

# Physico-Chemical Characterization of Two Jams Cow's Milk Fortified with Apple Pectin and Citrus

Acheheb Hakima<sup>1</sup>, Rabah wahiba<sup>2</sup>, and Moussous wahiba<sup>3</sup>

**Abstract**—Our work has focused on a test formulation of two jams made from cow's milk fortified with citrus pectin and apple to compare their physicochemical characteristics and microbiological sensory. Comparison of the two pectins revealed results similar of the characteristics jams cow milk.

**Keywords**— cow milk, apple pectin, jam, citrus pectin.

## I. INTRODUCTION

MILK is the basis for human consumption, it is a complete food which covers much of the food needs of the individual young adult stage and ensuring the growth of the child and care of the elderly. Algeria is the first dairy consumer Maghreb with 100 to 110 l /capita/year, is expected to reach 135 l /capita/year in 2010 [1].Pectin is used in many food preparations, as a gelling agent in jams and jellies, as a thickener and emulsifier in dairy products. Pectin is also used in pharmaceutical and cosmetic products for its gelling capacity [2]. The extraction process is based on the hydrolysis in acid medium followed by filtration and precipitation in alcohol such as 2-propanol [3], the extraction is usually performed at 90 °C for a minimum time of one hour [4], these conditions are often detrimental to the quality of pectin. Use pectin in the food sector has two advantages as Trilly and Bourgeois [5]:Technological interest or it is used to obtain the desired texture in food products (product quality),and therapeutic interest with respect to some pathological cases where it is incorporated into the feed formulation. It is in the context of formulating a jam made from cow's milk enriched with pectin that is built to work with a control of physico-chemical and microbiological parameters of the raw materials and the finished product (formulation and physicochemical comparison chemical and microbiological two jams made from milk fortified with apple pectin and citrus).

## II. MATERIAL AND METHODS

### A. Material

Raw materials used are raw cow's milk, industrial apple pectins and citrus, sugar trade, industrial vanilla, table salt, sodium bicarbonate.

### B. Methods

#### B.1. The physico-chemical analyzes of pectins

##### B.1.1. Determination of moisture

Humidity is the amount of free water on the surface of a solid adsorbent [6].The procedure according Sulebele and Alexander [7] is place in the oven at 60 °C a mass of pectin, dry for 3 to 5 hours until a constant mass.

The moisture content is given by the following formula:

$$H\% = \frac{M_0 - M_1}{M_0} \times 100 \quad \text{equa 1}$$

M<sub>0</sub>: Initial weight of the pectin (before drying) in g  
M<sub>1</sub>: mass of the final pectin (after drying) in g

##### B.1.2. Determination of ash

The ash content corresponds to the resulting white residue of incineration of the sample analyzed.It's a very important parameter for determining the purity of the pectins according to the procedure of Sulebele and Alexander [7] :

Introducing into a crucible (capsule) a previously weighed, amount of pectin, and burnt on a benzene spout during 30 min and put in a muffle furnace at a temperature of 600 °C for 4 hours, then cool in a desiccator and weigh. The ash content is calculated by the following formula:

$$C\% = \frac{M_1}{M_0} \times 100 \quad \text{equa 2}$$

M<sub>0</sub>: Initial weight of pectin (prior burning) in g  
M<sub>1</sub>: mass of the final pectin (after incineration) in g

##### B.1.3. Determination of content methoxyls

The determination of the percentage of methoxyls groups is used to determine the number of carboxyl groups esterified with methanol according to the procedure of Creedy (1990) [8]: • Wet mass of pectin (0.5 g) with 5 ml of ethanol with the addition of 100 ml of distilled water, 1 g of NaCl and 6 drops phenol red, to ensure the complete dissolution of the pectin.

- Slowly titrate the mixture with NaOH (0.1 N) until the indicator changes to pink (pH = 7.5), the color of the solution must persist for at least 30 seconds.
- Add to the neutral solution prepared, 25 ml of NaOH (0.25

\*Corresponding author: ACHEHEB hakima : Hassen badi, high school of agronomy, food sciences and nutrition department, Algeria. E-mail address: acheheb.hakima@hotmail.fr, Phone: +213 560 31 78 65 Fax: +213 65 45 45

<sup>1</sup>food Technology And Human Nutrition Department, Superior National School Of Agronomy El Harrach, ALGERIA

<sup>2,3</sup>food Department, Blida University, ALGERIA

N) with continuous stirring and let stand for 30 minutes, add 25 ml of HCl (0.25 N) plus a few drops of the indicator (phenol red). • Titrate the solution with NaOH (0.1 N) until the indicator changes. to pink. The percentage of méthoxyls groups is calculated using the following formula:

$$MeO\% = \frac{meq\ of\ NaOH(0.1N) \times P}{P_0} \times 100 \quad \text{equa3}$$

P<sub>0</sub>: taking weight (mg)

Meq: the equivalent weight of NaOH (0.1N) titration volume  
p: molecular weight of CH<sub>3</sub>O (31 g / mol)

#### B.1.4. Determination of the galacturonic acid content

The uronic acid assay was carried out using the principle of colorimetric method. The uronic acids assays based on the condensation of compounds of furfural (5-formyl furic acid), formed by heating in an acid medium, with various products, the most used are: carbazole, metahydroxydiphenyl, the arminine and indole. Carbazole (dibenzopyrrolle) used in our experiment gives with galacturonic acid in sulfuric acid and hot pink purple specific color [9]. The procedure according is:

- Put into a test tube, 1 ml of pectin solution to 0.35%.
- Saponify with 1 ml of NaOH (0.05 N) for 30 min at room temperature.
- Add using a bruvette 6 ml of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) concentrate slowly and steadily along the walls of the tubes immersed in ice water and stirring.
- Keep the mixture in a boiling water bath for 20 min and then rapidly cooling the tubes in an ice bath.
- Add 0.2 ml of the solution prepared carbazole later and carefully stir the mixture. Let stand 25 minutes in the dark. The absorbances were determined using a UV-Visible spectrophotometer at a wavelength of 530 nm. The galacturonic acid content is determined using a calibration curve. White (control) underwent the same treatment except that it is added 1 ml of NaOH (0.125N) instead of carbazole.

AGA content is calculated by the following formula (Thibault, 1979):

$$AGA\% = \frac{m}{M} \times f \times 0.91 \times 100 \quad \text{equa 4}$$

m = weight in g of AGM statement on the calibration curve.

M: weight in g of the sample analyzed.

f = dilution factor of the sample.

0.91:Conversion factor acid hydrate galacturoique ahydre galacturonic acid.

#### B.1.5. Identification of functional groups of the AGM and pectin by FTIR

Infrared spectra allow the identification of functional groups, the presence of specific absorption bands can be used to prove the presence or absence of certain functional groups in a given molecule. The infrared spectrum shows the transmittance graduated from 0 to 100% depending on the number of waves, a low transmittance is a minimum on the curve, a high transmission is a maximum on the curve. The absorption of the functional groups in organic chemistry strips are between 4000 and 1500 cm<sup>-1</sup>. Between 1500 and 660 cm<sup>-1</sup>, is a region often with many absorption bands due to the CH and CC bonds, this region is the fingerprint that is characteristic of a molecule [10]. The absorbed energy to excite a molecule to a higher level of infrared radiation causes

vibration. Therefore subject to the molecule absorbs some IR radiation and absorption bands are obtained, each energy absorption corresponds to the presence of specific structural features within the molecule. The strips can be in the form of narrow, thin or expanded with a medium or low absorption peaks, all depends on the structure of the molecule [11].

#### B.1.6 . Determination of the degree of methylation (DM)

The degree of methylation is the percentage of the acid groups esterified with methanol. DM is given by the following formula [12] :

$$DM = \frac{176}{31} \times \frac{MeO}{AGA} \times 100 \quad \text{equa5}$$

176: molecular weight of galacturonic acid

31: molecular weight of CH<sub>3</sub>O

MeO: content methoxyl groups

AGA: galacturonic acid content.

#### B.1.7. Determination of the intrinsic viscosity and molecular weight

The viscosity of the pectic substances, which are macromolecules, is a function of concentration (C) and their molecular weight [13] . To compute the intrinsic viscosity curve are reduced in viscosity according to the concentration is established. The intrinsic viscosity is given by the value of the ordered intersection of two lines [14] . The reduced viscosity is determined by the physical method based on the use of the capillary tube viscometer Uhlhode, according to the procedure of Lorient and *al.*, [15]: -Prepare an aqueous solution A 500 ml 0.155 M NaCl and 0.055 M EDTA, sufficient quantity for 500 ml Distilled H<sub>2</sub>O - Prepare A 0.2% pectin solution using solution A; it is the mother solution from which serial dilutions were prepared: 0.15%, 0.10% and 0.05%. -Set Temperature viscometer at 30 ± 0.1 ° C, then poured into the capillary tube 15 ml of solution A and calculate the time corresponding to the flow passage of the meniscus M1 at 2 M stock solution and dilutions undergo the same operation.

are The reduced viscosity (dl / g) is calculated by the following equation:

$$\eta_{re} = \frac{t-t_0}{t_0 \times (C)} \quad \text{equa6}$$

t<sub>0</sub>: flow time of the solvent (A) in the capillary tube (in s)

t: flow time of sample solution (in s)

[C]: the concentration of the sample (g / dl)

$\eta_i$  Intrinsic viscosity is related to the viscosity-average molecular weight (M<sub>v</sub>) by the Mark-Houwink relationship:

$$[\eta_i] = K \times M_v^a \quad \text{equa7}$$

Thus the molecular weight is calculated as follows:

$$\sqrt{\frac{\eta_i}{K}}$$

$$M = a \times \text{equa8}$$

Where K and a are constants dependent solvent, and temperature of the polymer. In the case of pectin: a = 1.34 and K = 1.4 10<sup>-6</sup> , [16].

#### B.1.8. Determination of gel strength

The method used for determining the gel strength is that of

Jacovliv which is based on visual determination of a limit gelling transparent glass tube [17], the procedure is:

- Prepare a sugar syrup tart containing 600 g of sucrose and 20 g of tartaric acid per liter, with a maximum temperature of 50 °C.
- Prepare the reagent with 2 volumes of the cooled syrup and 1 volume of methanol.
- Pour into glass tubes, known volumes of solutions containing the reagent, water and pectin extract to be tested.
- Stir and place the tubes in a water bath 15 °C for 30 min. Frosts are estimated formed by placing the tube to examine almost horizontally with a slight slope towards the hole.

The composition of the test tubes is shown in the following table:

| TABLE I<br>COMPOSITION OF TEST TUBES |   |
|--------------------------------------|---|
| Tube                                 | Composition   |
| 1                                    | 30 ml reactive<br>2 ml water<br>8 ml pectin extracted     |
| 2                                    | 30 ml reactive<br>2,5 ml water<br>7,5 ml pectin extracted |
| 3                                    | 30 ml reactive<br>3 ml water<br>7 ml pectin extracted     |
| 4                                    | 30 ml reactive<br>3,5 ml water<br>6,5 ml pectin extracted |

The grade of Jacovliv power gelling pectins will equal according to Açourene [17]:

$$\frac{10}{n} \quad \text{equa9}$$

n = the number of ml of the extract pectin needed to produce the limit gelling.

## B.2. The physico-chemical analyzes of cow milk

### B.2.1. Determination of the titratable acidity of the milk cow

The titratable acidity of the milk is the amount of lactic acid released by transformation of lactose to lactic acid in the presence of lactic acid bacteria. The principle is based on the titration of lactic acid with an alkaline solution (NaOH 0.11 mol / l) in the presence of a color indicator which is the result phenophthalein. The Dornic is expressed in degrees (°R). 1 D corresponds to 0.1 g / l of lactic acid.

Calculation and expression of results is as follows:

$$\text{Acidity}= 10. V (\text{°D}) \quad \text{equa10}$$

V: number of ml of sodium hydroxide consumed.

### B.2.2. Density Determination of milk

The density at 20 °C or the density of a body fluid is equal to the ratio of the mass of a certain volume of the body mass of the same volume of water at 4 °C. It is expressed in kg / m<sup>3</sup>, as a decimal number without units.

The principle is the determination of density by using a densitometer, it is a device used for determining the density of the milk (lactometer). For the determination of the density of our milk is first, must check the temperature of the milk should be 20 °C, to complete our test so inclined to avoid foaming that can skew analysis (at temperatures other than 20 °C, one must consult the shape of the correction). We plunges our lacto-densimeter in milk and is expected to stabilize the

device, then read directly the value found.

If the read temperature T is below 20 °C, the density D will be equal to :

$$D = D_{\text{lue}} + 0,2 (T_{\text{lue}} - 20) \quad \text{equa11}$$

If the read temperature T is greater than 20 °C, the density D will be equal to:

$$D = D_{\text{lue}} - 0,2 (20 - T_{\text{lue}}) \quad \text{equa12}$$

With 0.2 is the correction factor

We read the value directly from the intersection of lacto-densitometer with milk

### B.2.3. Determination of potential of hydrogen (pH)

The pH is the chemical potential of "H<sup>+</sup>" ions in a solution. It is measured using a pH meter.

### B.2.4. Determination of fat in milk (F)

Its principle is milk attack by sulfuric acid and separation by centrifugation in the presence of iso-amyl alcohol MG released.

The result is expressed in weight percent.

### B.2.5. Determination of the total solids content of milk (EST)

2 methods was used:

- Determination of dry matter by the official method: The dry extract product is the percentage of existing solids in the resulting desiccation of milk produced. The principle is generally based on milk by drying evaporation of the water absorbed or adsorbed form and to the weighing of the residuals. The procedure is to put in the cup of dried and weighed aluminum weigh 5 g of milk and then place it in the oven at 103 °C for three hours. Then put the cup in the dryer glass for 30 minutes before weighing. The milk solids is expressed in weight percent:

$$TSC(\%) = (M_1 - M_0) / (M_2 - M_0) \cdot 100 \quad \text{equa13}$$

where:

M<sub>0</sub>: the mass in grams of the empty cup  
M<sub>1</sub>: the mass in grams of the dish and residue after dryer  
M<sub>2</sub>: the mass in grams of the cup and of the sample.

- Determination of solids by the method of thermo-balance : The principle is based on the evaporation of water contained in the sample to be analyzed, under the heat source is in that case is the effect of infrared light. The procedure is to put it in the cup dried and weighed aluminum, 2 g ± 5 mg of analyte , after spreading over the entire surface of the cup, being careful not to touch the edges of the cup. The cup is placed in the unit and the unit is turned on by lowering the lid and pressing Start. After 19 minutes, an audible beep indicates the end of the drying process, and the result is displayed on screen of the device as a weight percentage of dry matter relative to the total.

### B.2.6. Determination of fat-free dry milk

It is calculated by the following formula :

$$FFD (\%) = TSC - F \quad \text{equa14}$$

### B.2.7. Determination of wetting

Wetting is a fraud resulting in lower levels of components. One can determine the degree of wetting in the following relationship:

$$\frac{B - C}{B} \times 100$$

wetting [%] = equa15

where:

B: non-fat solids g / l of the sample (for a standard milk fat dry extract is set at 87 g / l).

C: non-fat solids g / l of the test sample

### *B.3. Physico-chemical analysis of salt, sodium bicarbonate, vanilla*

#### *B.3.1. Determination of titratable acidity*

The titratable acidity of dry milk is the number of milliliters of sodium hydroxide solution at 0.1 mol/L necessary to neutralize to pH 8.4 a solution of reconstituted milk corresponding to 1 g of non-fat solid. The titratable acidity is expressed as per liter of lactic acid (degree Dornic).

#### *B.3.2. Determination of fat (Gerber method)*

This method is used to determine the fat content of reconstituted milk used to prepare dairy derivatives, and UHT milk. Its principle consists in a degradation of milk by sulfuric acid (0.1N) ( $H_2SO_4$ ) and centrifugal separation in the presence of iso-amyl alcohol of the fat released. The procedure is to put in a butyromètre 10 ml of sulfuric acid d = 1.825 avoid wetting the neck, and then 10 ml of distilled water plus 2.5 g of the sample without wetting the neck and butyromètre avoiding premature mixing milk with acid. Poured on the surface 1 ml iso amyl alcohol as without wetting the neck avoiding the mixing of fluids (if necessary wipe the neck butyromètre) stuffy carefully. The butyrometer stirred gently but thoroughly and quickly until no lumps. After a good stir, do not cool the butyromètre (if necessary warmed to 65 ° C in a water bath). The cap is adjusted so that the liquid level is in the upper part of the scale, centrifuged at 1500 rev / min for 5 min including the time necessary to reach the required speed. After 5 min, the butyrometer is immersed vertically cap down in the water bath and allowed 5 min. The butyrometer is maintained and adjusted vertically with the cap to coincide with the lipid phase and division are read quickly. The result is expressed in weight percent. According to NF-V04-210: The fat is separated less dense, it collects in a clear and transparent layer visible for direct playback on wide butyrometer.

The percentage of fat by weight of the product is given by the following formulation:

$$F=n_1-n_2 \quad \text{equa16}$$

with:

n<sub>1</sub>: represents the value reached by the lower level of the fat column.

n<sub>2</sub>: represents the value reached by the upper level of the fatty column.

The Fat content is expressed as a percentage or in g/l.

#### *B.3.3. Determination of pH (AFNOR, 1986)*

The pH measurement is made directly by means of a pH meter using the steps: •Set the temperature correction than the product; • Calibrate the pH meter by immersing the glass

electrode in a buffer solution; • Insert directly into the product; •Read the result directly on the dial of the pH meter.

#### *B.3.4. Determination of moisture (ISO 9001)*

Mean moisture content of the analyte mass loss of material when subjected to drying the procedure that follows, expressed

as a percentage. The moisture percentage of the sample is calculated by the following formula:

$$H (\%)=100-TSC \quad \text{equa17}$$

#### *B.4. Physicochemical analyzes jam*

##### *B.4.1. Determination of Brix*

Brix measures the rate of soluble solids contained in a sugar solution. It is determined with a refractometer.

##### *B.4.2. Determination of pH*

To determine the pH, a pH meter electronics Corning brand was used. The electrode was immersed in the solution to be analyzed jam after calibrated with pH buffer solutions (4) and (7). A direct reading of the pH is obtained.

##### *B.4.3. Determination of Total Acidity*

It corresponds to the set of fixed or volatile substances contained in the acid reaction jam. To evaluate their concentration, a base (0.1N NaOH) was acts on the jam prepared solution (100ml) according to the principle that an equivalent base neutralized acid equivalent in the presence of phenol phthalein. It is expressed as mg or citric meq/100 g of product acid.

##### *B.4.4. Determination of total sugars and reducing sugars*

The chemical assay method is based on the principle of reducing the copper and  $CuSO_4$  alkali  $Cu_2O$  the copper oxide formed is then oxidized by nascent iodine projecting from a solution of iodide and iodate potassium in sulfuric acid. The procedure is to titrate the excess iodine with sodium thiosulfate in the presence of starch after the first hydrolysis (reducing sugars) and a second hydrolysis (total sugars). To assay the entire sugar makes it all gearboxes, which means that hydrolyzes sucrose into glucose and fructose, reducing sugars themselves. For non-reducing sugars, the determination was made by polarimetric method (using a polarimeter) and total sugars by the chemical method and from the formula:

$$\text{Total sugar} = \text{reducing sugar} - \text{non-reducing sugars}$$

Determination of reducing sugars is described by AOAC 22,013: The procedure is to put in a porcelain dish previously incinerated and 10 grams of jam calibrated accurately weighed. Dried under vacuum at 70°C until two (2) consecutive weighings made at 2-hour intervals did not differ by more than 3 mg. A dryer was used to bring the sample dry.

#### *B.5. sensory evaluations*

The evaluation was intended to provide both an idea about the acceptance of different formulations of jam and an overall assessment of the products. The difference tests (rank order) and preference (Hedonic), as described by Watts [18] and Larmond [19].

##### *I.2.5.1. The test of rank order*

• It was to evaluate the color and texture parameters.

- The test was conducted at the temperature of the room without influence of external agents (noise, odors)
- I.2.5.2. The Hedonic test

This test was chosen to assess the general level of appreciation samples of jam, under the same conditions as the test of difference are: same panel, same place, same coding. In addition, distilled water was made available to the tasters to take your taste buds on their toes after each sample evaluated.

The qualification 'subjective' is frequently associated with the sensor technology, as opposed to 'objective' given to instrumental methods, since the taste of an individual varies from one day to another, and it differs from that of its neighbor [20]. To confirm the acceptable texture of our preparation, submitted to a tasting at laboratory physico-chemical analysis, tasting panels composed of 10 people for an assessment of the organoleptic quality of experimental jam. The criteria for the organoleptic quality of a jam are: flavor, appearance, taste, texture, consistency, color and odor.

Terms of evaluation are:

- It takes place in a specific sensory analysis room where lighting and temperature were controlled and hygienic conditions are met (to avoid contamination of our sample).
- $T_o$  is not quenched, the samples should be numbered (1 to 4).
- Subjects must be insulated to prevent communication between them, which may influence their judgments, so everyone must have his score sheet.
- After the tasting, it is necessary to neutralize the taste impressions using a water sealed jars at aluminum foil and store in the refrigerator.

#### B.5. The microbiological analyzes

Microbiological testing performed on the raw material and the finished product to ensure role: • Good profitability of production. • Minimize losses during poor manufacture. • Quality of the finished product for direct consumption. • Good hygienic quality and merchantability of the product. In general, the goal of microbiological control is guaranteed a safe hygienic and definite sensory level [21]. The microbiological analyzes included: raw materials (cow's milk, pectin, salt baking vanilla, honey) and finished products (jam). For this we have achieved: \*The enumeration of aerobic mesophilic flora total which is an indicator of the overall condition of the

product quality;

\*The detection and enumeration of groups of indicator organisms of fecal contamination are: -coliformes fecal and total coliforms. -streptococques fecal. -Clostridium Sulphite-reducing. -Pathogens, we investigated the presence of: -Research Salmonella -Research And enumeration of Staphylococcus aureus.

#### B.6. Test formulation of a jam based citrus pectin and apple

The study was conducted on a variety of citrus genus Citrus and apple variety Golden, then, tests were performed with four doses of pectin (0.40, 0.60, 0.80, 1%). Since the texture and taste are fundamental characteristics of a jam, the dose of pectin was modified to achieve the desired texture on the basis of the formula practiced on an industrial scale, the proportions

the various ingredients are shown in Table 2.

TABLE II  
FORMULATION OF A JAM PECTIN ENRICHED

| Ingredients           | Dose |      |      |   |
|-----------------------|------|------|------|---|
| Cow milk (ml)         |      | 1000 |      |   |
| Pectin (%)            | 0,4  | 0,6  | 0,8  | 1 |
| honey (g)             |      | 2    |      |   |
| Vanilla (g)           |      | 1    |      |   |
| Salt (%)              | 0,75 | 1,25 | 1,75 | 2 |
| sodium bicarbonate(g) |      | 1    |      |   |

When preparing it necessary to take into consideration the following points:

- Choose good quality raw materials and weigh with a balance of precision;
- Mix the ingredients in a beaker;
- Heat the water to 55 ° C for the preparation of reconstituted milk;
- Addition slowly to the mixture of ingredients in the reconstituted milk subjected to agitation using a mixer, agitation must be effectively and consistently to avoid the deposition of undissolved at the bottom of the tank particles;
- Adding the flavoring is done just before boiling;
- A boiling, from the heat is removed, the foam is removed to obtain a good texture and packing is carried out in pots in sterile polystyrene;
- The pots are closed tightly.
- Note the date of preparation of the test and stored at 4°C.

#### B.7. Statistical Analysis

In order to determine the average values of the results and standard deviations, we used the Excel 2007 software. To determine the significant effect or no substitution of NaCl by KCl at different dose, we conducted an analysis of variance (ANOVA) with STATISTICA7 software.

### III. RESULTS AND DISCUSSION

#### A.1. results of physico-chemical analysis of raw materials

##### A.1.1. Pectins

Pectin used is a brown fine powder without aftertaste. The results of physico-chemical tests on the powder pectin are shown in the following table:

TABLE III  
RESULTS OF PHYSICO-CHEMICAL TESTS ON THE POWDER PECTINS

| parameters                 | Apple pectin | Citrus pectin | industrial standard |
|----------------------------|--------------|---------------|---------------------|
| Moisture (%)               | 5.44±0.02    | 5.11±0.03     | 5.20                |
| Ash (%)                    | 0.91±0.01    | 0.85±0.04     | 0.89                |
| methoxyl content (%)       | 10.02±0.14   | 10.22±0.11    | 10.96               |
| Galacturonic acid (%)      | 79.50±0.30   | 78.53±0.32    | 80.50               |
| Degree of methylation(%)   | 64.08±0.29   | 64.88±0.19    | 65.00               |
| intrinsic viscosity (dl/g) | 2.04±0.01    | 3.14±0.04     | 3.80                |
| molecular weight (mg)      | 66000±0.31   | 67320±0.38    | 68200               |
| functional groups          | 1223±0.25    | 1280±0.35     | 1282                |
| gelling power (IFT grade)  | 1.40±0.01    | 1.41±0.02     | 1.42                |

-the galacturonic acid content ranged from 78.53 to 79.50% for citrus pectin and apple, ash content varies from 0.85 to 0.91%, these results are consistent with the company standard.  
 -the methylation degree ranges from 64.88 to 64.08% for the citrus pectin and apple, indicating that the two are highly methylated pectins with a gelling power of 1.41 and 1.40 respectively, which shows information about the quality of

these powdered pectins, sachat the methoxyl content of 10.22 and 10.02%, respectively, which implies a mastery of the parameters and steps of extracting pectin industrial scale. According to Luquet [22], to reduce microbial load on the one hand and to check the other, these functional properties (solubility and wettability) the thermal treatment applied during the production of pectins aims .The pectin is the driving force in gel formation, which plays a role in the organoleptic properties of products produced, but also protects against migration of microbes.

#### A.1.2. cow milk

the results of physico-chemical tests on the cow milk are shown in the following table:

TABLE IV  
RESULTS OF PHYSICO-CHEMICAL TESTS ON THE COW MILK

| parameters              | value        | Standard<br>(AFNOR,1986) |
|-------------------------|--------------|--------------------------|
| pH                      | 6.5±0.01     | 6-7                      |
| T(°C)                   | 16±0.31      | 7-16                     |
| Titratable acidity (°D) | 16±0.33      | 16-18                    |
| F(%)                    | 31.3±0.02    | 22-39                    |
| density                 | 1.027±0.39   | 1.020-1.029              |
| TSC(%)                  | 109.2±0.33   | 105-119                  |
| FFD(%)                  | 77.9±0.23    | 70-80                    |
| freezing point (°C)     | (-49.4)±0.31 | (-40/-55)                |

After the results, we note that cow's milk is rich in fat (31.3%), with a pH and acidity, respectively, 6.5 and 16°D. other parameters are also consistent with the AFNOR standard (1986) .According to Amiot and *al.*, [23], more milk is rich in fat, the higher its density will be higher and the non-fat solids content increases, so a skimming diminura milk density.

#### A.1.3. salt, vanilla, sugar and sodium bicarbonate

The results of physico-chemical tests on the salt, vanilla and sodium bicarbonate are shown in the following table:

TABLE V  
RESULTS OF PHYSICO-CHEMICAL TESTS ON THE OTHER RAW MATERIALS

| ingredients        | Moisture (%) | industrial<br>Standard |
|--------------------|--------------|------------------------|
| salt               | 71.56±0.01   | 70-77                  |
| sugar              | 72.23±0.01   | 70-75                  |
| vanilla            | 81.75±0.31   | 70-85                  |
| Sodium bicarbonate | 27.00±0.23   | 25-30                  |

The only parameter that could affect the analyzed raw material is moisture, it is respectively 27% and 71.56,72.23,81.75 for salt, sugar, vanilla and baking soda, these values are consistent with the standard with the good storage conditions. Salt used during the bittering auxiliary is considered in the processing of certain compounds which impart a bitter taste to the finished product that could make it undrinkable. Sugar is a preservative that allows the partial dehydration and increases the solids which will make it difficult to develop some germs materials, but sugar is also involved in the formation of gel and gives a better taste to the finished product.Sodium bicarbonate acts as an emulsifier between the aqueous and oil phase.

#### A.2. results of physico-chemical analysis of finished products

The results of physico-chemical tests on the finished products are shown in the following table:

TABLE VI  
RESULTS OF PHYSICO-CHEMICAL TESTS ON THE FINISHED PRODUCTS

| parameters                   | Test1      | Test2      | Test3      | Test4      |
|------------------------------|------------|------------|------------|------------|
| Degree brix(°B)              | 67±0.21    | 64.5±0.23  | 64±0.20    | 64.5±0.21  |
| pH                           | 6.5±0.05   | 7.18±0.03  | 7.09±0.05  | 7.05±0.03  |
| Acidity(mg citric acid/100g) | 16±0.18    | 17.22±0.16 | 16.65±0.18 | 17.88±0.17 |
| Sugar reducto(mg)            | 53.2±0.21  | 54.4±0.21  | 53.5±0.23  | 52.87±0.20 |
| Total sugar(mg)              | 56.58±0.35 | 53.78±0.31 | 53.88±0.31 |            |
| Moisture(%)                  | 29.8±0.28  | 28.8±0.27  | 29.2±0.28  | 31.66±0.30 |

After the results we note that:

-the brix varies from 64 to 67 ° B for 4 test formulation jam vahé milk enriched in pectin, and increasing the percentage of pectin incorporation little effect ( $p>0.05$ ) and the brix remains in compliance with industry standards (63-67 ° B). -the pH from 6.50 to 7.18 and 28.80 to the humidity of 31.66% and it can be noted that increasing the percentage of pectin significantly affects the pH and moisture content ( $p<0.05$ ) as the pectins by their water retention capacity can be at the origin of this phenomenon, acidity varies to 16 at 17.88 meq citric acid / 100g jam, which shows the correlation between the pH and acidity. -the reducing sugar content ranged from 52.87 to 54.4%, and we note that the higher the percentage of incorporation of pectin increases, the reducing sugar content decreases, but the total sugar content is not influenced because pectins are added in end of cooking.

#### A.3. results of microbiological analysis of finished products

results of microbiological analysis shows a complete absence of pathogens regardless of the percentage of incorporation of pectins which implies good microbiological quality of raw materials and compliance with good manufacturing conditions and storage without oubleir that treatments heat (pasteurization) have an efficiency on the destruction of pathogenic flora and extend the life of food products [24] .

#### A.4. results of organoleptic test of finished products

Organoleptic test results showed that:

- Test 1 has a better texture gelling, a sweet, caramel color and a pleasant smell, so using 0.4% of the dose leads to gelling pectins texture, beautiful color of the finished product

- Test 2 has a medium texture gelling, a bitter taste and a honey color with a pleasant smell, which allows us to say that a percentage of 0.6% pectin gives an unpleasant texture knowing the amount of salt, sugar, worth and bicarbonate are the same, - Test 3 has good gelling texture, caramel color, sweet taste and pleasant smell, and 0.8% pectin led to a nice texture, gelling with good color, - Run 4 offers a very gelling texture, sweet, brown color and an aftertaste, and 1% pectin leads to gelling texture too which makes unpleasant jam.

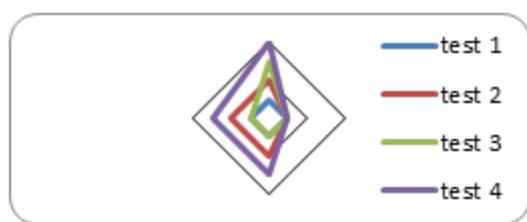


Fig 1 Sensory profile test formulation 4 jam cow milk enriched in pectin

#### IV. CONCLUSION

Through our study was to develop and compare two jams cow's milk fortified with apple pectin and citrus to assess the role of pectins in food industries. So it shows compliance of raw materials and finished products for, there was a stable brix with increasing percentage of incorporation of pectins in the formulation of jam. For pH setting, the results are shown that increasing the percentage of incorporation of pectins was increased the pH of finished and their moisture without it affecting acidity, total sugar or sugar products reducing jams milk. Microbiological analysis revealed a complete absence of pathogens Germs whatever the percentage of incorporation of pectins which implies good microbiological quality of raw materials and a good compliance with the conditions of manufacture and storage of jams. The results for the sensory analyzes show that the contribution of pectins whatever its origin (apples or citrus), shows a greater appreciation of consumers compared to samples prepared without added pectin, but this addition of pectin n 'way influences the chemical characteristics (Brix, total sugar, reducing sugar, acidity) and microbiological (total bacteria, yeasts and molds). salt (NaCl) was used as an additive in formulations jams cow milk, its influence is seen on the color of the product during the test of rank order. For the overall assessment of the product and the finished product most appreciated by the tasting panel is that corresponding to the formulation with 0.8% pectin (from apples or citrus) because the increase of 1% pectin led to a texture very gelling making jam milk too firm, and:

Ø It can lead to a jam quality (chemical and microbiological point of view) and make it competitive with the imported recipes and appropriate treatment products.  
Ø If the legislation does not prohibit the use of pectin as a food additive industry as permitted by the Codex Alimentarius, it can be used sparingly in the preparation of jam for a target audience, Ø For six (months), products produced and made the observation at room temperature show no signs of deterioration and variation in taste and color, proof that you can have an expiration date for extended in terms of months products.The study was limited to the qualitative aspect of the product rather than the economic evaluation that could be another research work, again, it worked only on pectin parameters and percentages of salt , on the other parameters (acidity, water, sugar) they will be more studies.

#### ACKNOWLEDGMENT

- AFNOR T47,212 (1986) "guide de bonne pratique pour le choix, le stokage, l'utilisation et l'entretien"

- ISO 9000 "management de la qualité, les exigences relatives à système de management de la qualité"
- NF V04-210 (2000) "lait - détermination de la teneur en matière grasse - méthode acido-butyrométrique"
- AOAC "official methods of analysis,xiiith edition" 1980, 22,013 - moisture in dried fruits, official final action

#### REFERENCES

- [1] K. benelkadiid, " industrie du lait en algérie " in journal el watan du 22 mai, 2005, pp10-12
- [2] J. pagan, A. ibarz, M. llorca, A. pagan, G.V. barbosa-canovas extraction and characterization of pectin from stored peach pomace " food research international", v 34, 2001, pp 605-612
- [3] U. kalapathy, A. proctor "effect of acid extraction and alcohol precipitation conditions on the yield and purity of soy hull pectin" , in food chemistry, v 73, 2001, pp393-396
- [4] M.T. iglesias, J.E. lozano, "extraction and characterization of sunflower pectin" , in journal of food engineering v 62, 2004, pp215-223J.
- [5] V. trilly and C.M. Bourgeois "technologie du lait : constitution, récolte , traitement et transformation du lait", 3eme edition, la maison rustique, paris, 1999, pp 4-363
- [6] J.F. drilleau, jf, salih, a.g. ,drilleau, j.f. , cavin, f.f. , divies, c. , bourgeois, c.m. "a survey of microbiological aspects of cider making" in journal of the institute of brewing,v 94, issue: 1,1988, pp5-8
- [7] S.solubele , alexander, m.m. ,solutebe, g.a. , "characterization of pectins from indian citrus peels" in journal of food science and technology-mysore, v 17, issue 4, 1980, pp 180-182.
- [8] D., creedy, R.Watson, D.R. Thompson, L.I. Gloria,"Commentary on Henderson A. Twentyman M, Eaton E, Creedy D, Stapleton P & Lloyd B, Creating supportive clinical learning environments: an intervention study" in Journal of Clinical Nursing 19, 177-182,in journal of clinical nursing,v 19 issue: 3-4,1990, pp596-598
- [9] deymie B., haas J. L., millet P., billon M. "high yield isomerisation of hops in beer prodn. - by extrusion and cooking with water and direct addn. of extruded material to wort", 1988,patent assignee name(s) and code(s): electricite de france(elec-c)cxlextal (clex-non-standard)derwent primary accession number: 1988-221662
- [10] decodts, g, barry j., bran, g. , loupy, a. ,pigeon p., sansoulet j. tetrahedron "alkylations in the absence of an organic-solvent - effects of additions of mineral oxides and ammonium-salts .2. easy synthesis of esters by alkylation of the acetate anion under economical and gentle conditions" ,volume: 39,issue: 16,1994,pp2673-2677
- [11] mahuzier G., demore, D. , kasselouri, A. , bourdon, O. , blais, J. prognon, P. , "enhancement of 5,10,15,20-tetra(m-hydroxyphenyl)chlorin fluorescence emission by inclusion in natural and modified cyclodextrins", applied spectroscopy, v 53, issue: 5,1999, pp523-527
- [12] souty, C. , picaud, J.L. , "vitellogenin synthesis in the fat-body of the marine crustacean isopoda, idotea-balthica-basteri, during vitellogenesis" , reproduction nutrition development,volume: 21,issue: 1,1981, pp95-101,(1981)
- [13] thibault, J.F., "les substances pectiques : les polymères des végétaux", edition castes et bordas N°3, paris, 1980, pp232-251
- [14] martine, V. ramon-portugal, F. , seiller, I. , taillandier, P. , favarel, J.L. , nepveu, F. , strehaiano, P. "kinetics of production and consumption of organic acids during alcoholic fermentation by saccharomyces cerevisiae" , in food technology and biotechnology,v37,issue: 4,1999
- [15] lorient, D. colas, B. , gobin, C. , "viscosity and voluminosity of caseins chemically modified by reductive alkylation with reducing sugars" , in journal of dairy research,volume: 55,issue: 4,1988, pp539-546,
- [16] cheremisinoff, N.P. "designing epdm products for extrusion applications" , in journal of macromolecular science-chemistry,volume: a26,issue: 8,1989,pp1231-1259
- [17] Acourene, A. , Ito-Kobayashi, M. , Ono, Y. , Furukawa, Y. , Takahashi, M. , Muramatsu, Y. , Umetani, M. , Takatsu, T "Colletotric acid, a novel 11 beta-hydroxysteroid dehydrogenase type 1 inhibitor from Colletotrichum gloeosporioides" ..journal of antibiotics,v61, Issue: 3, 2008, pp136-141
- [18] watts, m.l. "value of chemists" in chemical & engineering news,v69,issue: 50,1991, pp3-3
- [19] larmond, E. voicey, P.W. , "effect of deformation rate on relationship between sensory and instrumental measurements of meat tenderness by warner-bratzler method" ,in canadian institute of food science and

- technology journal-journal de l'institut canadien de science et technologie alimentaires,v10,issue: 4,1977,pp307-312
- [20] lindin, G.L. , khramtsov, V.F "calculation of the height of a protective layer of loose material with major caving of rock masses" ..,soviet mining science ussr,volume: 27,issue: 3,1991,pp171-176
- [21] bourgeois, L. and leveau S., "techniques d'analyses et de contrôle dans les industries agro-alimentaires", 2 édition, ISBN, 2-85206-599-1, lavoisier, tec et doc, 1991,paris, pp 454
- [22] luquet F.M., "lait et produits laitiers", tome I: vache, brebis, chèvres, edition tec et doc, paris, 1990,pp75-258
- [23] amiot J., fournier S., lebeuf, Y., paquin P. and simpson P. "composition et propriétés physico-chimiques des analyses du lait", in sciences et technologie du lait : transformation du lait, édition tresses internationales polytec,2002, pp2-73
- [24] bourgeois C.M., mescle, J.F., Zucca, J. " microbiologie alimentaire, tome 1, aspect microbiologique de la sécurité et de la qualité des aliments", édition tec et doc, collection science et technique agroalimentaire, 1996,pp654