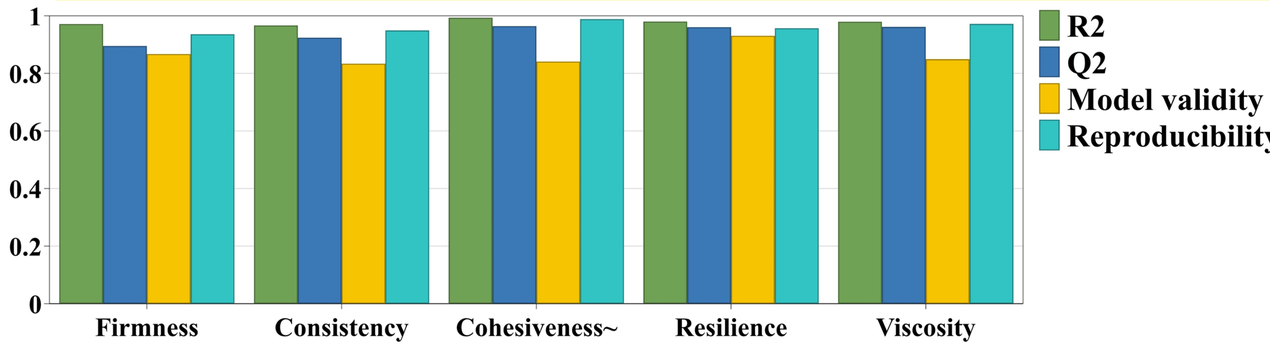

Figure 3. Summary of fit- mouthwash formulation

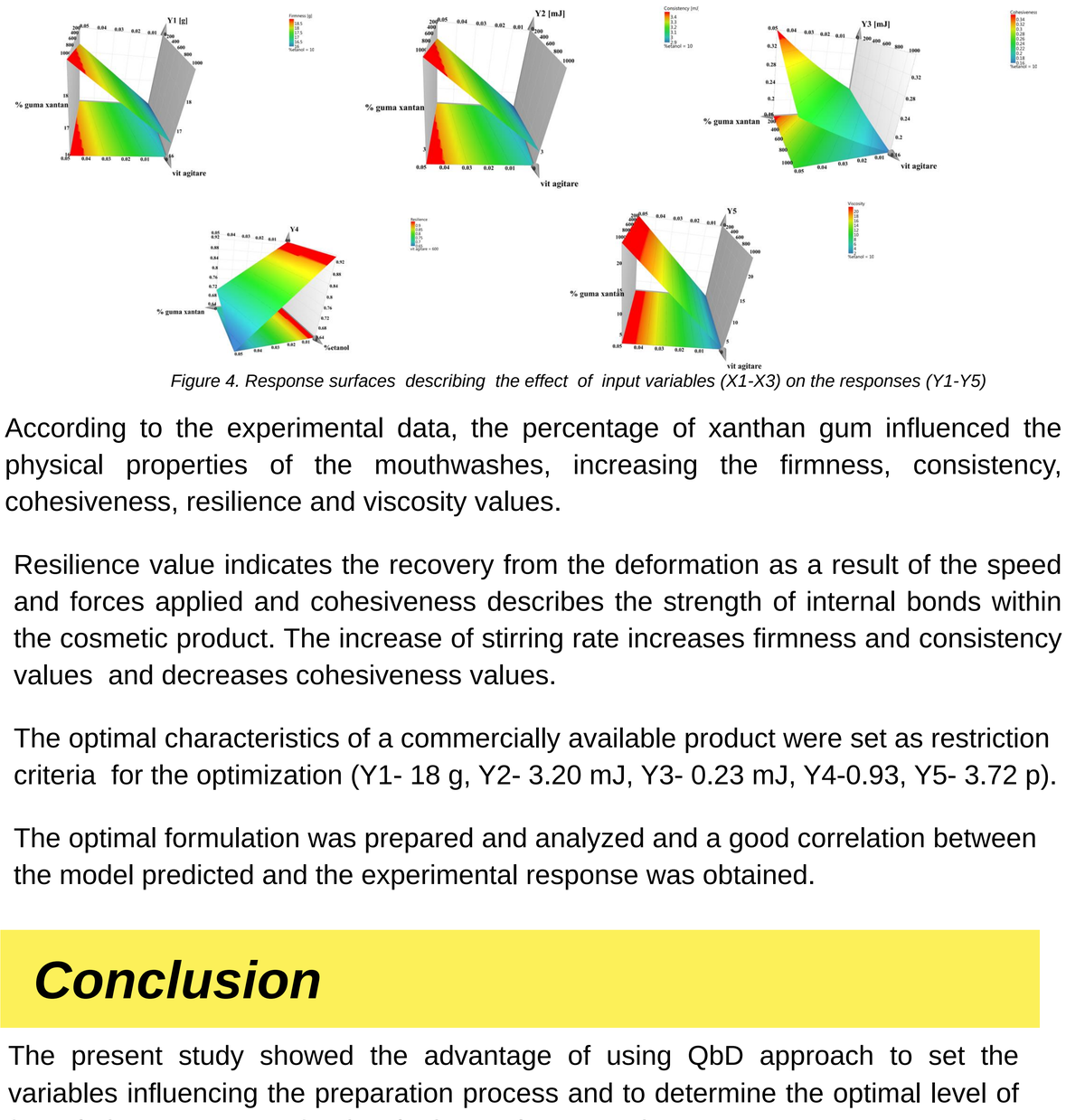

Figure 4. Response surfaces describing the effect of input variables (X1-X3) on the responses (Y1-Y5)

- According to the experimental data, the percentage of xanthan gum influenced the physical properties of the mouthwashes, increasing the firmness, consistency, cohesiveness, resilience and viscosity values.

- Resilience value indicates the recovery from the deformation as a result of the speed and forces applied and cohesiveness describes the strength of internal bonds within the cosmetic product. The increase of stirring rate increases firmness and consistency values and decreases cohesiveness values.

- The optimal characteristics of a commercially available product were set as restriction criteria for the optimization (Y1- 18 g, Y2- 3.20 mJ, Y3- 0.23 mJ, Y4-0.93, Y5- 3.72 p).

- The optimal formulation was prepared and analyzed and a good correlation between the model predicted and the experimental response was obtained.

Conclusion

The present study showed the advantage of using QbD approach to set the variables influencing the preparation process and to determine the optimal level of formulation parameters for developing oral care products.

Acknowledgements

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