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HYDROGELS APPLIED IN COSMETOLOGY IRRADIATED BY IONIZING RADIATION

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ABSTRACT

Hydrogels are three-dimensionally crosslinked polymers that exhibit high capacity to absorb water or solvents, without compromising its structure, allowing its application in cosmetic products, because it presents easy scattering and vehicular active principles. The use of ionizing radiation to obtain the hydrogels provides the absence of chemical initiators; sterilization; reticulation and adjustment of physical-chemical properties. In this work different types of hydrogels containing PVP concentrations 5%, 7.5% and 10% weight and different radiation 25 and 20 kGy doses were prepared, maintaining PEG concentrations 3% weight and agar 1% weight, based on literature studies. The samples were characterized by dehydration as a function of time, acidity, visual and sensorial analyzes and stability. The results obtained showed that all the compositions are stable, have a pH close to the skin and the compositions containing PVP 5% weight, obtained with radiation of 20 and 25 kGy dose undergo greater dehydration. In sensory research, the hydrogels containing PVP 7.5% weight, obtained with a radiation of 25 kGy dose, presented the best results in terms of absorption, sliding, odor, while the composition containing PVP 10% weight obtained with a radiation of 25 kGy dose, proved to be inadequate in the public perception. Therefore, the hydrogels obtained with PVP 7.5% weight, with radiation of 25 kGy dose were the most suitable for applications in cosmetic products.

1. INTRODUCTION

Anti-aging cosmetics contain active principles to suit the physiological and anatomical conditions of the skin in its individual variations. The care with its functional and anatomical integrity allows to intervene effectively with the objective of the esthetic improvement of the skin [1]. In this context, polymeric hydrogels assume a primordial role attributing their distinct characteristics to the functions of the formulated products, since they have swelling / relaxation characteristics of their structural chains, being great choices for formulations containing hydrophilic actives, and some have characteristics of film formation, making choice for topical formulations [2]. Thus, hydrogels based on poly N-vinyl-2-pyrrolidone-PVP, which are structured polymeric materials formed by three-dimensional polymeric nets are wholly or partly hydrophilic. These are distinguished by absorbing large amounts of water or biological fluid inside them without dissolving [3].

The PVP-based hydrogel is widely used as biomaterial because it does not exhibit toxicity, in addition, it has a high swelling property [4]. The high hydrophilicity and insolubility characterizing the hydrogels are explained by the presence of hydrophilic functional groups, and due to the interlacing and cross-linking of their chains, respectively. When swollen, the properties of the hydrogels associate with those of the living organism. In addition, the physical appearance (softness and elasticity) reduces possible reactions with cells that could cause internal inflammation [5]. In order to provide greater flexibility, greater extensibility, and ease of handling of the hydrogel, polyethylene glycol-PEG, which acts as a plasticizer in PVP-based hydrogels, is used to alter the viscosity of the system and increase the mobility of the macromolecules [6,7].

In the hydrogels based on PVP, obtained by ionizing radiation, the presence of the agar in small concentrations favors the gelation of the solution, providing them physical form before crosslinking, making the irradiation process viable [8]. Ionizing radiation is employed in the preparation of the hydrogel for the purpose of promoting crosslinking, in addition to conferring sterility. The PVP hydrogel obtained through ionizing radiation is suitable for use as a polymer matrix to form a controlled drug delivery system [4].

Considering these considerations, this work aims to contribute to the cosmetics area by presenting hydrogels containing 5%, 7.5% and 10% by mass of PVP, submitted to doses of 25 and 20 kGy by ionizing radiation for sterilization, maintaining the concentration of 3% by mass of PEG and 1% by mass of agar.

2. MATERIALS AND METHODS

Different types of hydrogels were prepared containing different concentrations of PVP (10%, 7.5%, 5% in weight) and at the radiation doses of 25 and 20 kGy, maintaining the PEG concentrations at 3% by mass and 1% based on literature studies. The Table 1 presents the compositions of the base hydrogels. These hydrogels were produced in order to define the most suitable composition for the final product [9].

Table 1: Compositions of the hydrogels.

Hydrogel	Radiation (kGy)	Composition (% in weight)		
		PVP	PEG	Ágar
HB1	25	10	3	1
HB2	25	7,5	3	1
HB3	25	5	3	1
HB4	20	5	3	1

The concentrations were based on data from the literature and preliminary studies, which were important for the definition of the most adequate basis for this application. To obtain the hydrogels, the reactants were pre-dissolved in water, and mixed hot. The concentration of the components in the final solution was adjusted by addition of water in sufficient quantity to reach 100% by mass (Fig. 1).

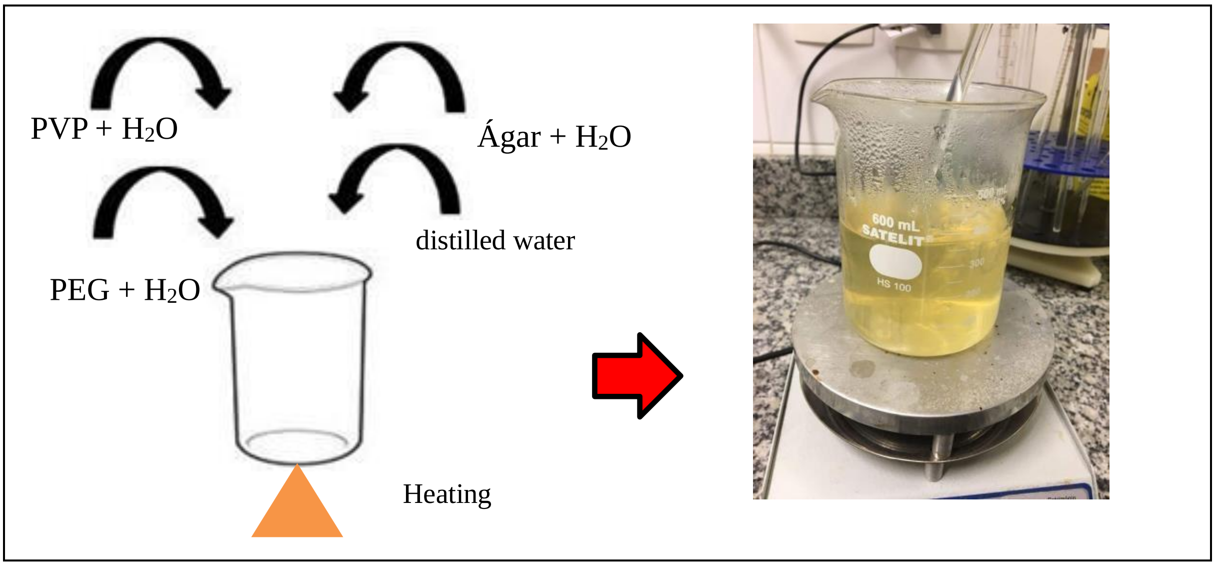

Figure 1: Preparation of hydrogels

The hydrogels, with a thickness of 3 mm, were obtained by pouring the hot solution into a sample holder, which, after cooling, was packed and sealed with polyethylene film (thickness of approximately 0.1 mm) for the hydrogel remain sterilized, as recommended for dressings used directly on the skin [10,11].

After the samples were prepared, they were irradiated to promote crosslinking between the chains. The samples were irradiated at room temperature in an electrostatic type electron accelerator of the model Dynamitron (Fig. 2) with maximum energy of 1.5 MeV, maximum current of 15 mA and dose rate of 11.3 kGy/s.

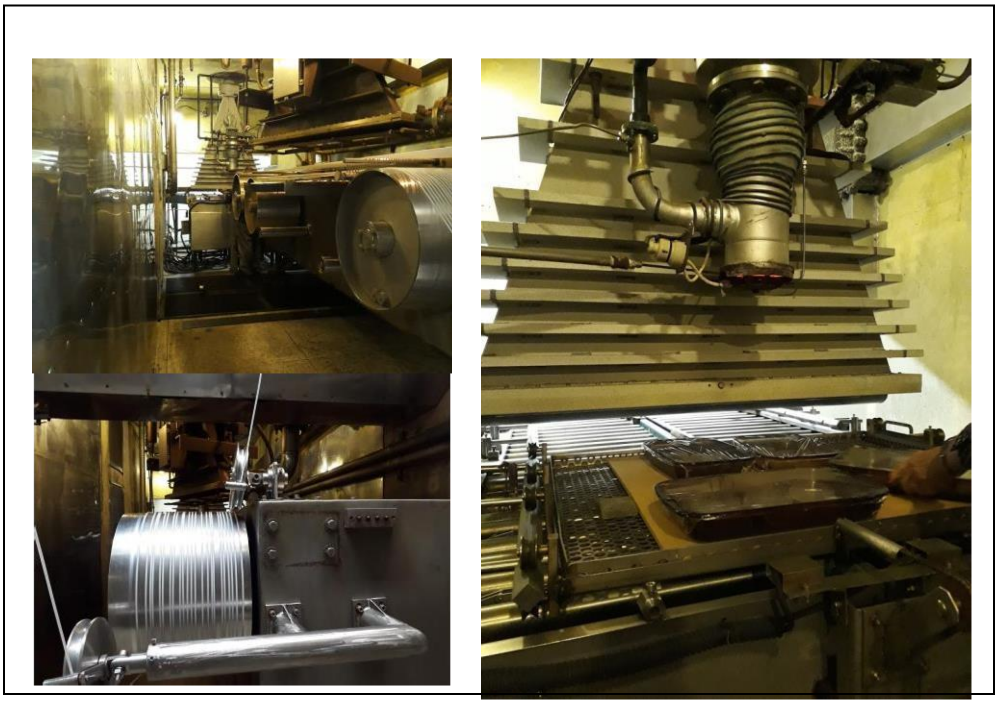

Figure 2: Equipment Radiation Dynamics, model Dynamitron.

The samples were irradiated in doses of 20 and 25 kGy.

2.1 Characterization of the hydrogels

The characterization of the hydrogels was performed following the following parameters: visual characterization, sensorial analysis, pH value, density and dehydration as a function of time.

- **Sensory analysis:** Sensory analysis was performed with a group of 100 students from the Cosmetology and Aesthetics Technology Course of the SENAC University Center by probabilistic method, not for convenience.

The participants of the research, after 10 minutes of application of the product in indicated area (back of the hand) answered the research instrument. Directly, using the Likert scale, the instrument proposes to the research participant indicators that evaluate the product according to the issues: absorption; slipping; aspect; odor and feeling of comfort after application.

As mentioned, the research used the Likert scale being: number 5 (five) associated with full satisfaction; and number 1 (one) associated with full dissatisfaction.

The great advantage of the Likert scale is the ease of the research participant to issue a degree of agreement on a given statement. Additionally, the confirmation of psychometric consistency in the metrics that used this scale contributed positively to its application in the most diverse researches [12].

To compute the results of the evaluation process it was considered that:

- Answers marked as: Satisfied fully and Satisfied partially, indicate the approval of the evaluated indicator;

- Answers marked as: Completely unsatisfied and partially unsatisfied, indicate disapproval as to the evaluated indicator;
- Answers marked as - Not satisfied or dissatisfied, indicate that there is no opinion as to the indicator evaluated.

The results presented range from 0% (meaning maximum reprobation) to 100% (meaning maximum approval).

- **pH:** The pH of the samples was determined using the Hanna peagometer, model HI-98128. Immediately after heating to obtain the physical gel and subsequently at room temperature.

- **Density:** The mass of each formulation was used in relation to the total volume of the obtained solution of 50 mL (D = m/v) at room temperature.

- **Dehydration as a function of time:** Samples were kept in their original containers at room temperature. The masses of the samples used in the experiments were of the order of 14 g. To obtain the percentage of dehydration the following relation was used:

$$\%D = [(W_i - W_f) / W_i] \cdot 100\% \quad . \qquad (1)$$

Where:% D = percentage of dehydration, Wi = sample initial mass and Wf = final sample mass.

3. RESULTS AND DISCUSSION

3.1 Visual Characterization

The Figure 3 shows the appearance of the hydrogels obtained.

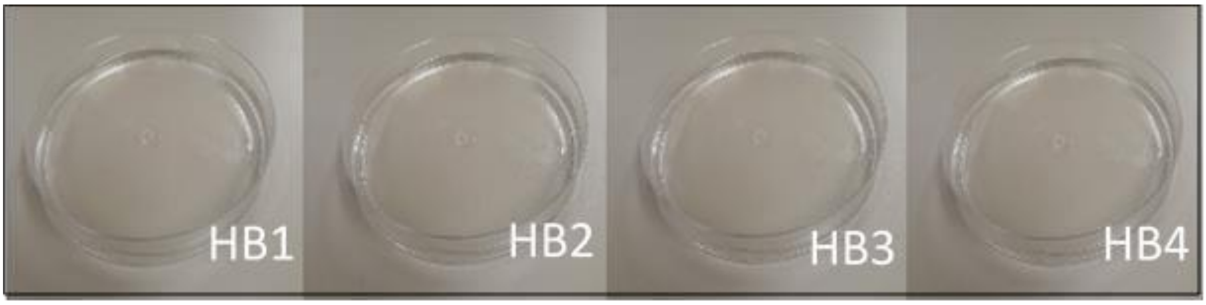

Figure 3: Base hydrogels obtained: HB1 (10%PVP-25 kGy); HB2 (7,5%PVP-25 kGy); HB3 (5%PVP-25 kGy); HB4 (5%PVP-20 kGy)

PVP is widespread in pharmaceutical technology in a classical approach in terms of increasing dissolution of poorly water-soluble drugs is in maximizing the porous structure of a water-soluble polymer matrix [13]. Therefore, this feature is also fundamental for cosmetics. The agar concentration used in this work favors the gelling effect and enables the irradiation and sterilization process [14]. The plasticizing action of PEG in the samples is identified at the concentration used.

- The hydrogels obtained were observed to have a viscous appearance;
- Hydrogel formulations showed a prevalently elastic rheological behavior, as recommended in the literature [15]
- All samples were observed of same aspect and samples HB3 (5% PVP-25 kGy) and HB4 (5% PVP-20 kGy) had a consistency not suitable for the final cosmetic product.

3.2 Sensory analysis

The degree of satisfaction is given by the sum of the partially satisfied and fully satisfied. The Figures 4 to 8 show the results obtained for the indicators of the degree of satisfaction in the sensorial analysis.

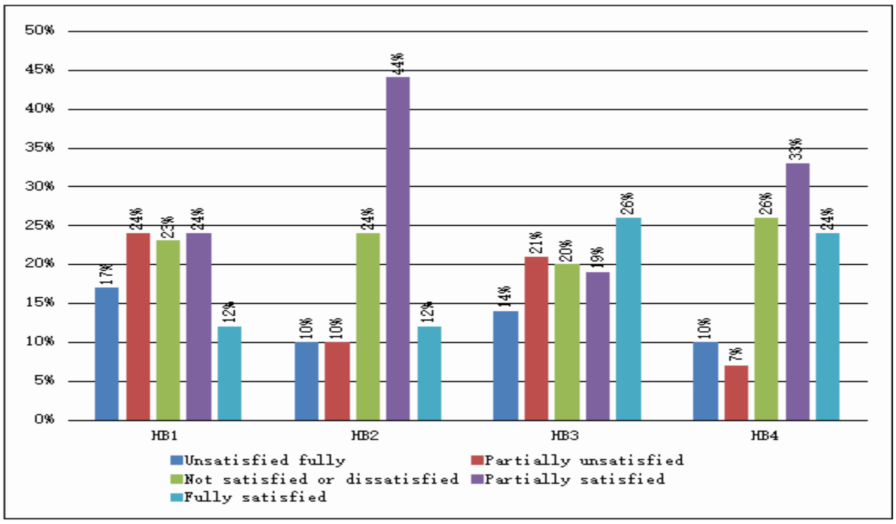

Figure 4: Degree of satisfaction for the absorption of the hydrogels

Regarding the degree of satisfaction for the absorption of the hydrogels, it was possible to observe that:

- HB1 hydrogels (10% PVP-25 kGy) presented the lowest degree of satisfaction with 36% approval;
- HB4 hydrogels (5% PVP-20 kGy) had the highest degree of satisfaction with 57%;
- Samples HB2 (7.5% PVP-25 kGy) and HB4 (5% PVP-20 kGy) had the highest satisfaction rates 56% and 57%, respectively.

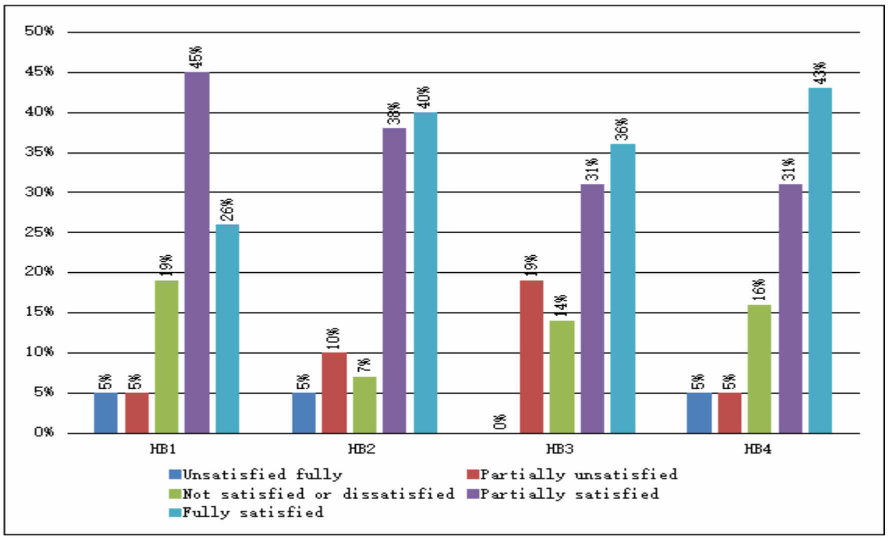

Figure 5: Degree of satisfaction for the slippage of the base hydrogels

Regarding the degree of satisfaction for the slippage of the base hydrogels, it was possible to observe that:

- All hydrogels presented satisfactory results, with HB1 (10% PVP-25 kGy) and HB2 (7.5% PVP-25 kGy) having the highest satisfaction levels with 71% and 78% of approval, respectively;
- The hydrogel with the lowest degree of satisfaction was HB3 (5% PVP-25 kGy).

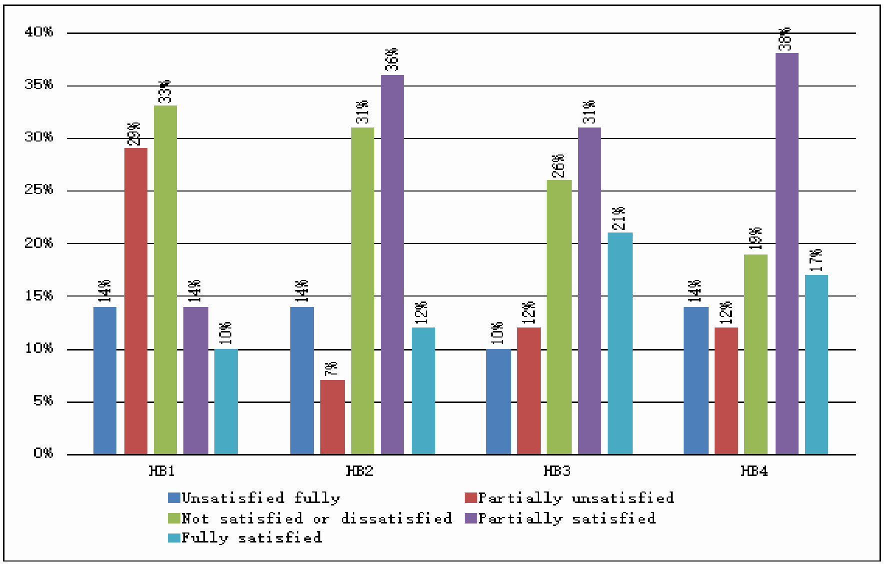

Figure 6: Degree of satisfaction for the aspect of the base hydrogels

Regarding the degree of satisfaction for the aspect of the base hydrogels, it was possible to observe that:

- HB4 hydrogels (5% PVP-20 kGy) were considered to have a better appearance with 55% approval;
- HB1 hydrogels (10% PVP-25 kGy) presented the lowest degree of satisfaction with 43% failure.

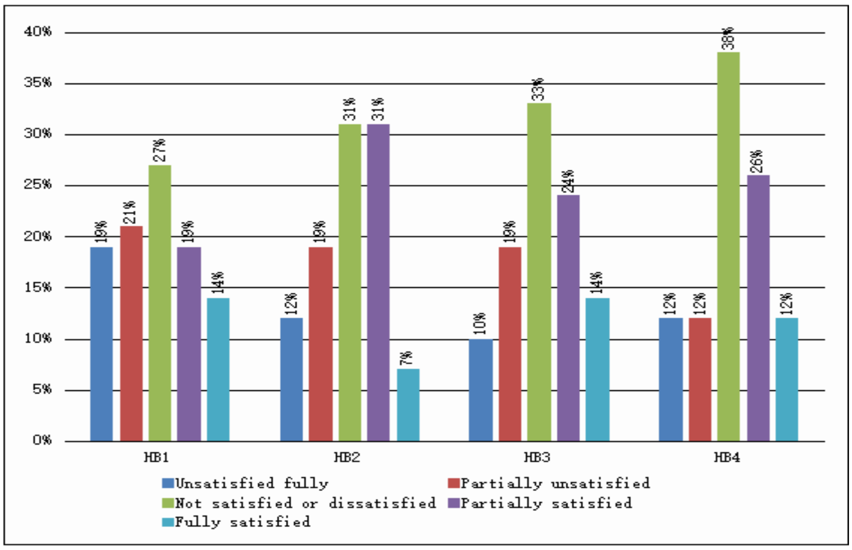

Figure 7: Degree of satisfaction for the odor of the base hydrogels

Regarding the degree of satisfaction for the odor of the base hydrogels, it was possible to observe that:

- The samples presented close results, since the odor they had is characteristic of PVP, agar and PEG.

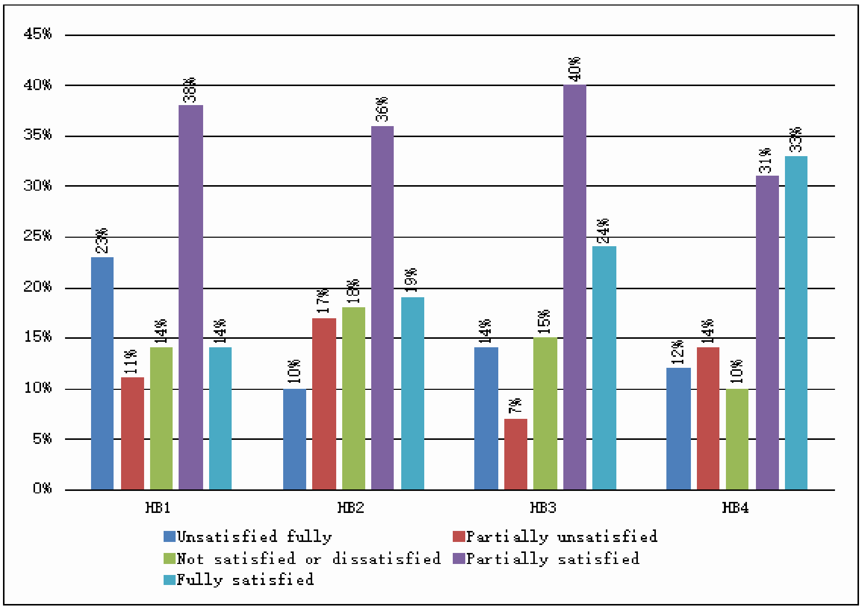

Figure 8: Degree of satisfaction for the feeling of comfort after application

All samples generated a feeling of comfort after 10 minutes of application.

According to the results obtained, the composition HB3 and HB4 were as the most adequate to obtain the desired product, with 64% of degree of satisfaction.

Hydrophilic gels have been widely used in cosmetic products and as a dermatological base because they are easy to spread, are not greasy and can carry water-soluble active ingredients and liposomes [16].

In this type of treatment the degree of patient / user satisfaction is important for the continuity of treatment [17].

According to the results obtained in the sensory analysis, the degree of satisfaction was met.

For each type of sensory analysis, a score was assigned according to the degrees of satisfaction. Table 2 presents the assigned scores for each hydrogel.

Table 2: Degree of satisfaction obtained for the dimensions evaluated

Hydrogel	Degree of satisfaction of hydrogels (%)					
	Absorption	Slippage	Aspect	Odor	Comfort	Total
HB1	36	71	24	33	52	216
HB2	56	78	48	38	55	275
HB3	45	67	52	38	64	266
HB4	57	74	55	38	64	288

By means of the obtained results, it is observed that by the sensorial analysis, the hydrogels HB2 and HB4 are the most indicated. Because the HB2 hydrogel has been obtained with a radiation dose of 25 kGy, in which effective sterilization takes place, this is the most suitable for obtaining the cosmetic product.

3.3 pH

The pH is a factor that influences the strength of the gel, because the lower the pH, the lower the strength of the agar gel [18]. The Figure 9 shows the pH obtained for the different hydrogels studied.

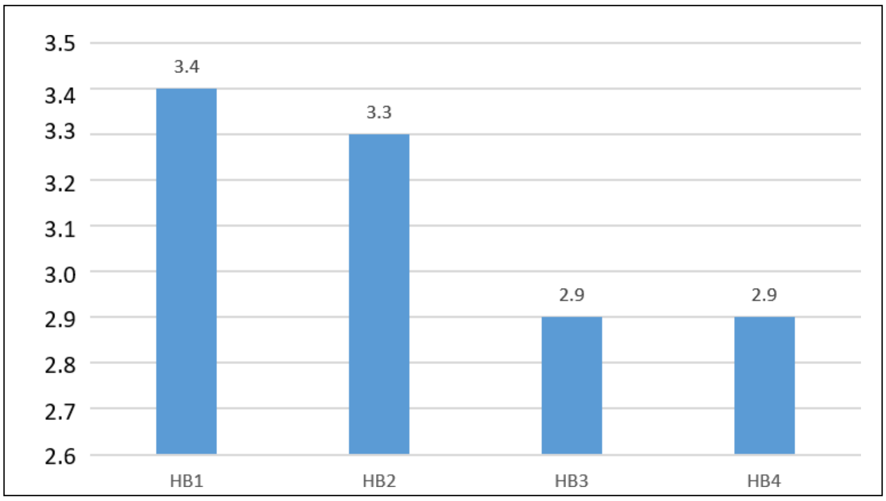

Figure 9: Results of the pH for different hydrogels.

By means of the obtained results it is observed that

- All hydrogels obtained have acid pH in the range of 2.9 to 3.4;
- The pH presented by the hydrogels decreases with the decrease of PVP;
- Comparing HB3 (5% PVP-25 kGy) with HB4 (5% PVP -20 kGy), it is observed that the radiation does not interfere with the pH of the obtained hydrogel.

The determination and control of the pH of the hydrogels, from a cosmetic and / or dermatological point of view, is extremely useful. The skin presents a slightly acid pH, which contributes to bactericidal and fungicide protection on its surface [19].

3.4 Density

Gamma rays are electromagnetic radiation, with basic absorption properties represented by the exponential decrease in radiation intensities that, through the matter, provide high penetration power, effectively used in the industrial processing of materials. In all types of ionizing radiation, penetration is inversely proportional to the density of the processed materials. Thus, product density and package dimensions are key factors in lightning applications [20].

As shown in Figure 10, comparing the density of the HB1, HB2, HB3 and HB4 hydrogels, the values obtained were slightly less than 1.00 g.cm^{-3}, close to the water density. The density of the hydrogels remained stable, very close, even with variations in the concentration of PVP (5%, 7.5% and 10%), and with different doses of radiation (20 and 25 kGy).

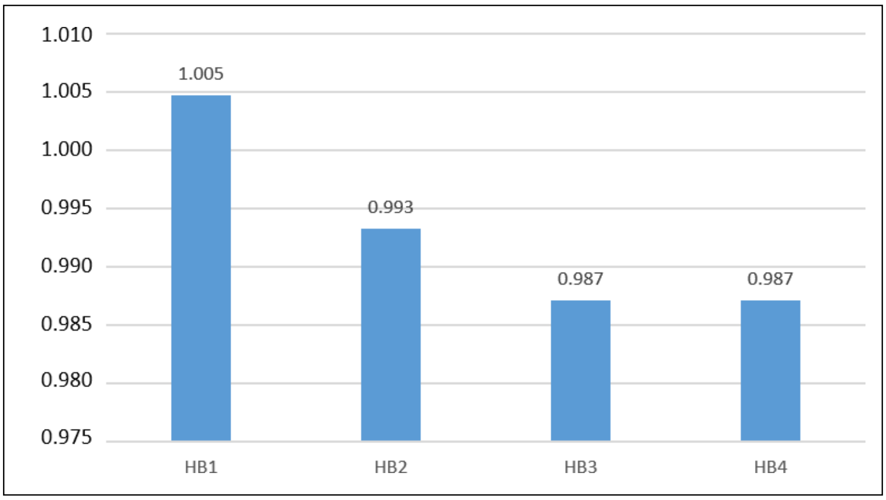

Figure 10: Results of the density for different hydrogels.

3.5 Dehydration versus time:

A cosmetic to be applied to the skin, whether in the form of a cream or any other form, capable of attaching itself to water, retaining it on its surface. In this way, it is convenient to evaluate the water retention power exhibited by the formulations and one of the embodiments of the mass loss determination attributed to the water evaporation of the formula [21].

Dehydration versus time was monitored for all formulations and Table 3 and Figures 11 and 12 show the variation of the percentage of dehydration as a function of time after irradiation.

Table 3: Variation of the percentage of dehydration over time after irradiation

% de Dehydration		Time (days)						
		30	60	90	120	150	180	210
	HB1	8.77	15.78	23.36	34.01	39.70	45.09	52.69
	HB2	11.26	19.96	31.77	54.26	54.84	58.10	62.91
	HB3	12.29	26.59	40.76	58.53	66.24	74.35	79.51
	HB4	1.01	17.41	29.10	40.96	50.79	61.44	67.15

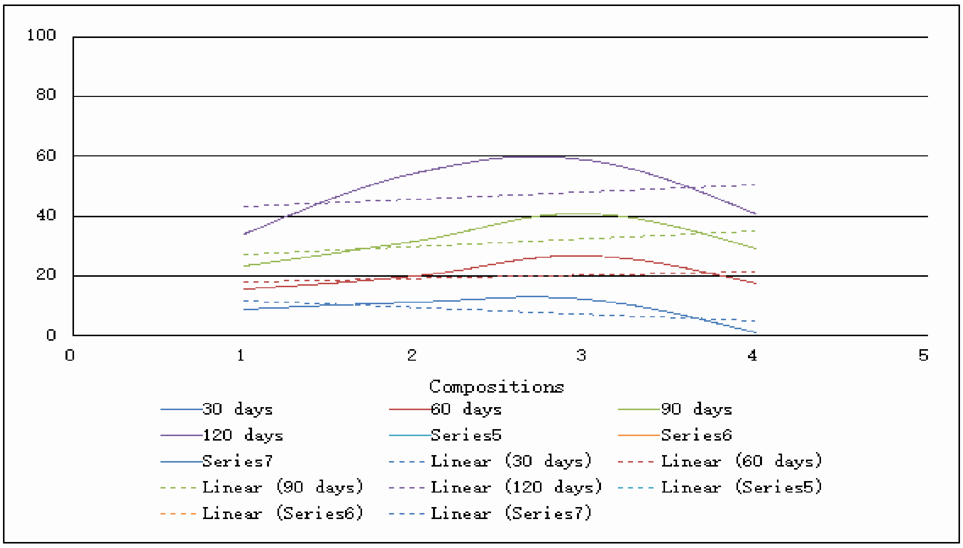

Figure 11: Variation of dehydration percentage as a function of composition

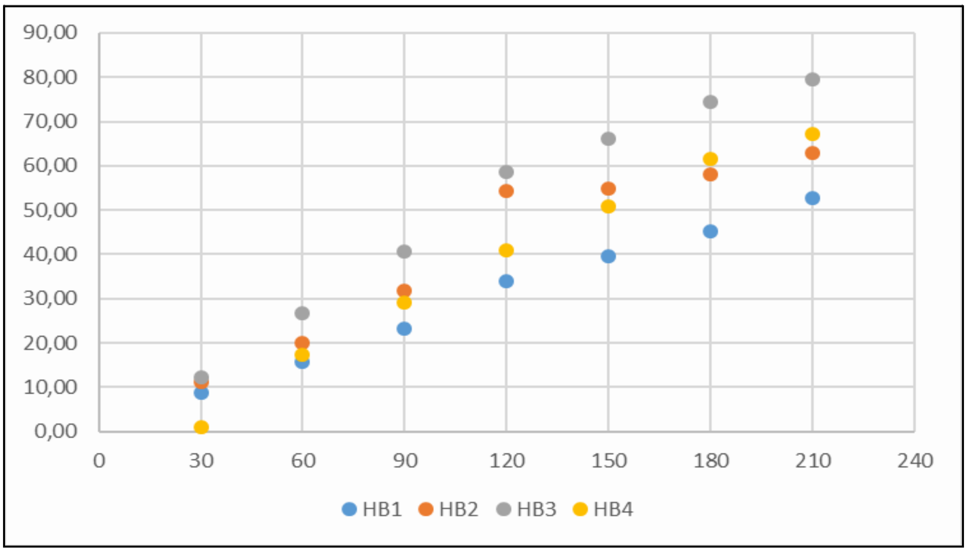

Figure 12: Variation of the percentage of dehydration as a function of time after irradiation

By means of the obtained results it can be observed that:

- Dehydration has increased over time for all hydrogels obtained;
- The lower the PVP concentration, the greater the dehydration of the obtained hydrogels. Probably, the crosslinks formed as a result of ionizing radiation are more spaced, the lower the concentration of PVP in the hydrogel.
- The hydrogels with 5% by weight of PVP were the ones that presented the highest degree of dehydration during the studied period.

4. CONCLUSIONS

Through the obtained results it is possible to conclude that:

- All compositions obtained formed hydrogels;
- Visually, all samples presented a similar appearance;
- Hydrogels with the highest index of satisfaction in the sensorial analyzes were the HB2 and HB4 hydrogels;
- The density of all samples is close to the density of the water;
- The hydrogels with the lowest degree of dehydration were HB1 and HB2;
- The hydrogel with the highest performance was HB2.

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