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Research Article

Research on the development of new alcoholic beverage like whisky from Vietnamese sticky rice

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Abstract

This study aimed to develop alcoholic beverage from Vietnamese sticky rice. The traditional famous technology of alcoholic beverage from cooked sticky rice in Nam Dinh province, Vietnam was researched and developed. The experiment has used raw materials as sticky rice variety DT21, treated soft water and yeast cake local production. After the solid and liquid fermentation processes of cooled cooked rice, the new type of alcoholic beverage like whisky was processed by the way to fractional distillation, removing the 3% primary and the 5% tail alcohol to obtain a 54% volumetric concentration middle distillate. Next, the whisky production technology was applied by storing the distillate in oak barrels and the addition of 4 $g.l^{-1}$ oak shavings type granular chip CSA 32, size 6-7 mm. After 12 months maturation in dry, clean, cool conditions, at a temperature of 20-30°C, and diluting with RO water, the finished alcoholic beverage with volumetric concentration 40% has amber color, typical aroma of whisky, very good sensory evaluation and meets Vietnam National Technical Regulation for alcoholic beverages.

Keywords

maturation, oak barrels, oak shavings, sticky rice, traditional alcoholic beverage, whisky

Abbreviations

QCVN – Vietnam National Technical Regulation; RO – Reverse osmosis; TCVN – Vietnam National Standard; v/v – volumetric concentration

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Introduction

Rice is an indispensable part of the Vietnamese people and created a rice-water civilization. The traditional technology of alcoholic beverage from cooked sticky rice has created many famous products with regional brands, including Hai Hau alcohol, Nam Dinh province. About 70% of market share of alcoholic drinks in Vietnam belongs to handmade by people themselves spirit from grain and other ingredients containing starch (rice, corn, sticky rice, cassava). Handmade alcoholic drinks from sticky rice are always highly appreciated by many users because of its natural, attractive aroma and rice sweet aftertaste.

However, unstable technology and lack of quality control makes a part of the produced drinks unsafe for consumers. There have been a series of announcements related to improving technological processes of this traditional drinks in Vietnam and many countries in the region such as Korea, Cambodia, and Nepal as well. The publications were related to selection of microbial strains, rice variety and quality of raw materials, application of enzyme preparations, distillation techniques, or sanitary conditions (Ho et al. 2023).

In fact, in addition to the traditional equipment system producing alcoholic beverage in households, some small cooperative-style facilities have also invested to improve capacity and hygienic conditions. After cleaning, rice is put in the trays and cooked in a heat-insulated steel cabinet, using electric energy or saturated steam. The fermentation process is carried out in stainless steel tanks, alcohol distillation is carried out in small-scale distillation tower. Some facilities have invested vacuum distillation equipment with reflux.

There is no unified technology for whisky production. Different countries apply different technological schemes and regimes depending on the characteristics of raw materials and available equipment systems. Applied around the world whisky technologies can be roughly divided into two groups – single malt, with using germinated barley malt, and grain, using and non-germinated raw materials. The technological process of whisky includes main stages such as wort processing, fermentation, distillation, maturation in oak barrels and product completion (Stewart et al. 2015; Marinov 2005).

According to Regulation of the European Parliament and of the Council of 17 April 2019, whisky is a spirit drinks produced by carrying out of distillation of a mash made from malted cereals, with or without whole grains of unmalted cereals, which has been saccharified by the diastase of the malt contained therein, with or without other natural enzymes, and fermented by the action of yeast. The legal name of 'whisky' or 'whiskey' may be supplemented by the term 'single malt' only if it has been distilled exclusively from malted barley at a single distillery (Regulation (EU) 2019/787). Except the single whisky, grain Scotch whisky is produced from 15-25% malted barley, 75-80% unmalted other cereal (barley, rye, oats). Pot Still Irish whisky is produced from minimum 30 % malted barley, minimum 30 % unmalted barley and other cereal. American grain whisky is made from 81.1% corn, 3.5% rye and 10.4% malt. The main ingredients in the production of Canadian whisky include rye, corn and barley. The maturation time for distillate in whisky production technology is minimum 3 years, while grain whisky does not require a longer storage period (Marinov 2005).

The use of oak wood in various forms has become an increasingly popular practice among winemakers. As a result of extraction, condensation and other processes, their chemical composition and sensory profile were modified. Further to the use of barrels, the application of alternative oak forms has become more and more popular (Yoncheva et al. 2024).

The research team has not discovered domestic or foreign publications on the development of amber-colored alcoholic beverage from sticky rice, produced by fractional distillation and stored with oak material. This experiment was conducted with purpose to develop a new alcoholic beverage according to the way to combine the traditional alcohol technology from sticky rice in Vietnam with European whisky production technology.

Materials and Methods

Materials

Sticky rice: Using DT21 variety - main material in the production of traditional sticky rice alcohol in Nam Dinh. The composition of raw sticky rice: Moisture 13.6%; Crude protein 7.85%; Crude fiber 0.27%; Starch 70.52%. Rice is straw yellow and still have thin layer bran on the grain, when the rice is milled, only the outer husk is removed. Cooked rice is glutinous with a characteristic aroma (Ho et al. 2023).

Yeast cake (starter culture): Using traditional yeast cake of Nam Dinh province.

Water: Using treated soft water obtained by reverse osmosis technology with pH 6.0 - 6.5.

Oak barrels: Made from Quercus robur, volume 30 L, Russian production.

Oak shavings type granular chip: CSA 32, size 6-7 mm, American production.

Experimental methods

The traditional technology of sticky rice alcohol was kept stable in all experiments. The experiments were conducted with a scale of 10 kg of sticky rice per batch.

Before each experiment, the production tools were washed with clean water and dried in the air. The ingredients such as sticky rice and yeast cake in conducted experiments are presented in Fig. 1.

a. Sticky rice **b**. Yeast cake

Figure 1: Raw materials for producing traditional sticky rice alcohol

Sticky rice was soaked in clean water with a temperature of 40-45℃ for 8-10 h so the rice to swell, creating better conditions for the cooking process. After that, the rice was washed with clean water and drained before cooking (Fig. 2).

a. Soaked rice **b**. Rice cooker using gas

Figure 2: Gelatinization of rice

The yeast cake was crushed into coarse powder. Cooked rice was evenly spread on a stainless-steel tray with dimensions 110×80×10 cm, the thickness of the rice layer is about 3 - 4 cm, let it cool (about 30-35℃) and mix with the yeast cake powder at the rate of 3% compared to raw sticky rice. (Fig. 3).

Figure 3. Mixing the yeast cake powder with cooled cooked rice

After mixing the yeast cake powder with cooled cooked rice, the barrel is closed but not too tightly to provide the oxygen for growing of the mould and yeast.

Rice solid state fermentation was performed for 3 - 4 d until rice has a slight aroma of alcohol, sweet taste and slightly spicy of alcohol. The solid fermentation process has ended when the surface temperature of the rice mass was increased and the height - decreased. Solid fermentation was carried out under aerobic conditions in a cool place in summer and kept warm in winter.

Liquor fermentation was continued for 7-8 days (depending on the ambient temperature) by adding purified water in the ratio of rice: water 1:3 and under anaerobic conditions until all the wort residue settles to the bottom of the plastic fermentation barrel of 50 L. This added water creates a liquid environment for the yeast to carry out the alcoholic fermentation process.

The distillation process was carried out in a batch distillation in pot still type distilator with a capacity of 50 L, using 10 L of pure water to rinse the fermentation barrel, dilute the boiled mash and to and create favorable conditions to separate volatile components in the pot.

In beginning of the second distillation, RO water was added to reduce the alcohol concentration to 20-22% v/v with purpose easily extracting the volatile components.

The obtained middle fraction was stored in 30 L oak barrels, adding 0, 2, and 4 g.l^{-1}oak shavings type granular chip in 3 conducted experiments. The control sample was performed by the same distillate and stored in clay jars. The volume of the distillate in every sample was 25 L (Fig. 4 and Fig. 5).

Figure 4. Oak barrel for maturation

Figure 5. Clay jar containing the control sample

After 6, 9, 12 months maturation in dry, clean, cool conditions, at a temperature of 20-30°C, the quality of the product was evaluated.

Analytical methods

The alcohol concentration was determined by the alcoholometer during distillation, by the pycnometer method during maturation.

Determination of aldehyde content in alcoholic beverage was conducted by titration method with iodine according to AOAC 972.09-1973 (2015).

Determination of ester content in alcoholic beverage was carried out by TCVN 11029:2015 (2015).

The color of the alcoholic beverage was measured according to EBC Method 9.6 (2018) at a wavelength of 430 nm on a UV-VIS spectrophotometer DR6000 - HACH.

After diluting with RO water, the control sample and maturated distillate to 40% v/v, the alcohol's sensory quality was evaluated with a committee from 8 tasting experts by TCVN 3217-79 (1979).

The composition of aromatic substances was determined by gas chromatography GC/ FID on a Clarus 500 Perkin Elmer instrument with Hydrogen gas generating equipment. Capillary column DB-ALC, 30m Length × 0,32 mm ID × 1,8µm df. Analysis program:

- Injector: 200°C, Spilite/ Spilitless: 5.3
- Oven 45°C/ 5min
- Ramp 1: 5°C. min^{-1} - 105°C/ 2min
- Ramp 2: 10°C/min - 185°C/ 9min
- Detector FID: 220°C, speed of air 350 ml. min^{-1}, H_2 - 40 ml. min^{-1}.

Alcohol concentration was measured on an Anton Paar. The concentrations of the volatile compounds were expressed of 100 % vol. alcohol as absolute anhydrous alcohol.

Experiments were conducted in two barrels, obtained data during maturation were processed by One-Way ANOVA, using software Minitab 18.

Results and Discussion

Traditional technology of sticky rice alcohol. The solid fermentation is the stage where the mould grows and develops on gelatinized cooked rice, synthesizes extracellular enzymes, that hydrolyze starch and other high-weight molecules into fermentable sugars and other nutrients for the yeast. During the fermentation, an amount of succulent water was produced and accompanied by effervescence due to the release of CO_2.

During liquid fermentation, ethanol and secondary products are formed, due to a sufficiently large amount of growth yeast biomass. When all the wort residue has settled to the bottom of the barrel, the fermented mash with ethanol concentration 10-12 v/v is taken to distillation.

Traditional yeast cake of Hai Hau district, Nam Dinh province, Vietnam in current experiment contains microbial strains of *Rhizopus microsporus, Saccharomycopsis fibuligera, Saccharomyces cerevisiae* (Ho et al. 2024). There are salient differences between the yeast strains employed for neutral spirits and those used in whisky, rum, and brandy fermentation (Spasov et al. 2023). In the presence of sugars, together with other essential nutrients such as amino acids, minerals and vitamins, *S. cerevisiae* will conduct fermentative metabolism to ethanol and carbon dioxide under anaerobic conditions. Yeasts will also produce numerous secondary metabolites which act as important beverage flavour congeners, including higher alcohols, esters, carbonyls and sulphur compounds. Therefore, yeasts are of vital importance in providing the alcohol content and the sensory profiles (Walker and Stewart 2016).

The distillation process in traditional technology was carried out by fractional method, which is similar to the technology of Scotch and Irish whisky. The first distillation was conducted by boiling the mixture in the pot on charcoal stove, volatile substances were passed on the cooled with water lid and condensed into obtained alcohol. The distillation process has ended when all the ethanol in the pot was recovered. The volume of the distillate after the first distillation was about 10 L with alcohol concentration 40-42% v/v. The sign to recognize the end of the distillation process is that the boiling temperature was reached about 102℃, and the alcohol concentration in the obtained latest distillate (measured with alcoholometer) was reduced to 12-14%.

After removing the primary and tail fraction, respectively containing 3 and 5% of the total alcohol degree after the first distillation, the alcohol concentration in middle fraction was reached about 54% v/v.

The middle fraction contains ethyl alcohol, aldehydes, esters, higher alcohols and other component with volatility close to that of ethanol, which have an important influence on the quality of finished alcoholic drinks.

Research on the developing of new alcoholic drink based on the whisky technology. The experimental plan includes the production of alcoholic drink from sticky rice by traditional Vietnams' technology in Hai Hau district, Nam Dinh province, following with fractional distillation and maturation of the middle distillate in oak barrels, adding oak shavings in experimental samples, the control sample - in clay jars.

Changes of the ethanol concentration during maturation. The changes of the ethanol concentration during experimentation with different ratio of oak shavings type granular chip was presented in Table 1.

Table 1. Changes of the ethanol concentration during maturation, % v/v

Maturation time, months	Control sample	Sample 0 g.l^{-1} shavings	Sample 2 g.l^{-1} shavings	Sample 4 g.l^{-1} shavings
0	54.05^{a}	54.05^{a}	54.05^{a}	54.05^{a}
6	53.70^{b}	53.40^{b}	53.47^{b}	53.45^{b}
9	53.52^{c}	53.20^{c}	53.22^{c}	53.18^{c}
12	53.45^{d}	53.06^{d}	53.08^{d}	53.02^{d}

*Means with the same letter in the same column are not significantly different from each other *($p<0.05$).*

After 12 months maturation in clay jars, the control sample had a decrease in ethanol concentration of about 0.5% v/v, while experimental samples had a decrease about 1.0%, which can be hypothesized to be due to the difference in material of clay jars and oak barrels with differently affecting on the evaporation of ethanol. The results of experiments showed that alcohol concentration decreased over time during storage, which was consistent with other publications (Marinov 2005).

Changes of the aldehyde's concentration during maturation (as acetaldehyde). Change of the aldehydes, measured as acetaldehyde during experimentation was illustrated in Table 2.

Table 2. Changes of the aldehyde's concentration during storage

Maturation time, months	Control sample	Sample 0 g.l^{-1} shavings	Sample 2 g.l^{-1} shavings	Sample 4 g.l^{-1} shavings
0	49.55^{d}	49.55^{d}	49.55^{d}	49.55^{d}
6	55.25^{c}	60.45^{c}	61.23^{c}	60.89^{c}
9	58.32^{b}	64.55^{b}	66.44^{b}	64.78^{b}
12	63.11^{a}	69.33^{a}	71.07^{a}	72.88^{a}

*Means with the same letter in the same column are not significantly different from each other *($p<0.05$).*

The results in the Table 2 showed that the concentration of aldehydes was increased by the maturation time, the reason was supposed to be due to the oxidation reaction of the alcohol and increased the concentration of the corresponding aldehydes. In addition to the fatty aldehydes formed during fermentation and the cyclic aldehydes obtained during distillation, some aromatic aldehydes are formed from the hydrolysis of lignin during storage (Marinov 2005).

Compared to the control sample, the increase of the aldehydes in the experimental samples was higher, which can be attributed to the higher intensity of the oxidation reaction due to oxygen permeating through the wooden barrel wall.

Changes of the ester's concentration during maturation (as ethyl acetate). Change of the esters in middle distillate, measured as ethyl acetate during experimentation was presented in Table 3.

Table 3. Changes of the ester concentration during maturation

Maturation time, months	Control sample	Sample 0 $g.l^{-1}$ shavings	Sample 2 $g.l^{-1}$ shavings	Sample 4 $g.l^{-1}$ shavings
0	270.5^{d}	270.5^{d}	270.5^{d}	270.5^{d}
6	282.4^{c}	295.4^{c}	298.6^{c}	300.4^{c}
9	290.5^{ab}	314.8^{b}	320.6^{b}	338.8^{b}
12	296.4^{a}	325.3^{a}	340.2^{a}	365.7^{a}

*Means with the same letter in the same column are not significantly different from each other *($p<0.05$)*.

The results of Table 3 showed that esterification processes increased the content of esters during maturation in the control and all other samples. This process played an important role in forming the product's aroma.

Esters in whisky mainly include compounds of ethanol and short chain fatty acids, mainly ethyl acetate, and compounds of ethanol with saturated and unsaturated fatty acids. In addition, the extraction of some components from oak material also creates the characteristic aroma of whisky. Not only the total quantity of ester content effect on the product quality, but more importantly - the composition and quantity of newly formed compounds (Marinov 2005).

Changes of the color intensity during maturation. Whisky has the basic characteristic of being amber in color, due to maturation in oak material. The color intensity of the distillate was changed during experimentation and shown in Fig. 6 and Table 4. During the first 6-9 months, the extraction process of colorants was stronger and the color intensity increased faster. After that, it was observed that the color intensity increased more slowly in experimental samples. The color intensity has not changed significantly in the control sample due to the absence of coloring agents.

Table 4. Changes of the color intensity during maturation, EBC

Maturation time, months	Control sample	Sample 0 $g.l^{-1}$ shavings	Sample 2 $g.l^{-1}$ shavings	Sample 4 $g.l^{-1}$ shavings
0	0.02^{b}	0.02^{d}	0.02^{d}	0.02^{d}
6	0.05^{ab}	2.65^{c}	3.68^{c}	4.30^{c}
9	0.08^{a}	3.85^{b}	5.10^{b}	6.25^{b}
12	0.07^{ab}	4.39^{a}	5.61^{a}	7.05^{a}

*Means with the same letter in the same column are not significantly different from each other *($p<0.05$)*.

Fig. 6 showed the color of the product after 12 months of maturation. Color intensity changes can be observed with the normal human eyes.

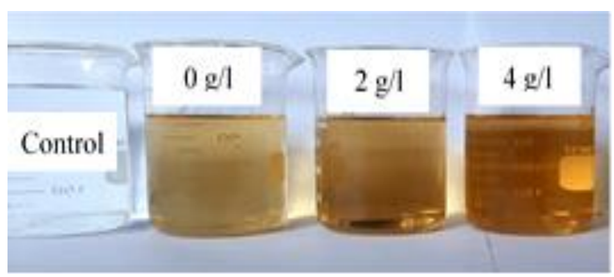

Figure 6. Color intensity after 12 months of maturation

From the oak material, many components as tannin, hemicellulose, lignin, terpenes, pectin were extracted into the matured distillate, after converting to the dissolved substances with low molecular weight, the smell, taste, and color of the whisky are formed (Marinov 2005).

Sensory evaluation of the finished product. The alcoholic beverage, received after diluting the control and maturated samples to 40% v/v concentration with RO water was not been coloured, sweetened and not been added any alcohol or flavouring substances.

Performing by the TCVN 3217-79 (1979), the results of sensory evaluation were presented in Table 5. The alcoholic beverage, received by maturation after 12 months had the highest sensory score, reaching the good grade, 16.28 and 16.56 in experiment with 2 and 4 $g.l^{-1}$ oak shavings, resp.

Table 5. Sensory scores of alcoholic beverage samples

Matura-tion time, months	Control sample	Sample 0 $g.l^{-1}$ shavings	Sample 2 $g.l^{-1}$ shavings	Sample 4 $g.l^{-1}$ shavings
6	14.43	14.87	15.12	15.54
9	14.91	15.36	15.76	15.91
12	15.35	15.84	16.28	16.56

During maturation process were occurred physical and chemical changes, new compounds are formed that determined the smell and taste of the finished product. The hydrolysis of hemicellulose from oak material formed different sugars that helped soften the taste (Marinov 2005).

The final product with 4 $g.l^{-1}$ added oak shavings has an amber color, the typical flavor of traditional sticky rice alcohol was significantly reduced, replaced by the typical flavor and aroma of whisky (Fig. 7).

Figure 7. Finished alcohol from sticky rice and whisky technology

The good quality of the finished drink can be hypothesized to be due to storage in oak barrels and the added ratio oak shavings. The presence of characteristic oak-wood phenols such as coniferaldehyde, sinapinaldehyde, syringaldehyde (aromatic aldehydes), and scopoletin (a coumarin) were found in the white wines Picapoll and Chardonnay fermented in barrels, but were not detected in the same wines fermented in stainless steel vats (Ibern-Gomez et al. 2001).

Determination of the chemical composition of finished product. Some components of the obtained alcoholic beverage in the sample with 4 $g.l^{-1}$ added oak shavings and maturated in 12 months were analyzed by gas chromatography, converted to absolute anhydrous alcohol and detailed in Table 6.

Table 6. Composition of finished drink, referred to absolute anhydrous alcohol

№	Compounds	Value, $g.l^{-1}$
1	Acetaldehyde	125.7
2	Methanol	22.6
3	n-Propanol	468.8
4	Ethyl acetate	650.5
5	Methyl propionate	6.3
6	Iso-butanol	450.5
7	Methyl iso-valerate	0.3
8	3-Methyl butanol	755.3
9	2-Methyl butanol	184.6
10	Ethyl butyrate	0.4
11	Iso-amyl acetate	2.6
12	Furfuraldehyde	2.9
13	Ethyl octanoate	0.8
14	Phenethyl alcohol	6.8
15	Phenethyl acetate	0.4
16	Ethyl decanoate	2.2

The multicomposition of chemical compounds was the reason for the good flavor and sensory value of the product. The Table 6 made the impression that quantity of the ethyl acetate and other higher alcohol content, referred to absolute anhydrous alcohol, was quite high. These data are considered to corresponding the sensory quality of the finished alcoholic beverage. Whisky consists of many trace elements coming from the raw materials used in its fermentation, distillation and maturation processes. These ingredients assure the exceptional organoleptic characteristics of the beverage (Pawlaczyk et al. 2019). The concentration of methanol is 22.6 $mg.l^{-1}$, significantly lower than the requirements according to (QCVN 6-3:2010/BYT 2010).

Conclusions

Originating from the high-quality traditional alcoholic beverage of Hai Hau district, Nam Dinh province, Vietnam, the project used fractional distillation and applied whisky technology to develop new drink by maturation of the middle distillate with an alcohol concentration of 54% v/v in oak barrels, adding of 4 $g.l^{-1}$oak shavings, in 12 months. After diluting with RO water to 40% v/v concentration, the finished alcoholic beverage has high quality, amber color, characteristic aroma and flavor of whisky, highly appreciated by many experts and tasters.

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