



Different concentrations of flour from amazonian tropical fruit residues on the composition of ternary mixture in edible ices

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ABSTRACT: To improve the nutritional and functional quality of agro-industrial waste, his study established a ternary mixture of flours made from pineapple, acerola, and passion fruit waste using the simplex-centroid design, as well as to evaluate their effects on the physicochemical and sensory characteristics of edible ice cream. Using mixture planning, nine ternary flour components were applied to edible ice cream formulations to evaluate acceptance scores for taste, texture, and overall impression attributes, as well as physicochemical parameters (pH, acidity, soluble solids, and color parameters) and microbiological parameters (thermotolerant coliforms at 45 °C, *Enterobacteriaceae* and *Salmonella*). The simplex-centroid design demonstrated that the cubic model best predicts the results for the analyzed sensory attributes. The response surface graphs indicated the area of greatest acceptance in the formulations with the maximum percentage established for the flour with acerola waste. The highest level of acceptability was given to the ternary mixture of 60%, 30%, and 10% pineapple, passion fruit, and acerola flours, respectively. The results indicated that sorbet enriched with flour composed of the ternary mixture is a product with the potential for exploitation, as well as contributing to the reduction of environmental impacts generated by the large and improper disposal of agro-industrial fruit processing residues in the ecosystem.

Key words: amazonian tropical fruits, agro-industrial residues, mixture planning, sensory analysis.

Diferentes concentrações de farinhas dos resíduos de frutas tropicais amazônicas para composição de mistura ternária em gelados comestíveis

RESUMO: Visando a qualidade nutricional e funcional dos resíduos agroindustriais, o estudo teve como objetivo estabelecer a mistura ternária de farinhas elaboradas com resíduos de abacaxi, acerola e maracujá utilizando o delineamento simplex-centroide, assim como avaliar seus efeitos nas características físico-químicas e sensoriais em gelados comestíveis. Com o planejamento de misturas, nove componentes ternários das farinhas foram aplicados em formulações de gelados comestíveis para avaliação dos escores de aceitação para os atributos sabor, textura e impressão global, bem como os parâmetros físico-químicos (pH, acidez, sólidos solúveis e parâmetros de cor) e microbiológicos (coliformes termotolerantes a 45 °C, *Enterobacteriaceae* e *Salmonella*). O delineamento simplex-centroide demonstrou que o modelo cúbico melhor prediz os resultados para os atributos sensoriais analisados. Os gráficos de superfície de resposta indicaram a área de maior aceitação nas formulações com o máximo percentual estabelecido para a farinha com os resíduos de acerola. O maior índice de aceitabilidade foi atribuído a mistura ternária constituída com 60%, 30% e 10% para as farinhas de abacaxi, maracujá e acerola, respectivamente. Os resultados indicaram que o sorbet enriquecido com a farinha composta pela mistura ternária é um produto com potencial para exploração, além de contribuir para a redução dos impactos ambientais gerados pelo grande e inadequado descarte de resíduos do processamento agroindustrial de frutas no ecossistema.

Palavras-chave: frutas tropicais amazônicas, resíduos agroindustriais, planejamento de misturas, análise sensorial.

INTRODUCTION

In recent years, the global production of fruits has increased due to a rise in consumption. Tropical fruits represent around 3% of worldwide agricultural production (FAO, 2023). Despite this low percentage, tropical fruits are of significant interest to the population because they are sources of bioactive

compounds, have noticeable sensory characteristics, and possess a high nutritional and functional capacity in the prevention and mitigation of degenerative diseases (ZUIN & RAMIN, 2018; ALVAREZ-RIVERA et al., 2021).

Traditionally, fruits are consumed in their natural state. With the increase in industrialization, waste generated by seeds, peels, and fruit leaves grows

exponentially, negatively impacting the environment. According to the latest data from the Food and Agriculture Organization of the United Nations (FAO), Brazil's tropical fruit production was 683.993 thousand tons in 2021, out of a gross per capita world total of approximately 25 million tons (FAO, 2023). This suggested that the total volume of agro-industrial waste in the country is high. Conversely, food industries are seeking strategies for the proper disposal of these wastes, aiming valorization based on their use in the development of new products with superior quality (GOEL et al., 2020).

Considering the growing demand from health-conscious and increasingly discerning consumers, the food industry has increased research and investment to make healthier food options available on the market, focusing on sustainability (PETKOVA et al., 2022). This trend extends to the ice cream segment (DUTTA et al., 2021), where flours developed from fruit residues eliminated in the processing cycle offer an alternative to include more nutrients in human food, as they are sources of bioactive compounds and dietary fibers (SANTOS et al., 2022).

Edible ices are prepared with emulsions of fats and proteins, classified according to their composition. Fruit ices, also known as sorbets, are obtained with the minimum addition of 3% pulp, juice, and/or fruit pieces and may also incorporate other ingredients (BRAZIL, 2022a), thus providing a non-dairy alternative to ice cream.

Regarding the global consumption of edible ice, New Zealand, the United States, Norway, Finland, and Denmark stand out, with 28.3, 20, 9.5, 9.5, and 6.5 liters, respectively (USDA, 2024). Meanwhile, in Brazil, the average market consumption was 4.99 liters, according to the latest estimates from the Brazilian Ice Cream Industries Association (ABIS, 2024).

The edible ice cream sector has been widely explored, with interest in innovative formulations that offer unique sensory, functional, and nutritional characteristics compared to products already on the market. Current products generally have lower levels of dietary fibers and natural antioxidants (GENOVESE et al., 2022), and can thus benefit from enrichment with fruit by-products, such as flour made from pulp residues.

Thus, this study established the mixing proportion of flours made from pineapple (PIWF), acerola (ACWF), and passion fruit (PFWF) residues. It aimed at the ternary composition of fruit waste flour (FWF) using the simplex-centroid design, applied in sorbet, and evaluated the sensory attributes

of flavor, texture, and overall impression. This aspect had not been explored until the present study. Additionally, the effects of adding the flours on the physicochemical and microbiological characteristics in sorbet formulation were also analyzed.

MATERIALS AND METHODS

Obtaining flours

Wastes from pineapple (*Ananas comosus* L. Merrill), acerola (*Malpighia emarginata*) and passion fruit (*Passiflora edulis* f. *flavicarpa*) pulp from Cooperativa Agrícola Mista de Tomé-Açú (CAMTA) located in the municipality of Tomé-Açú, Pará, Brazil, were dried in an oven with forced air circulation, at a temperature of 70°C for 180 minutes. Subsequently, they were crushed, ground (SL-30, Solab, Brazil) and sieved (16 mesh-1.19 mm) to obtain flours with uniform granulometry. They were vacuum packed in plastic packaging and stored in a dry and airy place at room temperature, for further analysis.

Experimental design for the composition of the proportion of flour usage

The simplex-centroid mixture designer was used to indicate the different proportions of PIWF, ACWF, and PFWF applied in nine edible ice cream formulations (Table 1). Figure 1 illustrates the vertices of the triangular model of the points corresponding to the ternary mixtures of the components, with restrictions on the maximum amounts (PIWF: 0.65, ACWF: 0.10, and PFWF: 0.35) for sensory attributes.

The independent variables were the concentrations of the flours used in the treatments and the responses of interest were the results of the acceptability test (MINIM, 2018) for the attributes flavor, texture, and global impression. The test was conducted with one hundred judges through a sensory evaluation, which had been previously submitted and approved by the Ethics Committee for Research with Human Beings of the Faculdade de Ensino Superior da Amazônia Reunida (FESAR) under CAAE N° 25855619.0.0000.8104. Table 1 shows the simplex-centroid mixture designer with the proportions of each mixture component.

Development of edible ice cream

For the preparation of the sorbet-type edible ice cream, açai pulp, glucose, sucrose, water and the developed FWF were used, with a total of nine formulations, using different concentrations of PIWF, ACWF and PFWF. The proportions of the components used are described in table 2.

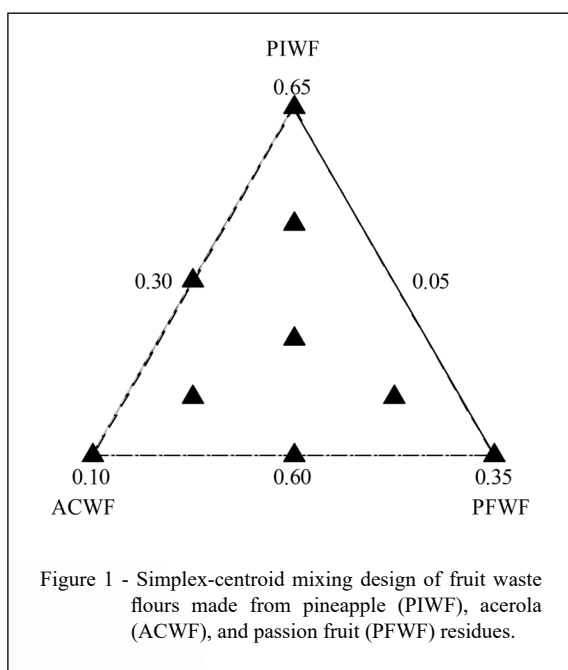
Table 1 - Composition of fruit waste flour mixtures from the simplex-centroid experimental design of the sensory attributes, flavor, texture, and global impression.

Treatments (factor levels)	Variables of mixtures						Sensory acceptance					
	Pseudocomponents			Real Proportions (%)			Flavor		Texture		Global	
	PIWF ¹	ACWF ¹	PFWF ¹	PIWF ²	ACWF ²	PFWF ²	MV ³	PV ⁴	MV ³	PV ⁴	MV ³	PV ⁴
1 (2)	0.625	0.075	0.300	62.500	7.500	30.000	5.83 ^a	5.94	5.64 ^a	5.70	6.40 ^a	6.35
2 (1)	0.600	0.050	0.350	60.000	5.000	35.000	6.37 ^{ab}	6.31	5.96 ^{ab}	5.9	6.76 ^{ab}	6.77
3 (0)	0.617	0.067	0.317	61.667	6.667	31.667	6.00 ^{abc}	6.14	5.61 ^a	5.72	6.62 ^{ab}	6.61
4 (-1)	0.608	0.058	0.333	60.833	5.833	33.333	6.54 ^{cd}	6.68	6.16 ^{ab}	6.22	6.73 ^{ab}	6.75
5 (-1)	0.608	0.083	0.308	60.833	8.333	30.833	6.44 ^{cd}	6.26	6.04 ^{ab}	5.89	6.51 ^a	6.68
6 (-1)	0.633	0.058	0.308	63.333	5.833	30.833	6.85 ^d	6.62	6.29 ^{ab}	6.16	6.73 ^{ab}	6.68
7 (2)	0.600	0.075	0.325	60.000	7.500	32.500	6.71 ^c	6.69	6.29 ^{ab}	6.29	6.69 ^{ab}	6.61
8 (1)	0.650	0.050	0.300	65.000	5.000	30.000	6.82 ^d	6.88	6.31 ^{ab}	6.34	6.78 ^{ab}	6.81
9 (1)	0.600	0.100	0.300	60.000	10.000	30.000	7.24 ^f	7.28	6.73 ^b	6.77	7.40 ^b	7.36

PIWF¹, ACWF¹, and PFWF¹: coded variables of the proportion of flour from pineapple (PIWF), acerola (ACWF), and passion fruit (PFWF) residues; PIWF², ACWF², and PFWF²: actual amount expressed in percentage of flour from pineapple (PIWF), acerola (ACWF), and passion fruit (PFWF) residues; MV³: measured values; PV⁴: predicted values; Means with equal letters in the same column indicate that there is no significant difference between the results ($P > 0.05$) by the Tukey test.

The sorbets were produced according to the methodology of LIU et al. (2018) with adaptations. The mixture formed was pasteurized at 85 °C for 30 seconds, followed by cooling. Subsequently, upon reaching a temperature between 30 °C and 40 °C, the previously pasteurized and cooled açai pulp was

added, performing a new homogenization. The FWF was incorporated into the syrup at the concentration established in table 2. Next, aeration and partial freezing of the sorbets were carried out in a producer (PSOFT-300, South Pole). The products were packed in 5 L plastic packages, previously identified, and stored in a freezer at an average temperature of -18 °C.



Physical-chemical evaluation

The pH, total acidity (TA), and soluble solids (°Brix) analyses were conducted in triplicates according to the protocols of the Association of Official Analytical Chemists (AOAC, 2005). The pH was determined using a pH meter (ST3100-F, OHAUS), and the TA was evaluated by titration with 0.1 M NaOH, with results expressed as a percentage of citric acid. The soluble solids content was determined using a digital refractometer (2WAJ, Biobrix), and the results were expressed in °Brix. Color parameters were measured with a portable colorimeter (CR-400, Konica Minolta) in a three-dimensional color diagram, with coordinates reading: L^* referring to brightness ranging from black (0) to white (+100), a^* ranging from green (-60) to red (+60) and b^* ranging from blue (-60) to yellow (+60).

Microbiological evaluation

Microbiological analyses for 45 °C thermotolerant coliforms, *Enterobacteriaceae*,

Table 2 - Components of the sorbet-type edible ice cream formulation.

Ingredients	Proportion (%)
Acid-free conventional pasteurized medium Açaí	66.00
Glucose	14.00
Sugar	5.00
Water	10.00
FWF	5.00

and *Salmonella* in the sorbets were conducted following the methodology of SILVA et al. (2010), before sensory evaluation, thus ensuring consumer safety. The results were compared to the parameters established in Normative Instruction (IN) No. 161, July 1, 2022, which provides microbiological standards for food, which converge with Collegiate Board Resolution (RDC) N° 724, of July 1, 2022 (BRASIL, 2022b).

Statistical analysis

The results obtained from the mixture planning (simplex-centroid) were analyzed using the statistical program Statistic 7.0 (STATSOFT INC, 2004), using analysis of variance and response surface methodology (RSM), having as independent variables the flours of the

residues of pineapple pulp (%), acerola (%) and passion fruit (%), and as variable responses the sensory attributes, flavor, texture, and global impression.

The other data obtained were subjected to analysis of variance (ANOVA) and means were compared using Tukey's test at a significance level of 5%. Results were expressed as mean \pm standard deviation.

RESULTS AND DISCUSSION

Experimental design of mixtures

The adequacy of the regression model (Table 3) was selected using the coefficients of determination (R^2) and analysis of the variance of the regression. The special cubic model showed the

Table 3 - Regression coefficients of analysis of variance of polynomial order models of sorbet ice cream formulations after the addition of FWF mixtures.

Models	Regression for mixtures				
	S	PRESS	R^2	R^2 (adjusted)	Lack of fit (P)
Flavor					
Linear	0.49	8.18	0.55	0.00	0.84
Quadratic	0.31	4.47	0.80	0.48	0.26
Special cubic	0.27	3.78	0.90	0.61	0.24
Texture					
Linear	0.38	2.36	0.93	0.00	0.73
Quadratic	0.26	2.96	0.78	0.41	0.28
Special cubic	0.17	3.29	0.96	0.74	0.18
Global impression					
Linear	0.29	1.86	0.15	0.00	0.60
Quadratic	0.12	1.52	0.92	0.81	0.13
Special cubic	0.11	1.32	0.96	0.82	0.06

S: standard deviation of the distance between data and fitted values; PRESS: predicted sum of squares; R^2 , R^2 (adjusted): determination coefficients; Lack of fit (P): model significance.

highest values for the R^2 and lack of fit closest in terms of confidence levels ($P > 0.05$) levels for the responses flavor, texture, and global impression.

By validating the proposed models for the experimental data, it was possible to write the mixture design function for the three variables (PIWF, ACWF, and PFWF) and the responses of interest, in this case, the sensory attributes (Eq.1-3). Which shows positive synergic effects only for the pure mixtures and for the binary mixture PIWF and PFWF.

$$\begin{aligned} \text{Flavor} = & 6.88 \text{ PI} + 7.28 \text{ AC} + 6.31 \text{ PF} - \\ & 4.58 \text{ PI}^* \text{ AC} + 6.35 \text{ PI}^* \text{ PF} - 41.23 \text{ AC}^* \text{ PF} + \\ & 22.63 \text{ PI}^* \text{ AC}^* \text{ PF} + 0 \end{aligned} \quad (1)$$

$$\begin{aligned} \text{Texture} = & 6.34 \text{ PI} + 6.77 \text{ AC} + 5.93 \text{ PF} - \\ & - 3.38 \text{ PI}^* \text{ AC} + 5.56 \text{ PI}^* \text{ PF} - \\ & 20.80 \text{ AC}^* \text{ PF} + 22.75 \text{ PI}^* \text{ AC}^* \text{ PF} + 0 \end{aligned} \quad (2)$$

$$\begin{aligned} \text{Global} = & 6.79 \text{ PI} + 7.37 \text{ AC} + 6.75 \text{ PF} - \\ & 2.79 \text{ PI}^* \text{ AC} + 3.01 \text{ PI}^* \text{ PF} - \\ & 1.64 \text{ AC}^* \text{ PF} + 7.49 \text{ PI}^* \text{ AC}^* \text{ PF} + 0 \end{aligned} \quad (3)$$

Where: $\text{PI}^* \text{ AC}$ = PIWF and ACWF interaction; $\text{PI}^* \text{ PF}$ = PIWF and PFWF interaction; $\text{AC}^* \text{ PF}$ = ACWF and PFWF interaction; $\text{PI}^* \text{ AC}^* \text{ PF}$ = PIWF, ACWF, and PFWF interaction.

Thus, the model can be used to predict the results according to DEJAEGHER & HEVDEN (2011), as models with R^2 equal to or above 0.90 and statistically significant regression coefficients can estimate experimental data with suitability for forecasts.

Evaluating the response surface and contour (Figure 2A) for the flavor attribute, it is possible to observe that, for ternary mixtures, as the ACWF concentration increased, the sensory scores increased, where the darkest regions are among the lowest proportions of PIWF and PFWF. Similar behavior can be inferred on the response surface and the texture contour diagram (Figure 2B) of the nine sorbets formulations, whereas the amount of ACWF gradually increased through the maximum percentage established, the values for the texture attribute also increased, demonstrating a directly proportional relationship. In relation to the global impression (Figure 2C), the area of acceptance of the proportion of PIWF and PFWF is larger, when compared to the other sensorial attributes analyzed, which implies higher scores for the formulations, showing greater acceptance.

In view of the surfaces and contours of triangular responses (Figure 2), it is evident that the enrichment of edible ice cream with the maximum

percentage established for ACWF positively interferes with sensory acceptance, which differs from the study by ISMAIL et al. (2020) with pomegranate peel powder for application in ice cream, which negatively affected the scores of sensory scores, however convergent to PIWF and PFWF, where at high concentrations they showed negative effects for all sensory attributes, showing lighter areas and less sensory acceptance.

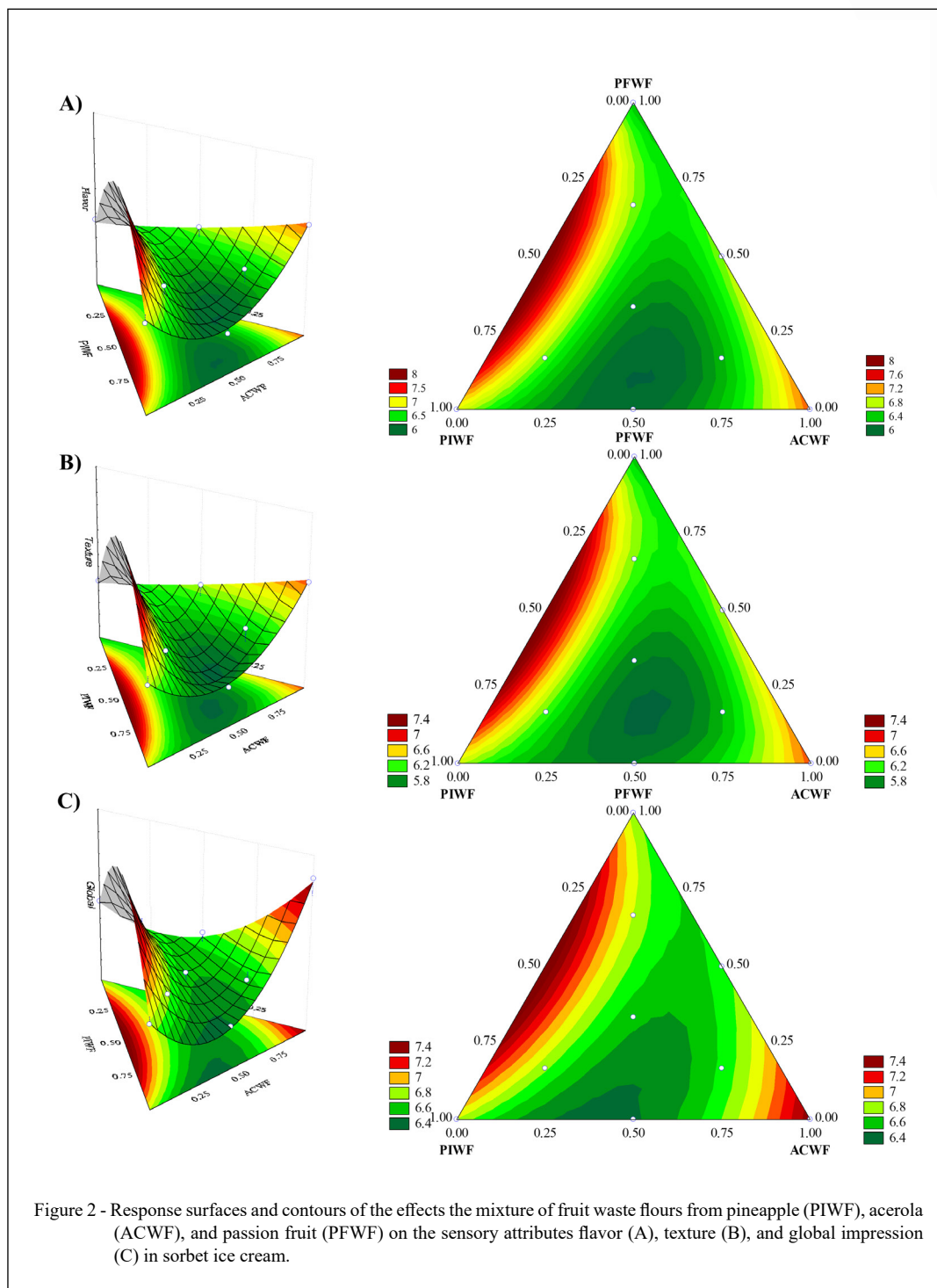
From the regions of interest of the triangular response and contour surfaces, figure 3 illustrates the estimated responses of the mixture of flours from the simplex-centroid designer, where it is noted that the estimated optimal area for the sensory attributes flavor, texture, and global impression corresponds to the amount of 60.98% of pineapple flour (PI), 7.32% of acerola (AC) and 31.71% of passion fruit (PF) for application in edible ice cream, with the nine formulations being the best approximates the predicted percentages since it contains the maximum proportion established by the ACWF, impact factor for acceptance.

Physical-chemical evaluation

Table 4 shows that the addition of different proportions of FWF caused a significant change in the physicochemical properties of the edible ice cream formulations. The pH of the edible ice cream formulations ranged from 4.17 to 5.01, however not significantly different ($P > 0.05$) between the samples of formulations 1-3 and 8-9, as these formulations had similar percentages of the three flours from fruit residues.

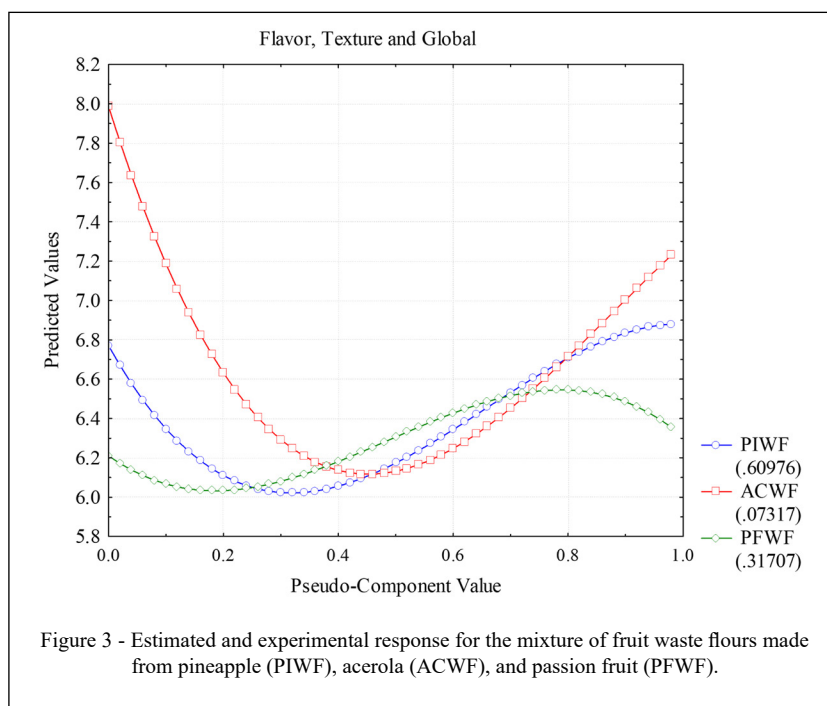
Regarding the titratable acidity content, the sorbet samples did not differ significantly ($P > 0.05$), demonstrating high acidity; the opposite behavior occurred with the pH values. Bearing in mind that pulp, juice, or pieces of fruit can influence this parameter, as they tend, in general, to present greater acidity than milk (the base ingredient for ice cream) (PALKA & SKOTNICKA, 2022). This fact can be justified due to the addition of flour formed with different concentrations of waste from pineapple, acerola, and passion fruit since there is only this change between edible ice cream formulas.

The soluble solids content showed a significant difference, except between samples 6 and 9 ($P > 0.05$), ranging from 17.00 to 24.40 °Brix. According to VANATHI & PALANI (2020), flavor is one of the most important attributes in edible ice cream and SYED et al. (2018) stated that high levels of soluble solids contribute to product acceptance, as they act positively on the texture and creaminess of ice cream and sorbet.



The composite flours added to the sorbets significantly influenced the color of the final products ($P \leq 0.05$). This is justified because each flour is composed of different types and proportions of fruit waste. The L^* values show low luminosity, and the a^* and b^*

parameters presented positive values, demonstrating more intense colors for the red and yellow hue, probably related to the addition of açaí pulp, which according to GHANDEHARI YAZDI et al. (2020) these color changes are closely dependent on the compounds added.



Microbiological evaluation

According to the values reported for the microbiological evaluation of edible ice cream (Table 5), the products were able to be safely consumed by judges in the sensory evaluation, as they revealed satisfactory hygienic-sanitary conditions. All results are within the parameters established in IN No. 161, of July 1, 2022 and in accordance with the terms of the RDC No. of July 1, 2022 (BRASIL, 2022b).

CONCLUSION

The simplex-centroid design showed, in ternary mixtures, ideal proportions of PIWF, ACWF and PFWF at 60.98%, 7.32%, and 31.71% for the sensory attributes flavor, texture, and global impression. It was evidenced that the addition of flours in edible ice cream showed a greater sensorial acceptance area of the formulations with a higher

Table 4 - Physicochemical properties of sorbet-type edible ice cream formulations enriched with FWF.

Formulations	pH	Acidity (g/100g)	°Brix	Color coordinates		
				L*	a*	b*
1	4.26 ± 0.01 ^{bcd}	0.19 ± 0.10 ^a	21.30 ± 0.01 ^f	24.08 ± 0.08 ^d	7.05 ± 0.03 ^c	3.54 ± 0.06 ^f
2	4.33 ± 0.05 ^b	0.19 ± 0.10 ^a	17.00 ± 0.06 ^g	25.43 ± 0.04 ^a	7.48 ± 0.03 ^d	4.63 ± 0.09 ^c
3	4.25 ± 0.01 ^{bcd}	0.19 ± 0.08 ^a	21.90 ± 0.13 ^{dc}	23.28 ± 0.02 ^f	6.26 ± 0.02 ^f	3.68 ± 0.06 ^f
4	4.29 ± 0.02 ^{bc}	0.19 ± 0.10 ^a	21.60 ± 0.09 ^{ef}	24.09 ± 0.04 ^d	6.17 ± 0.01 ^g	3.69 ± 0.04 ^f
5	5.01 ± 0.09 ^a	0.45 ± 0.01 ^a	22.00 ± 0.11 ^d	24.70 ± 0.05 ^b	7.78 ± 0.02 ^c	4.05 ± 0.07 ^{dc}
6	4.17 ± 0.01 ^{dc}	0.43 ± 0.02 ^a	24.40 ± 0.05 ^a	24.24 ± 0.02 ^c	7.78 ± 0.03 ^c	3.97 ± 0.07 ^c
7	4.12 ± 0.03 ^c	0.44 ± 0.05 ^a	22.90 ± 0.22 ^b	24.24 ± 0.02 ^c	7.48 ± 0.03 ^d	4.24 ± 0.05 ^d
8	4.21 ± 0.02 ^{cdc}	0.43 ± 0.12 ^a	22.50 ± 0.16 ^c	24.14 ± 0.02 ^{cd}	9.54 ± 0.02 ^a	6.78 ± 0.16 ^a
9	4.22 ± 0.01 ^{cdc}	0.42 ± 0.19 ^a	24.10 ± 0.14 ^a	23.85 ± 0.02 ^e	8.58 ± 0.03 ^b	5.41 ± 0.05 ^b

Values are expressed as mean ± standard deviation. Means with equal letters in the same column indicate that there is no significant difference between the results (P > 0.05) by the Tukey test.

Table 5 - Microbiological results of sorbet-type edible ice cream formulations.

Formulations	*Coliforms at 45 °C (UFC/g)	** <i>Salmonella</i>	** <i>Enterobacteriaceae</i> /g
1	<10	Absence	<10
2	<10	Absence	<10
3	<10	Absence	<10
4	<10	Absence	<10
5	<10	Absence	<10
6	<10	Absence	<10
7	<10	Absence	<10
8	<10	Absence	<10
9	<10	Absence	<10

*Not established by law. **Reference values according to IN N° 161 and RDC N° 724 (BRASIL, 2022B).

content of ACWF and lower content of PIWF and PFWF, for the specified maximum proportions.

The agro-industrial fruit residues can be an alternative for application in food, in view at the benefits in functional terms and adding value.

ACKNOWLEDGEMENTS

This study was funded by the call for projects for innovation and applied research projects (APIPA - n° 04/2019) of the Pró-Reitoria de Pesquisa, Pós-graduação e Inovação (PROPPG) at the Instituto Federal de Educação, Ciência e Tecnologia do Pará (IFPA), Castanhal, Brasil. And was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES), Brasil - Finance code 001.

DECLARATION OF CONFLICT OF INTEREST

We have no conflict of interest to declare.

AUTHORS' CONTRIBUTIONS

IRM: Conceptualization, formal analysis, and writing (original draft). JMS: Formal analysis, visualizations, and writing (review and editing). ACCB, TGMW, and LHSM: Methodology (supervision), investigation (software), and writing (review and editing). MRSPJ: Supervision, validation, and acquisition of finance. All authors critically revised the manuscript and approved the final version.

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