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Production, characterization and acceptability of different alcohol-based pineapple liqueurs

Produção, caracterização e aceitabilidade de licores de abacaxi elaborados com diferentes bases alcoólicas

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Abstract: This study carried out the assessment of physical, chemical parameters and the acceptability of pineapple (*Ananas comosus* L. Merri) liqueurs produced with "cachaça", double-distilled, flavored "cachaça"with pineapple peels and grain alcohol. Analyses encompass alcohol content, density, pH, soluble solids total, fixed and volatile acidity, dry residue and sensory analysis through acceptance/affective testing with 100 tasters using a nine-point hedonic scale to assess the attributes of color, taste, odor and overall impression; a test of purchase intention was also included. The mean alcohol content found for all the treatments was 25% v/v, which is in accordance with restrictions by the Brazilian legislation. The values found for physical and chemical analyses revealed significant difference among the treatments at 5% significance level. The liqueurs had good sensory acceptance with means above six; 49% of the tasters claimed probability of purchasing different alcohol-based pineapple liqueurs. The liqueurs produced exclusively with "cachaça"received the most satisfactory acceptance regarding the overall average for the analyzed items.

Key words: *Ananas comosus* L. Merri, exploitation of residues, sensory analysis,"cachaça"

Resumo: A avaliação dos parâmetros físico-químicos e a aceitabilidade de licores de abacaxi (*Ananas comosus* L. Merri), produzidos com diferentes bases alcóolicas: cachaça, cachaça bidestilada aromatizada com cascas de abacaxi e álcool de cereais, foram realizadas neste estudo. Realizaram-se análises de teor alcoólico, densidade, pH, sólidos solúveis, acidez total, fixa e volátil, resíduo seco e análise sensorial por meio do teste afetivo de aceitabilidade com 100 provadores utilizando escala hedônica de 9 pontos para avaliar os atributos cor, sabor, odor e impressão global, e teste de intenção de compra. O teor alcóolico médio encontrado para todos os tratamentos foi de 25% v/v, valor que se encontra dentro do limite da legislação brasileira. Todos os licores apresentaram boa aceitação sensorial, obtendo médias acima de 6 e 49% dos provadores alegaram que provavelmente comprariam o produto licor de abacaxi elaborado com diferentes bases alcoólicas. O licor preparado apenas com cachaça foi o mais aceito quanto à média global dos quesitos analisados.

Palavras-chave: *Ananas comosus* L. Merri, aproveitamento de resíduos, análise sensorial, cachaça

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INTRODUCTION

Most part of fruit production in Brazil is directed to "in natura" consumption (Lima, Nebra & Queiroz, 2000). Many Brazilian fruits have great market potential; therefore, studying processes that increase their value or their application in food products is of great interest (Ongaratto & Viotto, 2009). However, the market is increasingly demanding transformed products such as jams, candies, juice and liqueurs. Such increase is not only associated with adding value to fruits but it also generates major issues regarding the residues from industrial processes that could be exploited to develop different and innovative products derived from fruits with higher contents of several nutrients (Lima, Nebra & Queiroz, 2000).

The food industry has great interest in tropical fruits, especially for its peculiar flavor. Pineapple is a fruit much appreciated in various regions of the world, becoming one of the main products of the national fruit production. Despite the abundance of the cultivation of this fruit in Brazil, the industrial use is still low against the consumption of fresh fruit, requiring the search for alternatives to its use so as to tap the excessive crops, mainly by industry, to the manufacturing of non-traditional products, such as, for example, alcoholic beverages, depending on the sugar concentration, acidity and sensory characteristics (Araujo et al., 2009). Another feature of this fruit is the great waste production after processing. The remains of peels, crown and other parts are not used by the industry. The edible part represents from 22.5 to 35% of the fruit; the residue is discarded and especially the peels, contain considerable amount of adhered pulp which is disposed together with the the other parts (Prado et al., 2003)

From the technological and commercial point of view, the exploitation of pineapple either for "in natura" consumption and industrial use has increased considerably because of great variety of nutrients and possibilities of application in human diet; in addition, the fruit stimulates a burst of flavors. When processed, the pineapple provides products directed to several ends including the processing of beverages such as liqueur (Lima, Nebra & Queiroz, 2000).

Liqueurs have been reinvented regarding either the technological evolution and the diversity of flavors presenting increased purchases in Brazil with annual numbers around seven million liters, which represents 2.9% of the Brazilian market for alcoholic beverages dividing the consumers' attention between informal liqueurs and major national and international brands (ABRABE, 2014).

Statistical data indicate that the consumption of liqueurs in Brazil grows 5.1% per year, which encourages investments in this sector and increases opportunities for the market (Alveset al., 2010).

According to the Brazilian legislation, liqueurs have alcohol content between 15 and 54% in volume, at 20 ºC, and sugar percentage above 30 g L^{-1}, developed with potable ethyl alcohol of agricultural origin (grain alcohol), alcohol from batch distillation, or still alcoholic beverages added with extracts or substances derived from plants or animals, flavoring substances, dyeing, and other additives allowed through administrative act (Brasil, 1994).

Two are the basis to produce liqueurs: alcohol macerated fruit or distillation of fruit-based macerates. The quality relies not only on the proper mixture of ingredients, but especially on the process of preparation (Penha, 2006).

"Cachaça" is the typical and exclusive denomination of Brazilian sugarcane spirit produced in Brazil, with an alcohol content of 38-48% in volume at 20 ° C, obtained from the fermented mash of sugarcane with peculiar sensory characteristics (Brasil, 2009).

The history of "cachaça" is intertwined with the history of Brazil. It was the first distilled spirit in Latin America, discovered between the years 1534 and 1549 from the sugar production process. There arose the alcoholic spirit of genuine Brazilian cane, called "cachaça". Today is the third in the world, with about five thousand brands, thirty thousand producers in Brazil and annual volume of around 1.3 billion L per year. The Brazilian sugarcane spirit is considered the third most consumed distilled in the world and in the domestic market has 86% market share of the distilled beverages (spirits) in market. It is estimated that in Brazil there are 1.2 million "cachaça" sales points (IBRAC, 2011).

Distillation is a physical process that allows chemical separations, consists of the passage of the liquid phase of a substance or mixture, under heating, into a gaseous state, which then returns to the liquid state by cooling (Lima et al., 2001). It is a process that concentrates the alcohol and secondary compounds, largely defining the chemical composition of the spirit, providing quality to the final product (Oshiro&Maccari, 2005).

According to Lima (1964), the distillate is collected in three fractions to remove unwanted compounds. The head is the condensed liquid in the first minutes of distillation, corresponding to 0.7 to 1.0% of the total volume of the original content, presents alcohol content above 65 ° GL and must be discarded. The heart is the fraction that will be collected until the alcohol content in the recovery tank reaches the pre-set value. Generally, 1 to 1.5 °GL above the desired value for bottling. This fraction is contains about 80 to 90% of ethanol. This is the noble fraction, that means, the liquor to be commercialized. The tail, last fraction, can also be called weak water due to its low alcohol content.

The bi-distillation, as its own name says, is to conduct at least two successive distillations, which may be performed both in intermittent stills as in continuous columns. This process allows obtaining a superior quality spirit, with suitable alcoholic content and acidity, and pleasant flavor and aroma (Nogueira & Filho, 2005).

There are currently only a few brands of bi-distilled sugarcane spirit on the Brazilianmarket, sincemost of the industry commercializes it in the mono-distilled form (Silva et al., 2013).

Seeking good consumers' acceptance of fruit liqueurs such as pineapple, the possibility for value added and new flavors and the reuse of residues, the objective of this study was to develop different alcohol-based pineapple (*Ananas comosus* L. Merri) liqueurs – "cachaça", bi-distilled flavored "cachaça" and grain alcohol - to fully exploit the fruit and assess physical, chemical and sensory differences among the prepared formulations.

MATERIAL AND METHODS

Liqueurs production

Liqueurs were produced with fresh pineapples of cultivar "pérola", grain alcohol (Rioquímica, 93.8°), granulated sugar

(Cristal), which were purchased at the local market of Goiânia, Goiás (Brazil). Cambeba Ltda - located at the city of Alexânia, Goiás - provided non-aged organic "cachaça" (Mono-distilled sugarcane spirit) which was also used as a raw material do produce the liqueurs. This product has been certified by the International Federation of Organic Agriculture Movements, UnitedStates Department of Agriculture and by the Biodynamic Development Institute (Instituto Biodinâmico de Desenvolvimento), Brazil, obtaining the IBD-GO 012 certificate.

The liqueurs production was conducted in the Vegetable Laboratory of the Food Engineering Department at the Agronomy School, Federal University of Goiás (UFG).

Sanitation and pineapple pulp obtainment

The first stage of the process was the washing of the selected pineapples in running water using a brush to remove coarse dirt. Subsequently, the fruits were sanitized in chlorine solution (200 ppm) for 15 minutes and then manually peeled off with a stainless steel knife; the peels were stored in a regular refrigerator (5ºC) to be used when developing the double-distilled flavored "cachaça". The distillation was carried out on the same day the peels were obtained. The pulps were manually chopped and then grinded by using an industrial blender, Siemsen, Brusque-SC, to reach homogeneity.

Sugar syrups in the proportion of 1.2 Kg of granulated sugar for 1 L of filtered water were produced (1,2 : 1). The syrups were mildly heated only for sugar dissolution.

Three treatments were elaborated following the formulations as presented in Table 1.

Table 1. Formulations of pineapple liqueurs

Treatment	Pineapple (g)	Alcohol (mL)	Alcohol base	Sugar Syrup (mL)
A	100	225	Grain alcohol	175
B	100	225	Cachaça	175
C	100	225	Bi-distilled sugarcane spirit	175

Distillation

The bi-distilled sugarcane spirit was obtained from a mono-distilled spirit. Distillations were carried out in batches in a 12 L handcrafted copperstill with 1.5 L of "cachaça", 1.3 kg of chopped pineapple peels and 4.5 L of filtered water; the vapor temperature was controlled with an infrared thermometer (ICEL Manaus, TD-961) at 75 ºC. In order to separate the head, heart and tail fractions, thefirst fraction of distillate volume (100 mL) was discarded (head) anddistillation then continued to an alcoholic degree of 45 ± 1 ºGL (heart) (1 L), the remaining fraction (tail) was also discarded. All the distillations were carried out in triplicate.

Maturation

After production, the liqueurs were maintained in a controlled temperature room (20ºC), on vertical direction, inside cardboard boxes away from the light under manual agitation on a daily basis for 18 days. On the 19^{th}, liqueurs filtration was carried out in polypropylene filter to remove pineapples solid residues. At day 45^{th} of maturation, physical, chemical and sensory analyses were carried out.

Physical and Chemical Analyses

All physical, chemical analyses were carried out according to methodologies presented in official methods of analysis by the Association of Official Analytical Chemistry (AOAC) (AOAC, 2012). The analyses regarded dry residue; relative density; total, fixed and volatile acidity; pH; alcohol content; soluble solids and color.

Sensory Analysis

One hundred untrained tasters were submitted to an acceptance test with a nine-point hedonic scale (9 = "like extremely", 5 = "neither like or dislike" and 1 = "dislike extremely"). The tasters also answered a form of purchase intention with a scale of impressions encompassing from "definitely would buy the product" to "definitely would not buy the product" (Silva & Silva, 2012).

Statistical Analyses

An entirely random design was used with three treatments and three repetitions each. The results were submitted to analysis of variance (ANOVA) with subsequent comparison through Tukey Test at 5% significance level using the Statistical Analysis Software (SAS) (SAS Institute, 1997). All physical and chemical property analyses of the pineapple liqueurs were carried out in triplicate.

RESULTS AND DISCUSSION

Physical and chemical analyses

The alcohol content found in the analyzed samples after 45 days of maturation was 25% v/v, which indicates no alterations during this period and liqueurs within the restrictions established by Brazilian legislation - 15 to 54 % v/v (Brasil, 2009). According to Penhaet al. (2001), liqueurs with alcohol content of 18 ºGL receive the most satisfactory consumers' acceptance. For Catão et al. (2011), during the process of maturation, it is possible to occur oscillations in the alcohol content of a distillate due to room temperature and humidity, that is, the higher the humidity the higher the loss in alcohol. Conditions of low humidity lead to relatively varied concentrations because of the loss of water through the package pores. Such variation was not observed for the studied liqueurs.

Tables 1, 2 and 3 present the results obtained for the physical, chemical analyses of dry residue, color, acidity, soluble solids, pH and density conducted for the three treatments. The results pointed no difference at 5% significance level for mean dry residue and relative density for the three treatments (Table 2). Regarding soluble solids content, treatments 2 and 3 differed from treatment 1 (Table 2).

Table 2. Mean values for dry residue (g mL^{-1}), soluble solids (°Brix), pH and relative density (% v v^{-1}) for the three treatments of pineapple liqueurs

Treatment	Dry residue	Soluble solids	pH	Relative density
1	0.008^{c}	36.01 ^{b}	4.0155 ^{a}	0.969927 ^{b}
2	0.0485^{a}	38.51 ^{a}	4.0155 ^{a}	0.969977 ^{a}
3	0.0084^{b}	38.51 ^{a}	3.9844 ^{b}	0.966833 ^{c}

*ab Means followed by the same letter in the column do not differ according to Tukey test (p>0.05)
** (1) pineapple liqueur with grain alcohol, (2) pineapple liqueur with double-distilled flavored alcoholic beverage and (3) pineapple liqueur with "cachaça"

The pH values found for the treatments varied from 3.9 to 4.01. Those values can be associated with the pH value in the pineapple pulp – roughly 3.99 (Oliveiraet al., 2012). The value 3.9 found for the liqueur containing "cachaça" was statistically different from the other types of liqueur; the lowest value was 0.78%. When compared with other liqueurs, treatment 3 was 8% higher than 3.6 – value found by Viera et al.(2010), who produced *Myrciariadúbia* liqueur also using grain alcohol; and 7.4% higher than pH of 3.63 in the acerola (*Malpighia emarginata*) liqueur with pineapple developed by Nascimento et al. (2010).

Almeida et al.(2012) found values 16 to 25% higher than treatments 1, 2 and 3 (Table 2) for pH in tangerine peel liqueur, which varied from 4.66 to 4.9. While Nunes(2011) obtained 2.84 - 37% lower for tamarind liqueur in relation to our "cachaça"-based pineapple liqueur. However, according to Viera et al. (2010), this low pH is an important factor for restricting the development of pathogenic and deteriorating bacteria in addition to providing the stability of ascorbic acid – a vitamin that is more stable in acid pH (Nunes, 2011).

Mean values of total acidity for the three liqueurs (Table 3) varied from 0.004388 to 0.005767 mg acetic acid 100 g^{-1}, where the liqueur with grain alcohol differed statistically from the others in 23.91%. Studying tangerine peel liqueur, Almeida et al.(2012) found values 7.5% higher for all the treatments with different concentrations of peels and processing time; while Nunes (2011) found 1.48% titratable acidity for tamarind liqueur.

Table 3. Mean values for volatile, fixed and total acidity (mg of acetic acid100 g^{-1})

Treatment	Volatile acidity	Fixed acidity	Total acidity
1	0.006489^{a}	0.004137^{b}	0.005767^{a}
2	0.002007^{c}	0.004889^{a}	0.004388^{b}
3	0.002532^{b}	0.003761^{c}	0.004388^{b}

*ab Means followed by the same letter in the column do not differ according to Tukey test (p>0.05)
** (1) pineapple liqueur with grain alcohol, (2) pineapple liqueur with double-distilled flavored alcoholic beverage and (3) pineapple liqueur with "cachaça"

The variation of fixed acidity (Table 3) was between 0.003761 and 0.004889 mg acetic acid 100 g^{-1} with significant difference among the three treatments. However, there was higher variation for volatile acidity – between 0.0020 and 0.0064 mg acetic acid 100 g^{-1} (Table 3) – and the three treatments revealed significant difference. Acidity values may vary according to the period of liqueur maturation due to the alcohol basis composition since it provides the formation of volatile acids through the reaction of alcohols with acids and esters. The parameters can increase significantly as result of the reaction of ethanol oxidation (Caldeiraet al., 2010). Sample 1 – with grain alcohol – obtained a value three times higher for volatile acidity than the other samples, which can be explained through the composition of grain alcohol that promoted the formation of acids that are more volatile than other alcohols.

The values of soluble solids (Table 2) varied from 36.01 to 38.51, the former was found for grain alcohol-based liqueur that was statistically different from the others but identical to the value found by Nascimento et al. (2010), 36 ºBrix for acerola-pineapple liqueur. Viera et al. (2010) obtained a close value - 33 ºBrix – for *Myrciaria dúbia* liqueur. Almeida et al.(2012) with tangerine liqueur and Penha et al. (2001) with acerola pulp liqueur obtained values that are lower than what found in this study; the former found values between 23.31 and 24.06 ºBrix – values obtained according to the period of maturation – and the latter 25.47 ºBrix.

The value of soluble solids expressed in °Brix for the *Vangueria infausta* liqueur was 42, indicating that this fruit has considerable quantities of solid substances dissolved in the liqueur mostly composed of sugars (Mustafa & Munyemana, 2012), as well as the tamarind liqueur with 43 ºBrix found by Nunes (2011). According to the author, fruits maturation, sugar addition and even the remaining used in the respective liqueur production can influence this parameter (soluble solids). The high content of soluble solids in our pineapple liqueurs are believed to be attributed to the high content of sugar added when preparing the syrup – proportion of 1.2 kg of sugar for 1 L of water.

Regarding the relative density parameter (Table 2), there was statistical differences among the treatments averages. A possible explanation could be that the alcohol bases were not aged, since the ageing influences the enrichment of components with higher densities in beverages (Chaves, 2002). The average found for the three liqueurs was less than 15% of the value found by Nunes (2011) for the tamarind liqueur, and 16% of the value found by Almeida et al. (2012) for the tangerine peel liqueur. According to those authors, the density is proportional to the contents of soluble solids since the alcohol and sugar components are adjusted.

The values of color (Table 4) indicate that the liqueurs are slightly turbid and yellowish, which is expected due to the handmade production and the absence of clarification stage.

Table 4. Mean values of L*, a*, b*, Chrome and H°

Treatment	L*	a*	b*	C	H°
1	21.77 [b]	1.36 [a]	7.19 [c]	7.3175 [c]	79.2890 [c]
2	22.31 [a]	0.89 [b]	10.05 [a]	10.0893 [a]	84.9392 [b]
3	21.41 [c]	0.76 [c]	9.98 [b]	10.0089 [b]	85.6452 [a]

*abc Means followed by the same letter in the column do not differ according to Tukey test ($p>0.05$)
** (1) pineapple liqueur with grain alcohol, (2) pineapple liqueur with double-distilled flavored alcoholic beverage and (3) pineapple liqueur with "cachaça"

For parameters L*, a* and b*, the liqueurs are statistically different among the treatments at level 5%. Liqueur 1 presented stronger yellow color. We also studied the chrome parameters (C) and the hue-angle (H°). According to Bodart et al. (2008), the higher the chrome the higher the purity or intensity of the color; if the value is zero, it means that the color is grayish. Were found low values for this parameter (Table 4) pointing to a low density of color in the liqueurs - which indeed can be observed in all of the treatments – and light yellow liqueurs. Parameter H° indicate the color shade or the color per se; value 0° implies the red color, 90° the yellow color, 180° the green color and 270° the blue color (Bodart et al., 2008). The values obtained in degrees for the studied liqueurs varied from 79.2890° to 85.6452°, corroborating their yellowish color (Table 4).

Sensory analysis

A hundred both male and female untrained tasters took part in the acceptance test for different alcohol-based liqueurs - 43 women and 57 men, between 18 and 65 years old. The major percentage of tasters was between 18 and 30 years old (66%) followed by 31 and 42 (14%); 7% did not declare their ages.

Table 5 presents the mean values of the tasters' scores for color, taste, odor and overall acceptance. According to the scores for each attribute, there was no rejection to the samples, which is indicated by scores above five in all assessments.

Table 5. Mean values for attributes of color, taste, odor and overall impression obtained for sensory analysis

Treatment	Color	Taste	Odor	Overall impression
1	6.75	6.37	6.24	6.61
2	6.73	6.59	6.17	6.61
3	6.88	6.72	6.30	6.80

* (1) pineapple liqueur with grain alcohol, (2) pineapple liqueur with double-distilled, flavored alcoholic beverage and (3) pineapple liqueur with "cachaça"

According to the sensory assessment, the liqueurs presented average acceptance of 86%, percentage calculated in relation to the scores of the averages indicated by each assessor for the four attributes (color, odor, taste and overall impression) with values above five, in a scale from one to nine. Viera et al.(2010) developed a study on camu-camu liqueur and found average acceptance of 72.3% in a scale of 1 to 5; Andrade et al. (1997) studied the araçá-boi (*Eugenia stipitata*) liqueur with different formulations and verified acceptance of 96% for one the formulations with five-day maceration time.

The liqueurs produced with grain alcohol and double-distilled "cachaça" obtained the lowest percentages of acceptance - 88 and 80%, respectively. In turn, the liqueur produced exclusively with "cachaça" obtained the most satisfactory value, 91%, indicating a great level of acceptance. Rota and Faria(2009) aimed at obtaining a distillate using double-distillation and observed higher acceptance for double-distilled "cachaça" regarding the attributes of taste, overall impression and aftertaste when compared with mono-distilled "cachaça". Franco et al. (2009) obtained similar results revealing that the process of double-distillation promotes significant changes and improves the sensory quality of "cachaça", which was not proved regarding the double-distilled pineapple liqueurs with pineapple peels. Therefore, despite adding the odor of pineapple peels to the "cachaça", the product did not reach better acceptance. A possible explanation would be that when double distilled with pineapple peels, the "cachaça" incorporated their odor and lost its own peculiar odors when mono-distilled, which did not please the consumers.

According to Dutcosky (2011), the acceptance of a product regarding its sensory characteristics requires a minimum acceptance rate of 70%; in this study, the sensory assessment indicated that our liqueurs have good potential for consumption.

All the attributes assessed in the acceptance test – color, taste, odor and overall impression – indicated that the "cachaça"-based liqueur presented better acceptance and consequently higher averages (Table 5). Such acceptance can be attributed to the taste that the applied alcohol provided the liqueur with; the organic "cachaça" without the ageing process – a popular beverage for the public that has the habit of consuming alcoholic beverages – associates the "cachaça" taste and odor with the "cachaça" -based liqueur.

Treatments 1 and 2 presented close averages regarding color and overall impression. For parameter taste, the mean values were distinct; the "cachaça"-based liqueur obtained an average 1.9% higher than the others indicating better acceptance (Table 5).

Passos et al. (2013) studied a mixed liqueur containing carrot and orange and found values 8% higher for attributes taste (7.21) and overall impression (7.41) than the values found for our pineapple liqueurs (Table 5).

The color attribute presented the highest average followed by overall impression, taste and odor, respectively, indicating that the color of the pineapple liqueur was the most attractive attribute. Treatment 3 – light yellow shade – obtained better acceptance than the liqueurs of dark yellow shade; differences confirmed through color physical analysis.

In contrast, Feitoza et al. (2009) carried out a sensory analysis for their "cachaça"-based pineapple liqueur and obtained the highest average for overall aspect attribute followed by odor, color and taste, with values 7.43, 7.42, 7.32 and 7.3, respectively, 9, 17, 6 and 8% higher than our findings for "cachaça"-based liqueurs (Table 5). Although the values found by Feitoza et al. (2009) had been higher, we must consider the consumers' profile, an extremely important factor when assessing different types of beverages. Another associated factor that could have influenced those different results is the alcohol content; Penha et al. (2001) produced the acerola liqueur and found 18.31 °GL, liqueurs with alcohol content around 18 °GL have greater consumers' acceptance.

Regarding the test of purchase intention (Figure 1), 20% of the tasters declared that were not sure whether they would purchase the product, 7% claimed that they probably would not purchase the product and the remaining 5% stated that they certainly would not purchase the product. Nonetheless, 19% of the tasters declared that they certainly would purchase the product, and 49% probably would.

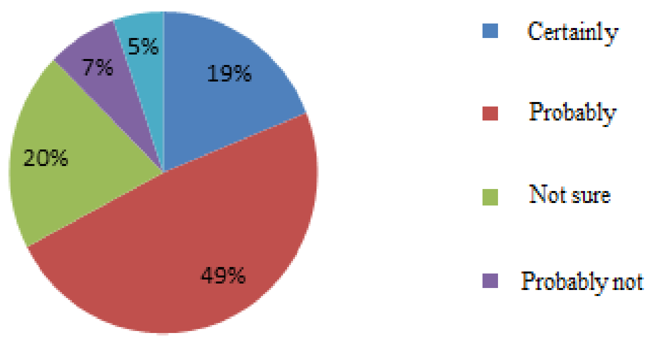

Figure 1. Test of purchase intention for experimental pineapple liqueurs

CONCLUSIONS

1. It is possible to produce liqueurs with different alcohol bases, value added and flavors by fully exploiting the fruit.
2. The use of different alcohol bases to produce pineapple liqueur added distinctive characteristics to the product.
3. The liqueurs received good consumers' acceptance, which indicates the viability of producing pineapple liqueur using any of the analyzed alcohol bases.

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