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Comparative Study on Physico-chemical and Sensory Properties of Vinegar Produced from Apple Varieties

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ABSTRACT

Background: The Aures region is known for its great apple production, the most cultivated varieties are the *Golden delicious*, the *Royal gala* and the *Starkrimson*, each year the region experiences huge losses caused by hail, which has a negative socio-economic impact.
Methods: In order to value these losses in apple cider vinegar, we made a comparative study of the color (juice and vinegar), physicochemical parameters (TSS, pH and total acidity) for 49 days at 30°C and protected from the light andsensory analysis of the three vinegars (VGD, VRG and VSK) obtained by the traditional method "Orléans".
Result: The results showed that the three vinegars (VGD, VRG and VSK) meet the national and international standard, the acetic acid content was 7.27, 7.18, 6.30 respectively for VSK, VRG and VGD, while sensory analysis performed on the hedonic scale by 70 adults (women and men aged between 25-45 years), reported that the *Golden delicious* vinegar was most appreciated.

Key words: *Golden delicious,* Natural fermentation, Physico-chemical parameters, *Royal gala,* Sensorial analysis, *Starkrimson,* Vinegar.

INTRODUCTION

Vinegar is very popular as traditional ingredient for cooking, pickling andpreservation. It is made from sugar or starch by an alcoholic and acetous fermentation and produces ethanol as a by-product (Jamaludin *et al*., 2018).

Apples are one of the most cultivated fruits in eastern Algeria and are a good raw material for apple cider. Apples are mainly used in the production of vinegar because it is inexpensive and good taste acidic seasoning fruit vinegar drink with high nutritional values (Song *et al.* 2019).

Apple cider vinegar (ACV), which is the most commonly manufactured vinegar globally, contains various organic acid(s) and phenolic substances (Karaalp *et al.,* 2018). The slow Orleans process is employed for the manufacture of high quality vinegars, the submerged process, which is now the main approach, does not employ surface providing materials and depends on carefully selected cultures of Acetobacter growing in aerated deep culture. However, the vinegar tends to be more cloudy and less aromatic, as there is less opportunity for flavour development to occur, for example that catalysed by the esterases (Bamforth *et al.,* 2019). When using appropriate materials, the sensory qualities of vinegar produced *via* this slow method are superior than those obtained using rapid methods. For this reason, slow fermentation is preferred for the obtention of apple vinegar and other types of vinegar derived from different fruit juice, or even balsamic vinegar (Dabija *et al.* 2014).

The objective of this study is to value the huge losses of apples in the Aures region due to hail andtheir suitability for cider production. For this purpose, different fermented apple products were selected (*Starkrimson*, *Royal gala* and *Golden delicious*) to focus on aspects related to their preparation, including the fermentation process, but also on the properties of the products, such as physicochemical properties or sensory properties andfinally the color.

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MATERIALS AND METHODS

Biological material

The apples used in this study were procured from a rod damaged by hail in 2019 in the town of Oued Taga which is located 32 km east of the Wilaya of Batna, Eastern Algeria. It is represented by the following geographical coordinates 35°19′ 48″ North, 6°13′ 48″ East with an altitude of 1532 m. It is characterized by a dry and cold semi-arid climate, which is suitable for apple trees.

The apple trees represent about 72% of the total area planted with an average production of around 68,400 quintals, this production is represented by three main varieties (*Golden delicious* 65%, *Starkrimson* 16% and *Royal gala* 14%) as informed by official from DSA, Batna.

The physico-chemical characteristics of these apples vary according to their variety, the *Golden delicious* is characterized by his yellow color with a crunchy sweet texture are TSS 12-15°Brix and a tangy taste are malic acid

4-6 g/l. The *Royal gala* is bicolor it is crunchy, very juicy and a sweet taste due to the TSS between 12-14°Brix and less acidic than the *Golden delicious has* 3-5g/l malic acid. The *Starkrimson* of crunchy texture, juicy with a neutral flavor are TSS and between 10-13°Brix, very low acidity (2-4g/l).

Apple juice production

Three apple varieties (*Starkrimson, Royal gala and Golden delicious*) that were damaged by hail (Fig 1) were recovered during the season from September to November 2019 and transported immediately to LAPAPEZA laboratory (Batna 1 University) because they are very perishable due to their water and sugar content and must be processed very quickly (Mounir *et al.,* 2018).

Apple juice was freshly prepared by mechanically squeezing fruit (Fig 3), (Wang *et al.,* 2020). In order to avoid the appearance of a mold film on the surface of the apple juice, we must proceed first to blanching fruits by soaking apples for a few seconds in boiling water andthen we immerse them quickly for a few seconds in cold water with ice cubes. This operation aims to prevent microbial contamination which will interrupt the fermentation processes. Both juices were preserved at 4°C until filtered in order to remove the seed and fibers (Tsegay *et al.*, 2019).

Vinegar production

We dispensed 500 ml of each apple juice into glass bottles with a diameter to height ratio of 1/3 and then covered with cling film to ensure anaerobic conditions is left to ferment at 25°C (Fig 4). When the alcoholic fermentation is completed, the cling film was replaced with a dry and clean cloth (Fig 5), to allow the start of acetic fermentation by a slow process (15 days) as described by Dabija *et al*., (2014); we used a food plastic tube to facilitate the necessary samples. When the veil of the vinegar mother is formed on the surface of the vinegar, it means that the number of active acetic bacteria

Fig 1: Apple damaged by hail.

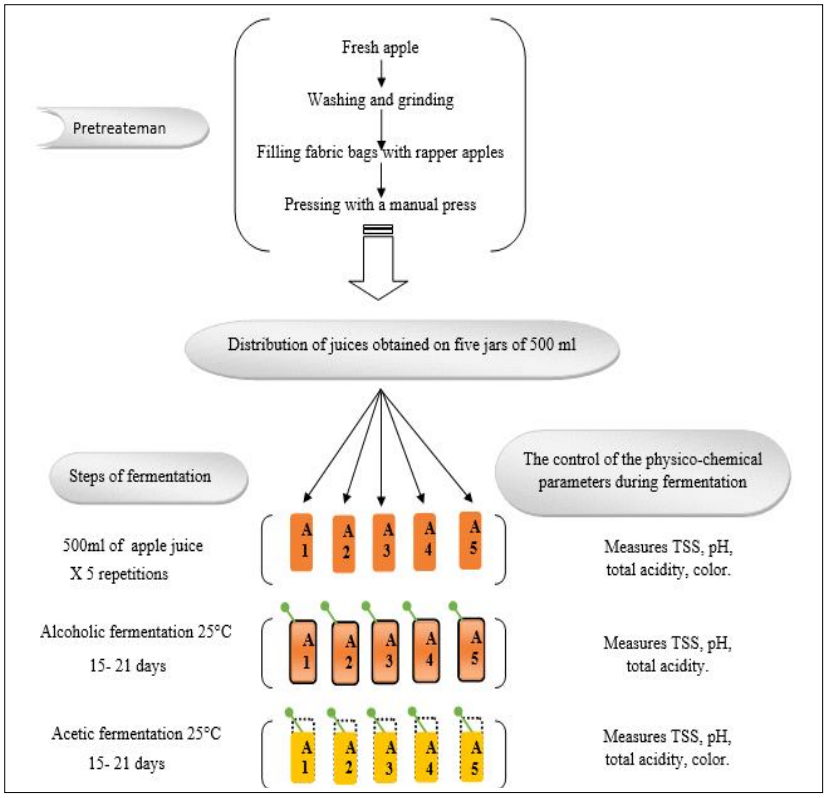

Fig 2: Diagram of apple vinegar production.

Fig 3: Manual traditional press.

Fig 4: Alcoholic fermentation.

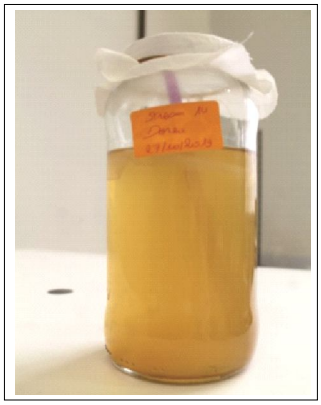

Fig 5: Acetic fermentation.

is high, Monitoring measurements of physico-chemical parameters allows to choose the time of the end of fermentation, all the tests were repeated five times (Fig 2) and the results were recorded to Riekstina Dolge *et al.,* (2012) The extraction yield was measured according to Viana *et al.,* (2017).

Physico-chemical parameters measurements

pH was measured using a pH meter (HANNA instruments pH211, made in Romania), (Tan, 2005). Total acidity was estimated by titration method using phenolphthalein as an indicator (Sharma *et al.,* 2018). Total soluble solid (TSS), were measured in °Brix using a refractometer (ATAGO made in Japan) according to Kang *et al.* (2020). The results were obtained by the following equation: Sucrose (%) = [Total sugars (%) - Reducing sugars (%)] × 0.95 (Boulal *et al.,* 2013).

Color parameters (lightness L*, a* and b*) according to the CIE D65 standard illuminant and the 10° observer of apple vinegar was determined with a CR 10 (Konica Minolta Sensing, Japan) according to Adebayo-Oyetoro *et al.* (2017). The results are obtained by direct reading on the econ of the device.

Sensory evaluation

The sensory analysis of the apple cider vinegar samples was conducted using the hedonic evaluation test. The nine-point hedonic scale method described by Sharma *et al.* (2018) was followed to perform the sensory evaluation by 70 tasters formed by men and women aged 24-45 years (students, professors andadministrators, from the Department of Agronomy of Batna 1 University, Algeria). They indicated and scored the 5 proposed vinegars (03 manufactured apple cider vinegars and 2 commercial vinegars using a 5 and 9 points scale, with 1 for “extremely unpleasant” and 9 for “extremely pleasant”. The attributes were color, brightness, evenness, sweetness, saltiness, bitterness, odor, flavor (ISO 4121:2003). The samples were served in white plastic glasses and were marked with the letters A, B, C, D, E.

Statistical analysis

Statistical analysis of quantitative data of physicochemical parameters obtained from the 5 replicates, during 46 days of fermentation monitoring were analyzed by ANOVA and kusckal and wallis test using SPSS 20.0 software version 2011 for Windows, followed by Tukey’s post-hoc test. Significance was evaluated at 5% level. Vinegar and fermentation time were treated as main factors.

A comparison of the color components (L*, a*, b*) between t0 and t49 for the three replicates was performed by the student’s t-test, using SPSS 20.0 version 2011 for Windows.

The statistical analysis of the qualitative data obtained from the sensory evaluation of 03 manufactured apple cider vinegars and 02 commercial vinegars (by 70 adults aged between 22-45 years) was performed by analysis of variance using multivariate ANOVA to determine the significant threshold for the sensory attributes and $p<0.05$ was considered statistically significant followed by a principal component analysis (PCA) and a linear correlation analysis was performed by SPSS 20.0 for Windows (trial version).

Results and Discussion

Extraction yield

According to the results presented in Table 1, all 3 apple varieties presented high yields for both juice and vinegar.

Table 1: Apple juice and apple vinegar yield for apples harvested in December 2019.

Variety	Weight	Juice yield		Vinegar yield	
Royal gala	5 kg	3,5 liters	70%	3.15 liters	90%
Starkrimson	5 kg	3,1 liters	62%	2.97 liters	95.8%
Golden delicious	5 kg	3,4 liters	68%	2.92 liters	85.88%

This may be due to the juice extraction method (fresh squeezing method) which was reported by Wang *et al.,* (2020) as the best method compared to the two other methods (spiral juice method and broken juice method). Our results showed higher rates than those reported by Mounir *et al.* (2018) with 0.68 l/kg as a yield rate. On one hand, this could be explained by the use of a stainless steel electric crusher for crushing fresh apples and then pressing them of using a manual press with steel grids and this process can affect the yield rate. On the other hand; the maturity of the apples used in their study because mature apples give lower yields due to the difficulty of the extraction procedure (Guiné *et al.,* 2021).

We have found that *Starkrimson* variety exhibited the highest yield of juice and vinegar, followed by *Royal gala* and *Golden delicious* varieties. (Mounir *et al.* 2018) used the variety *Golden delicious* in their study and reported a low yield.

Physico chemical properties of apple vinegars

The evolution of the fermentation for the 03 apple juices (*Starkrimson* juice, *Royal gala* juice and *Golden delicious* juice) was monitored by measuring the physico-chemical parameters (pH, acidity, TSS) for more than 8 weeks and the color of fresh apple juice and apple cider vinegar, in order to determine the difference between these three organic vinegars. A close perusal of data presented in Fig 6, 7 and 8 exhibited significant variations in fruit physico-chemical characteristics of different apple varieties.

a. Comparison of the 03 apple juices TSS values during their fermentation

The Brix value refers to the total solubility content in vinegar. It is also an indicator for the total sugar amount in the sample based on the total soluble solid (TSS) (Jamaludin *et al.*, 2017). Fig 6 showed a decrease in the total sugar levels of the three apple juices during alcoholic fermentation (15.6° to 8.58, 13.6 to 4.48 and 12.2 to 4.14 for *Starkrimson*, *Royal Gala* and *Golden delicious* juices respectively, with a significant difference in TSS for all of them (p = 0.000). The *Starkrimson* variety recorded the highest TSS the while the lowest TSS was recorded in the *Golden delicious* variety. Kishor *et al.*, (2017) reported that these appreciable differences in TSS values between different apple varieties could be explained on the basis of genetic differences with respect to the various genotypes, which can affect their synthesis *via* photosynthesis and also their subsequent breakdown into simple metabolites.

Bongers *et al.,* (1994) and Joshi *et al.*, (2018) reported the same TSS rate for the *Golden delicious* variety. In contrast; Tian *et al.,* (2018) gave a higher TSS corresponding to 13.11.

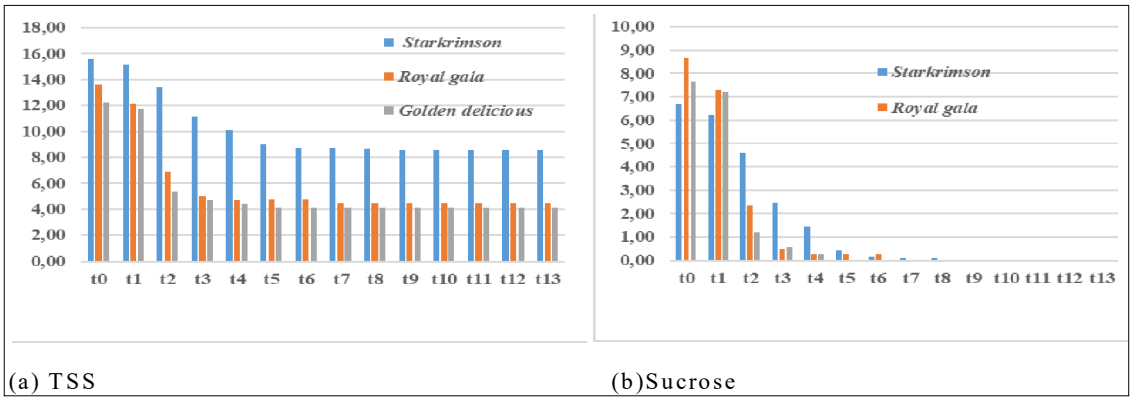

Fig 6: Monitoring of sugar decrease during the fermentation of the 03 apple juices.

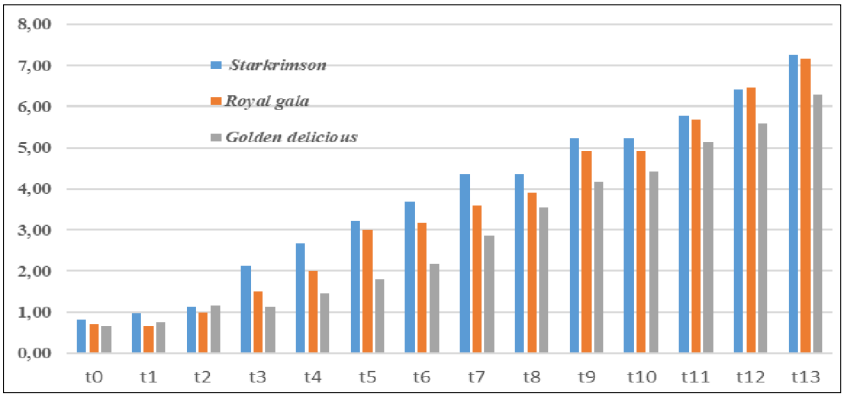

Fig 7: Monitoring of acetic acid production during fermentation of the three apple juices.

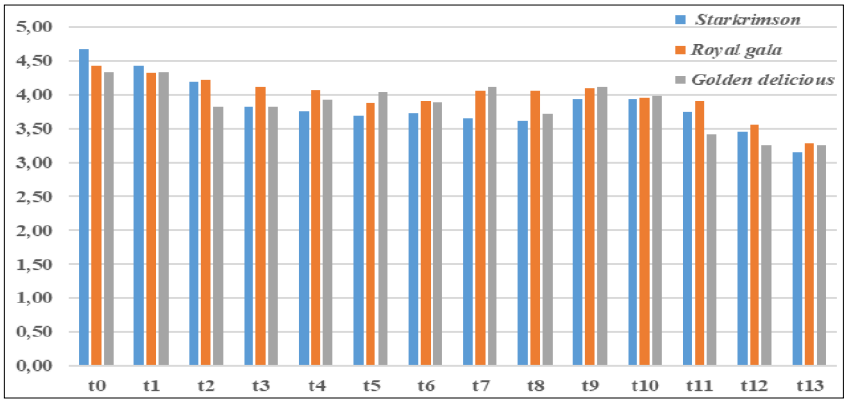

Fig 8: Monitoring of the pH variation during fermentation of the three apple juices.

b. Comparison of the percentage of obtained acetic acid of the three apple juices during their fermentation

Acetic acid production increased during t3 for all apple juices (Fig 7) and reached its maximum value at the end of acetic fermentation (t13). The results showed a significant variation in acetic acid content for the three studied vinegars (p=0.032), where 7.26% for *Starkrimson* vinegar, 7.18% for *Royal gala* vinegar and 6.30% for *Golden delicious* vinegar. Our results are consistent with those of Martín *et al.* (2017) and Mahamuni *et al.* (2020) who reported that the concentration of acetic acid varied from 5% to 20% (w/v) in vinegars produced from other alcoholic bases than wine and also with those of Dabija *et al.* (2014) who reported a level of 3.9 to 9%.

c. Comparison of the pH values of the three apple juices during their fermentation

The pH value indicates a measure of acid strength in food, thus the lower pH value shows the higher acidity (Jamaludin *et al.,* 2017).

Our monitoring showed that the pH level decreases over time for the studied apple juices. For the *Starkrimson* variety, its apple juice showed an important pH decrease (4.68 to 3.16). But for the other varieties, this decrease was less: *Royal gala* apple cider vinegar (4.42 to 3.29) and *Golden delicious* apple cider vinegar (4.33 to 3.26) (Fig 8).

The differences in the acidity level of the fruits are attributed to the presence of varying amounts of organic acids in these fruits. The obtained values agreed with Selvanathan *et al.*, (2020) who reported an acidity value of 3.12% and Wilczynski *et al.* (2019) reported acidity values between 3.37 and 4.24. The kruskal-wallis test showed a significant difference in values of the 03 varieties (p=0.032).

As can be seen in Fig 10, the total acidity of *Royal gala* apple cider vinegar was the lowest of all because it has the highest pH value (pH is inversely proportional to its total acid content). In the study of Joshi *et al.* (2018), the pH ranged from 3.35 to 3.62 for the *Golden delicious* variety which is in agreement with our results (3.26).

Color

The color of the three samples changed from brown to light yellow (Fig 9). This is due to the variation of physico-chemical parameters (Table 2), probably the increase of the acetic acid concentration, which favors the sedimentation of fibers (pectin) andexplains the regularity, the light yellow color and the brightness of the obtained vinegars. Also, the decrease in the sugar rate consequently decreases the enzymatic browning reactions (Nizar, 2009).

The correlation test for of the *Starkrimson* variety parameters showed that the “L and b” values are not correlated with the physico-chemical parameters while the “a” value is positively correlated “1” with the acidity and negatively correlated”-1" with TSS and pH. L, a and b values of the *Royal gala* variety, are positively correlated with TSS and pH “1” and negatively correlated with acidity “-1”. Finally, for the *Golden delicious* variety, L and b parameters are negatively correlated with TSS and pH “1” and positively correlated with acidity “-1”, however; the “a” parameter is positively correlated with TSS and pH “1” and negatively correlated with acidity “-1”.

Sensory analysis

The sensory analysis was performed by 70 participants, for the three obtained vinegars by fermentation of the juice of three apple varieties (VGD, VRG and VSK) and the two commercial vinegars (VNC and VPC), using the hedonic test, to assess their acceptances and preferences. It allowed us to classify the vinegars by calculating the average grade for the five assigned vinegars. The VGD vinegar rated 7.01 moderately pleasant, the (VNC, VSK and VRG) vinegars rated 6.47, 6.54 and 6.56 respectively as slightly pleasant and the VPC vinegar 5.23 neutral. (Fig 10).

The average rating for each attribute allowed us to classify the vinegars as follows; (VNC, VGD, VRG and VSK) very pleasant for the color, (VGD, VRG and VSK) moderately pleasant for the shine, (VNC, VGD, VRG and VSK) very pleasant for the regularity, VSK very pleasant for sweet taste, (VNC and VGD) very pleasant for acid taste

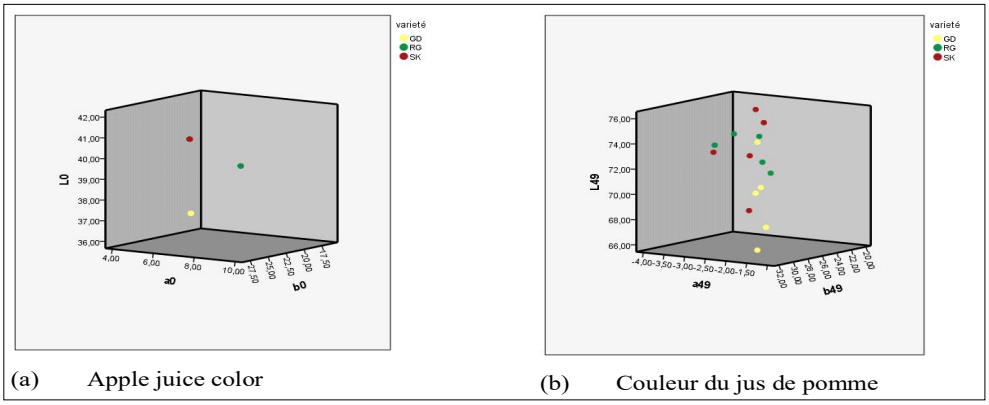

Fig 9: 3D representation of the color variation between juice and obtained vinegar for the three apple varieties.

Table 2: The influence of the physico-chemical parameters on the color of the three obtained vinegars at the end of fermentation.

Varieties	Juice apple						Vinegar apple					
	TSS	TA%	pH	L	a	b	TSS	TA	pH	L	a	b
Starkrimson	15,60	0,84	4,68	41,0±0	6,95±0	26,3±0	8,58	7,27	3,16	72,6±2,99	-3,31±0,40	22,39±1,17
Royal gala	13,6	0,70	4,42	40,0±0	9,25±0	25,75±0	4,48	7,18	3,29	72,6±1,38	-3,24±0,42	22,52±3.68
Golden delicious	12,2	0,66	4,33	36,6±0	4,00±0	17,7±0	4,14	6,30	3,26	69,9±2,83	-1,83±0,49	29,0±2,46

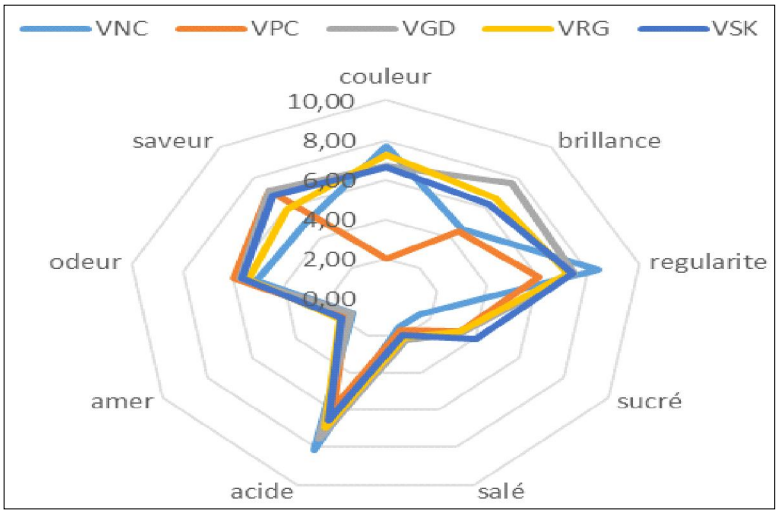

Fig 10: Spider representation of the Sensory profiles plots of mean attribute values for VNC, VPC, VGD, VRG et VSK.

and (VPC, VGD and VSK) moderately pleasant for flavor. All vinegars have a slightly pleasant smell and no salty or bitter taste was noticeable (neutral). There is a significant difference (p=0.000) between the 3 vinegars where the VGD was evaluated as the best one on all levels.

(VNC) commercial unflavored vinegar, (VPC) commercial apple vinegar, (VGD) *Golden delicious* apple cider vinegar, (VRG) *Royal gala* apple cider vinegar, (VSK) *Starkrimson* apple cider vinegar.

CONCLUSION

The comparative study of physico-chemical parameters of acetic fermentation of three apple juices, harvested in the region of Oued Taga, Algeria, has allowed to classify the three obtained vinegars according to their yield in acetic acid: VSK (7.27%), VRG (7.18%) and VGD (6.30%), which is a very acceptable yield compared to the sugar content of the initial juice. These results meet the recommendations of Algerian and international standards of apple cider vinegar which is higher than 5%. The results of the measurement of the color of the three juices showed a different color between D0 and D49, the color of our vinegar is light yellow.

The sensory analysis carried out for five vinegars showed that three vinegars achieved by acetic fermentation (VGD, VRG and VSK) and the two commercial vinegars (VNC and VPC), allowed us to classify these vinegars according to the rating of the resulted attributes; (VGD) rated moderately pleasant, (VRG), (VSK) and (VNC) rated slightly

pleasant, (VPC) rated neutral. These results are encouraging for the industrial sector, because the *Golden delicious* is the most abundant variety in this region andthe valorization of the losses in vinegar will have a positive socio-economic impact, in order to improve the sensory quality of this vinegar, a study on the aromatization by medicinal plants is in progress for the vinegar (VGD).

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Conflict of interest: None.

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