Title: PERSIMMON VINEGAR POWDER AND PROCESS FOR PREPARING THE SAME

Abstract: This invention is a method of making persimmon vinegar powder which involves the technique of adding extracts from persimmon leaves to concentrated persimmon vinegar and spray-drying the liquid. This technique is the best method of manufacturing persimmon vinegar powder, for it enables the control of the scent and taste so as to make it easy to drink, and also reduces the loss of nutrients during the curing processes. The persimmon vinegar powder made, using this invention, goes through the following processes: getting persimmon vinegar concentration through concentrating persimmon vinegar with a vacuum concentrator to 12~18% Bx; using the weight of the concentrated persimmon vinegar as a standard, adding and stirring in persimmon leaf extract 15~30% and cyclo-dextrin 5~9% to the persimmon vinegar concentrate; again, using the weight of the persimmon vinegar concentrate as a standard, adding and dissolving lactose 1~5%, malt-o-dextrin 25~30%, gum homogenized liquid and producing the powder. According to this invention, the acidity of the persimmon vinegar is eliminated and the unique taste and scent of persimmon leaves are added to produce a persimmon powder that appeals to the senses, which is also standardized through using concentrated liquid and is economic.
patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.
Persimmon Vinegar Powder and Process for Preparing the Same

[Simple explanation of the figures]

Figure 1 is graph that show the changes in the concentration of the persimmon vinegar concentrate

Figure 2 shows the process of the manufacture of the persimmon leaf extract visually

Figure 3 shows the process of the manufacture of the persimmon vinegar powder visually

[Purpose of the invention]

[The technical area which includes this invention and pre-existing techniques]

This invention is about persimmon vinegar powder and the process of its preparation.

More specifically, it is about a method of producing persimmon vinegar powder through which extracts from persimmon leaves are added to concentrated persimmon vinegar and spary-dried. This technique is the best method of manufacturing persimmon vinegar powder, reduces the loss of nutrients during the curing process.

Persimmon (Diospyros kaki) is an alkali fruit which is nutritious and contain 11-15% sugar such as fructose and glucose, about 20-50mg/100g of Vitamin C (which is about 5-12 times more than an apple), and more minerals than an apple.

Not only that, it is full of Vitamin A and tannic acid (which is produces the astringent tastes). Tannic acid works to heal diarrhea or stomachaches, and is known also to lower blood pressure without affecting the heart rate, so that there are many researches going on about persimmon as a medicine, as well as a fruit.

The national produce of persimmon was 155,111 ton (in 1992), but has been increasing yearly to about 233,570 ton in 1997. There in also an increase in the areas, which produce persimmon. The returns form producing and selling persimmon are greater than that of apples or other fruits, and so, significant increase in production area and amount is expected. In order to match such expectations, there is a need for the development of methods in using persimmon as a food and also as a medicine.

On the other hand, making persimmon into vinegar and consuming the product has been used as a way to maximize the effect of the beneficial nutrients in persimmon for along time; in a book called "San-Lim Kyung-jae" of the late Cho-sun Dynasty, persimmon vinegar is written as a new fruit vinegar. Vinegar is widely used all over the world as a seasoning, and since the TCA cycle theory was announced by krebs from England and kipadan from US in 1963, its constituents have been known to have important influences on the metabolism of the human body.
There are many organic acids that are involved in the TCA cycle such as acetic acid in vinegar and vinegar is known to be helpful in recovering from tiredness and making the body become alkali through helping with the TCA cycle while not storing lactic acid. Various fruits and grains have been used as the ingredients of vinegar, and persimmon vinegar, which is made through fermenting persimmons, has a better flavor that other vinegars such as apple vinegar or brown rice vinegar.

Persimmon vinegar also contains a large amount of tannin, which is a polyphenol compound, and has the effect of strengthening blood pressure and blood vessels. It also contains acetic acid -- its main constituent -- and many organic acids that are involved in the TCA cycle, which makes it good for recovering from stress resulting from extreme exercise or work, and also increases appetite and prevents geriatric diseases.

Despite the fact that persimmon has been used traditionally as a fermenting ingredient to make vinegar for a long time, there are only a few studies done about persimmons and persimmon vinegar. Only today, as the interest in health food increased, studies on the effect of persimmon consumption in the human body and standardized methods for making persimmon vinegar have started. Sugahara did a research on the preference for persimmon beverages (resource: Sugahara, T., et al., on the preference for Kaki beverage. Nippon Shoukoubir kgoyo Gakkaish, 33(4), 281–284, 1987). Nakasima et al produced persimmon vinegar using actobacter (which they separated and extracted during the fermentation of persimmon) and studied the change in the constituents of persimmon vinegar as result. (resource: Nakasima, W., et al., Changes in the composition of persimmon vinegar induced by Acetobacter spp. isolated from 'Sanja' persimmon fruits during the fermentation. Nippon Shokubin Kogyo Gakkaish, 34(12), 818-825, 1987). Among Japanese patent rights regarding persimmon vinegar are method of making alcohol beverages using persimmons (resource: Open Japanese patent right No, 56-25104) and method of making persimmon vinegar that reduces the concentration rate of alcohol in blood (Open Japanese patent rights No, 61 22403).

Within Korea, there has been a report that persimmon vinegar of an acidity of 5% was made by separating superior bacteria that produce acetic acid and cultivating these bacteria. (resource: Won-suk Cha, Jeon-Hye Park, Jin Gyu Kim: Study on the production of persimmon vinegar. Theses written in Sang-Ju Agricultural Specialized University, 20, 29–32, 1980) also, there was a research done through separating and identifying the major bacteria involved in the fermentation of persimmon vinegar and finding out the amount of acetic acid during a developmental study on fast-produced persimmon vinegar.

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(resource: Myung-Chan Kim et al, Production of vinegar using persimmon, Korea industrial Bacteriology Magazine, 8(2), 103-111, 1990) There has been continuous studies in the manufacture of persimmon vinegar such as, Study on the Manufacture of Persimmon Vinegar using Saccharomyces cerevisiae FWKS 260 and Acetobacter acetic (resource: Chong-Yun Won et al. Study on the development of fast-produce vinegar using persimmon, Korean Nutritional Food Academic Committee Fall Theses Presentations, p. 55, 1994) and study on the Production of Persimmon Vinegar using Persimmons of Low-Quality due to Low Temperature Storage (resource: Jung-Hwa Hong et al. The production of vinegar using persimmons of low quality due to low temperature storage, Korea Nutritional Food Academic Committee Magazine, 25(1), 123-128, 1995). As Korean patent rights, there are: method of making persimmon vinegar (resource: Republic of Korea (ROK) patent Right Announcement No. 93-008190, No. 93-151732, and ROK patent Right Application No. 98-002239), method of producing crude drug ingredients using persimmon vinegar (resource: Open ROK patent Rights No. 96-028815), method of making fast-produce persimmon vinegar (resource: ROK Patent Right Announcement No. 98-149279), method of making beverage constituents containing persimmon vinegar (resource: Open Korean Patent Rights No. 97-052221), and method of making persimmon vinegar using fertilization dregs of white silky fowl. However, all previous studies were limited to the production of persimmon vinegar or the simple application of already-produced persimmon vinegar to beverages, and failed to extend to the study on the technique of processing liquid persimmon vinegar into a more conveniently edible form.

Persimmon vinegar is used as an acidic seasoning for cooking in Korea, but another major use is to consume it directly as a health food. Generally, it is diluted and consumed with oysters or such as a health food, but even though it is extremely diluted, the acidic taste and scent of persimmon vinegar makes it very difficult and painful to drink, and because it is in liquid form, it is inconvenient to carry around and difficult to use as an ingredient for cooking. Thus, a method to improve these disadvantages was in demand. In short, in a way to overcome the extremely strong taste and smell of persimmon vinegar, its inadequacy in direct consumption, the difficulty in handling and mixing with other foods, and other limits in use and application because of its liquid form, the method of making persimmon vinegar into a powder form has been attempted.

So, as a technique of powderizing persimmon vinegar, a method of trapping persimmon vinegar molecules using dextrin's molecule trapping quality, trapping persimmon vinegar's scent using gum, and then, through a drying process, obtaining powder persimmon vinegar was announced as No. 98-000188 in the Open ROK Patent Rights. However, this method of making powder persimmon vinegar was just an application of the general method of powderizing liquid, and using this method, there is a difficulty in adequately lessening the acidic taste of persimmon vinegar and capturing its unique flavor.
Especially, because the method involves the pre-processing of persimmon vinegar or maturing the vinegar in a sealed container, the manufacturing process was complicated and took a long time, which made it inadequate for the powderization of persimmon vinegar. Also, because this method used the original liquid of persimmon vinegar, the volume of the container used in the process had to be very large, and not only that, the resulting powder contained a low rate of persimmon vinegar constituents and had unstabilized concentration rate so as to need a separate standardizing process.

Thus, the method desperately needed important. Also, when consuming the resulting powder, the flavor of the persimmon vinegar wasn't adequately masked, so the problems that existed when the liquid persimmon vinegar was directly consumed still remained.

Therefore, in developing a method to powderize persimmon vinegar, a process that allowed the control over the smell and taste of persimmon vinegar to make it adequate in direct consummation, and also reduce the loss of nutritional constituents had to be found. This process also had to be standardized and simple so that it was possible to produce high quality persimmon vinegar powder conveniently.

[The technical task of this invention]

We, the inventors, put an effort in powdering persimmon vinegar to make it easy to consume. As result, we found out that when mixing persimmon leaf extract and other ingredients with persimmon vinegar concentrate and then powderizing the mixture, the acidic taste of persimmon vinegar can be eliminated and the unique flavor of persimmon leave is added to produce a persimmon vinegar powder that appeals to the senses, which is also standardized through using concentrated liquid and is economic. And so, we where able to finish this invention.

In short, the main purpose of this invention is present a method to produce a concentrate from persimmon vinegar, and to add persimmon leaf extracts and other ingredients to this concentrate and after putting this through stirring and homogenizing processes, spray-dry and obtaining powder persimmon vinegar.

Another purpose of this invention is to provide powdered persimmon vinegar made through such methods.

[The composition of the invention and its functions]

The persimmon vinegar powder made through this invention, goes through the following processes: getting persimmon vinegar concentration through concentrating persimmon vinegar with a vacuum concentrator to 12-18° BX using the weight of the concentrated persimmon vinegar as a standard, adding and stirring in persimmon leaf extract 15-30% and cyclo-dextrin...
5~9% to the persimmon vinegar concentrate: again, using the weight of the persimmon vinegar concentrate as a standard, adding and dissolving lactose 1~5%, malto-dextrin 25~30%, gum 5~9% in the stirred liquid, and afterwards, homogenizing the liquid: spray-drying this homogenized liquid and producing the powder.

Below, a more detailed explanation about the manufacturing procedure of persimmon vinegar powder through this invention is presented in sections.

First Process: Obtaining the persimmon vinegar concentrate

Obtain persimmon vinegar concentration through concentrating persimmon vinegar with a vacuum concentrator to 12~18% BX: in this process, any persimmon vinegar that used persimmons as its main ingredient can be used as is homemade or commercially bought. For the purpose of standardizing the process, controlling the increase in volume during the process, and providing the best powderizing condition in which the active constituents of the persimmon vinegar can be maintained, the persimmon vinegar liquid must be concentrated to 12~18% BX, or more preferably, 14~16% BX. We, the inventors, confirmed that the best concentrated rate of persimmon vinegar to make into powder form is approximately 15% BX, through finding out the best concentrated rate of persimmon vinegar for maintaining its active constituents by analyzing the color, viscosity, pII, acidity, and organic acid.

Second Process: stirring

Using the weight of the concentrated persimmon vinegar as a standard, add and stir in persimmon leaf extract 15~30% and cyclo-dextrin 5~9% to the persimmon vinegar concentrate: in this process, the persimmon leaf extract is added to alleviate the acidic taste of persimmon vinegar, and also to enhance its unique flavor. Persimmon leaf extract is made through the following processes: washing the persimmon leaves and steaming it for 1~10 minutes at 100~120°C, then drying it for 1~2 hours using a 35~50°C hot wind: adding 8~12 liters of water per 1 kg of dried persimmon leaves and boiling for 10~30 minutes at 90~100°C, then filtering with a compressing filter and obtaining just the liquid: and using a vacuum concentrator, concentrating this liquid to 12~18% BX. Using persimmon leaf extract that has the same concentration rate with the persimmon vinegar using concentrate enables the strict standardization of the process and the production of high quality powder when spray-dried. Also, cyclo-dextrin is added to capture the taste and smell of persimmon vinegar, and among cyclo-dextrin, it is best to use cyclo-dextrin 7 which captures the smell most efficiently. The stirring in of the mixture of persimmon leaf extract and cyclo-dextrin is done for 30~180 minutes at 0~20°C, and a brown liquid mixture can be obtained after this process.
Third Process: Homeogenizing

When the mixture is sufficiently stirred through the second process, using the weight of the persimmon vinegar concentrate as a standard, add and dissolve lactose 1~5%, malto-dextrin 25~35%, gum 5~9% in the stirred lique, and afterwards, homogenize the liquid: in this process, lactose is added to soften the acidic taste of persimmon vinegar, and the biting sour taste lessens significantly due to the addition of lactose. Also adding malto-dextrin makes it easier to powderize the liquid, and one or two of the following ingredients -- black carrageenan, agar, alginate, guar, gum, arabic gum, roost bean gum, xathan gum, pectin, and carboxymethyl cellulose -- are added according to weight in order to prevent the absorption of humidity when powderized, and also to make the powder look shiny. It is recommended to use arabic gum. Homogenizer for 2~10 minutes at 0~20℃, making sure that the mixture is mixed thoroughly. Thus, a homogenized liquid is obtained.

Fourth Process: Spray-drying

The homogenized liquid obtained through the third process is spray-dried using a spray-dryer and made into powder. The spray-drying is done using a spray-dryer, at 150~180℃ in-air, 80~100℃ outlet air, and 15000~25000rpm speed of the atomizer. The homogenized liquid is added to the dryer at a rate of 20~40ml/min. Through this spray-drying process, a brown, flowing, high-quality persimmon vinegar powder can be obtained.

Below, we would like to explain this invention in further details through real examples.

These examples are presented only for the purpose of explaining the invention in more specific way, and according to the summary of this invention, it is clear to all that this invention is not to be limited by these examples.

Example 1: A physic chemistry analysis of the qualities of the persimmon vinegar concentrate in order find out the best-concentrated rate of persimmon vinegar to make into powder form, various rates of persimmon vinegar concentrate were provided from persimmon vinegar and their color, viscosity, pH, acidity, organic acid, and smell were analyzed and evaluated. When we measured the original liquid of commercially sold persimmon vinegar (persimmon 100%, acidity under 4%, Dae-Yang Natural Co.) to find out the concentration rate of solid material with a sugar concentrate measurer (Atago, Japan), it showed as 5.4%° BX.

We took this persimmon vinegar liquid and vacuum-concentrated it at 55℃ to make persimmon vinegar concentrate samples of 20° BX, 30° BX, 40° BX, 50° BX, 60° BX, and 70° BX. We also collected the condensate obtained during the process of concentrating the vinegar to 20°BX, and 40°BX to use in the analysis.
Example 1-1: The color of the persimmon vinegar concentrate

The original persimmon vinegar liquid 5.4° Bx, the persimmon vinegar concentrate samples of 20° Bx, 30° Bx, 40° Bx, 50° Bx, 60° Bx and 70° Bx all of which were equally diluted to 5.4° Bx, and the condensate from the process of concentrating the vinegar to 20° Bx and 40° Bx were analyzed using a color difference meter (color and color difference meter, Hunter Lab ColorQuest II, USA). The lightness (L), redness (a), and yellowness (b), were measured, and the color difference compared to the unconcentrated original persimmon vinegar liquid (△Eab) was calculated to find out the change in color depending on the level of concentration. (reference: Table 1) The lightness, redness, and yellowness of the standard white board used for comparison were 92.68, 0.81, 0.86, accordingly. (source: Jae-chul Song, Hyun-jung Park, Study of Food constituent Material. Ui-San University Publisher, Ui-San, p. 80-84. 1995)

<table>
<thead>
<tr>
<th>calculated  △Eab</th>
<th>Difference in color</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 - 0.5</td>
<td>Very little difference (almost none)</td>
</tr>
<tr>
<td>0.5 - 1.5</td>
<td>Little difference</td>
</tr>
<tr>
<td>1.5 - 3.0</td>
<td>Just discernable through senses</td>
</tr>
<tr>
<td>3.0 - 6.0</td>
<td>Plainly discernable difference</td>
</tr>
<tr>
<td>6.0 - 12.0</td>
<td>Very bit difference</td>
</tr>
<tr>
<td>over 12</td>
<td>Categorized as a different color</td>
</tr>
</tbody>
</table>

As the result of measuring the color of the persimmon vinegar concentrates as described above, the lightness and yellowness of the original persimmon vinegar liquid was 56.48 and 17.95 accordingly, and showed a slight brown persimmon color. However, when diluting a 20° Bx concentrate to 5.4° Bx (same as the original liquid), the lightness and yellowness decreased to 12.39 and 5.84 accordingly, showing a dark brown color. (reference: Table 2)

Just looking at the 20° Bx concentrate, the color difference was 45.739 from the original liquid, which confirmed the fact that persimmon vinegar changes its color when concentrated, and as the concentration becomes higher, the lightness and yellowness continues to decrease and the color changes to a darker and stronger brown. This showed that even though the concentration was done in a vacuum-concentrator at a low temperature of 55°C, there is a significant browning effect. So, in order to maintain the original persimmon color of persimmon vinegar, it is best to concentrate the liquid to the necessary concentration level at a low temperature within a short period time.
Table 2: The color and the difference in color among the persimmon vinegar concentrate by concentration level

<table>
<thead>
<tr>
<th></th>
<th>Lightness (L)</th>
<th>Redness (a)</th>
<th>Yellowness (b)</th>
<th>Difference in Color (ΔEab)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.4° Bx original liquid</td>
<td>56.45</td>
<td>3.16</td>
<td>17.95</td>
<td></td>
</tr>
<tr>
<td>20° Bx concentrate diluted to 5.4° Bx</td>
<td>12.39</td>
<td>2.44</td>
<td>5.84</td>
<td>45.729</td>
</tr>
<tr>
<td>30° Bx concentrate diluted to 5.4° Bx</td>
<td>9.26</td>
<td>1.97</td>
<td>4.53</td>
<td>49.104</td>
</tr>
<tr>
<td>40° Bx concentrate diluted to 5.4° Bx</td>
<td>8.61</td>
<td>1.96</td>
<td>4.20</td>
<td>49.820</td>
</tr>
<tr>
<td>50° Bx concentrate diluted to 5.4° Bx</td>
<td>6.68</td>
<td>2.15</td>
<td>3.77</td>
<td>51.789</td>
</tr>
<tr>
<td>60° Bx concentrate diluted to 5.4° Bx</td>
<td>5.64</td>
<td>2.42</td>
<td>3.32</td>
<td>52.908</td>
</tr>
<tr>
<td>70° Bx concentrate diluted to 5.4° Bx</td>
<td>4.40</td>
<td>3.09</td>
<td>2.87</td>
<td>54.219</td>
</tr>
<tr>
<td>Concentrate from 20° Bx concentrate</td>
<td>96.01</td>
<td>-0.44</td>
<td>0.53</td>
<td></td>
</tr>
</tbody>
</table>

Example 1-2: Texture of the persimmon vinegar concentrates

The outward viscosity of the original persimmon vinegar liquid 5.4° Bx, the persimmon vinegar concentrate samples of 20° Bx, 30° Bx, 40° Bx, 50° Bx, 60° Bx and 70° Bx were measured using a cylinder-type viscosity measurer (Haake RV20 U.K): the change in shear stress was measured while the shear rate was increased from 0 s⁻¹ to 1,500 s⁻¹ in 1 minute at a temperature of 20°C, and the flowing characteristics of the fluid and the outward viscosity were analyzed (resource: Hyeon-Chul Choi et al. The limology characteristics of pear juice. Korean Food Science Academic Magazine, 27(6), 845-854, 1995). The change in outward viscosity of the original persimmon vinegar liquid of 5.4° Bx and the other persimmon concentrates with various concentrate levels are shown in Figure 1. On Figure 1, (●) refers to the fluid’s flowing characteristic of the 5.4° Bx original persimmon vinegar liquid, (□) the 20° Bx persimmon vinegar concentrate, (■) the 30° Bx persimmon vinegar concentrate, (△) the 40° Bx persimmon vinegar concentrate, (□) the 50° Bx persimmon vinegar concentrate, (○) the 60° Bx persimmon vinegar concentrate, and (●) the 70° Bx persimmon vinegar concentrate. As shown in Figure 1, the shear stress increased slowly by the concentration level until 30° Bx, and the slope of the Newtonian fluid. However, as the concentration level increased beyond 30° Bx, the shear stress increased suddenly and showed the flowing characteristics of a pseudoplastic fluid. When comparing the appearance (shear stress/speed, slope of graph) at the shear speed of 600 s⁻¹, there was a big difference from the point of the concentration level of 30° Bx. Thus, we were able to see that when concentrating persimmon vinegar to a high concentration level over 30° Bx, the viscosity becomes very high from the outward viscosity, the adequate concentration level to simplify and make efficient the manufacture process was a low concentrating level under 30° Bx.
Example 1-3: The acidity and pH of the persimmon vinegar concentrate

The pH and acidity of the original persimmon vinegar liquid of 5.4° Bx, the persimmon vinegar concentrate samples of 20° Bx, 30° Bx, 40° Bx, 50° Bx, 60° Bx, 70° Bx, and the condensate from the process of concentrating the vinegar to 20° Bx were measured directly with a pH meter (Orion SA520, USA): the pH meter was put in a sufficient amount of the sample and 1. ON of NaOH was added until the pH of the sample became 8.1(±0.2) at room temperature. The acidity was calculated by finding out the amount of acetic acid (1. ON NaOH 1ml = acetic acid 0.06g) using the following equation: acidity(%) = (0.06g) number of ml of used 1. ON NaOH x 100/(weight of sample(g)). (Reference: Table 3)

As shown in Table 3, the pH and acidity of the original 5.4° Bx persimmon vinegar liquids was 3.81 and 5.00 accordingly, but as the concentration level increased, the pH level also increased and the acidity decreased to the point where, at 70° Bx, the pH and acidity were 4.63 and 1.04. The low pH and high acidity of 2.71 and 4.29, which showed that a lot of the acids escaped the needed to be done as less as possible as needed to adequately eliminate the biting taste and smell and to spray-dry.

[Table 3] The pH and acidity of persimmon vinegar concentrate

<table>
<thead>
<tr>
<th></th>
<th>pH</th>
<th>Acidity</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.4° Bx original liquid</td>
<td>3.81</td>
<td>5.00</td>
</tr>
<tr>
<td>20° Bx concentrate diluted to 5.4° Bx</td>
<td>4.23</td>
<td>2.54</td>
</tr>
<tr>
<td>30° Bx concentrate diluted to 5.4° Bx</td>
<td>4.34</td>
<td>2.02</td>
</tr>
<tr>
<td>40° Bx concentrate diluted to 5.4° Bx</td>
<td>4.43</td>
<td>1.65</td>
</tr>
<tr>
<td>50° Bx concentrate diluted to 5.4° Bx</td>
<td>4.51</td>
<td>1.45</td>
</tr>
<tr>
<td>60° Bx concentrate diluted to 5.4° Bx</td>
<td>4.58</td>
<td>1.25</td>
</tr>
<tr>
<td>70° Bx concentrate diluted to 5.4° Bx</td>
<td>4.66</td>
<td>1.04</td>
</tr>
<tr>
<td>Condensate from 20° Bx concentrate</td>
<td>2.71</td>
<td>4.29</td>
</tr>
</tbody>
</table>

Example 1-4: The analysis of organic acid within persimmon vinegar concentrates

The original persimmon vinegar liquid of 5.4° Bx, the persimmon vinegar concentrate samples of 20° Bx, 30° Bx, 40° Bx, 50° Bx, 60° Bx, 70° Bx, and the condensate from the process of concentrating the vinegar to 20° Bx were filtered through a 0.25um membrane filter, and their organic acid content was analyzed with HPLC: Supelco gel(300)×7.8mm ID. Supelco Co.) was used as the column, and 0.1% phosphoric acid was used as the solvent. Using acetic acid, citric acid, lactic acid, malic acid, and oxalic acid the solvent movement speed 0.5ml/min, 20°C temperature, and standard samples of organic acids were found through UV. (Reference: Table 4)
Table 4. The amount of organic acids in persimmon vinegar concentrates by concentration level

<table>
<thead>
<tr>
<th></th>
<th>Acetic acid (%)</th>
<th>Citric acid (%)</th>
<th>Lactic acid (%)</th>
<th>Malic acid (%)</th>
<th>Oxalic acid (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.4° Bx original liquid</td>
<td>2.342</td>
<td>0.018</td>
<td>0.177</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>20° Bx concentrate diluted to 5.4° Bx</td>
<td>1.216</td>
<td>0.030</td>
<td>0.133</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>30° Bx concentrate diluted to 5.4° Bx</td>
<td>1.054</td>
<td>0.033</td>
<td>0.309</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>40° Bx concentrate diluted to 5.4° Bx</td>
<td>0.882</td>
<td>0.034</td>
<td>0.379</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>50° Bx concentrate diluted to 5.4° Bx</td>
<td>0.751</td>
<td>0.036</td>
<td>0.404</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>60° Bx concentrate diluted to 5.4° Bx</td>
<td>0.593</td>
<td>0.034</td>
<td>0.282</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>70° Bx concentrate diluted to 5.4° Bx</td>
<td>0.492</td>
<td>0.028</td>
<td>0.343</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Condensate from 30° Bx concentrate</td>
<td>2.159</td>
<td>-</td>
<td>0.0013</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

* - : Undetected

As shown in Table 4 studying the change of the organic acid content of the original 5.4° Bx persimmon vinegar liquid and each persimmon vinegar concentrate sample, the amount of lactic acid, lactic acid, and citric acid were found (in this order), and no malic or oxalic acids were found.

Also, the 2.7° Bx solid matter from the original persimmon vinegar liquid contained 2.342% of acetic acid, which is 86.7% of the total acid amount in the solid matter. However, when concentrated, although the amount of citric acid and lactic acid did not change much, there was a large decrease in the amount of acetic acid. For instance, in the 70° Bx concentrate, when diluted to 2.7° Bx the acetic acid content of the total acid amount in solid matter decreased to 18.2%. Judging by the fact that in the condensate (of 2.5° Bx) obtained while concentrating the vinegar to 20° Bx, the acetic acid content of the total acid amount in solid matter was 83.3%, the loss in acetic acid comes in the vinegar concentrates results from its leaving the vinegar in the form of the condensate. As a result, although it is better to have a high acetic acid content when using persimmon vinegar as seasoning in order to produce its unique flavor with only a little amount, the biting taste and smell becomes a disadvantage when consumed directly as a health food, persimmon vinegar should be concentrated to the maximum of 20° Bx. This will allow the elimination of the too strong taste and smell (making it easier to consume directly) and make the vinegar more susceptible for spary drying.

Putting together Example 1 as described above, the most adequate concentration level for the production of persimmon vinegar powder is 12~18° Bx (maximum of 20° Bx).
Example 2: The production of persimmon leaf extract.

Persimmon leaf extract is produced and added in order to alleviate the acidic taste of persimmon vinegar, to better the flowing quality of the powder, and to add the unique flavor and taste of persimmon leaves to the powder: 10kg of persimmon leaves were washed many times and steamed for 2 minutes at 110°C, then dried off 1 hour using a 40°C hot wind. Next, 10 liters of water per 1kg of dried persimmon leaves were added and it was boiled for 2 hours at 100°C, and then filtered with a compressing filter so that only the liquid was collected. This liquid was concentrated, using a vacuum concentrator, to 15° Bx. 3,000ml of persimmon leaf extract was obtained through this process and was sealed and stored at a low temperature storage room of 4°C.

Example 3: The production of persimmon vinegar powder and the calculation of the best combination ratio.

In order to find out the best combination ratio in powderizing persimmon vinegar, 7 different combinations of mixed liquid were prepared and spray-dried using a spray-dryer (Okahawara L-8, Japan). The quality of the powder of each combination was evaluated (reference: Table 5 and Table 6): in this evaluation, 15° Bx and 20° Bx persimmon vinegar concentrate and 15° Bx persimmon leaf extract was used, as it was shown through Example 1 that the active constituents of persimmon vinegar such as pH, acidity, and organic acids were quite maintained and the biting flavor of persimmon vinegar was quite eliminated at these concentration levels. Cyclo-dextrin was added in the persimmon vinegar concentrate and stirred in a mixer for 2 hours at 20°C. In this mixture, lactose and malto dextrin DE 10~20%, and Arabic gum was added and dissolved. Afterwards, the liquid was homogenized using homogenizer for five minutes at 5°C and made into powder using a spray-dryer. The condition in which the spray-dryer was used was: inlet air temperature 160°C, outlet air temperature 90°C, atomizer speed 20,000 rpm, and feed rate 30ml/min.

[Table 5] Combination ratio of the constituents of persimmon vinegar powder

<table>
<thead>
<tr>
<th>Combination ratio</th>
<th>Persimmon vinegar concentrate (g)</th>
<th>Persimmon leaf extract (g)</th>
<th>Cyclo-dextrin (g)</th>
<th>Lactose (g)</th>
<th>Malto-dextrin (g)</th>
<th>Arabic gum (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>20° Bx, 100.0</td>
<td>-</td>
<td>3.0</td>
<td>4.5</td>
<td>18.0</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>20° Bx, 100.0</td>
<td>-</td>
<td>10.5</td>
<td>7.0</td>
<td>42.0</td>
<td>10.5</td>
</tr>
<tr>
<td>3</td>
<td>20° Bx, 100.0</td>
<td>-</td>
<td>9.0</td>
<td>6.0</td>
<td>36.0</td>
<td>9.0</td>
</tr>
<tr>
<td>4</td>
<td>20° Bx, 100.0</td>
<td>-</td>
<td>-</td>
<td>7.0</td>
<td>52.5</td>
<td>10.5</td>
</tr>
<tr>
<td>5</td>
<td>20° Bx, 90.0</td>
<td>15° Bx, 10.0</td>
<td>7.5</td>
<td>2.5</td>
<td>32.5</td>
<td>7.5</td>
</tr>
<tr>
<td>6</td>
<td>20° Bx, 80.0</td>
<td>15° Bx, 20.0</td>
<td>7.5</td>
<td>2.5</td>
<td>32.5</td>
<td>7.5</td>
</tr>
<tr>
<td>7</td>
<td>20° Bx, 70.0</td>
<td>15° Bx, 30.0</td>
<td>7.5</td>
<td>2.5</td>
<td>32.5</td>
<td>7.5</td>
</tr>
</tbody>
</table>
Table 6: The viscosity and the characteristics perceived through the senses of persimmon vinegar powder

<table>
<thead>
<tr>
<th>Combination ratio 1 powder</th>
<th>Humidity absorption</th>
<th>Volume of molecules</th>
<th>Sour taste</th>
<th>Quality of powder</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>+ + + + +</td>
<td>+ + + + +</td>
<td>+ + + +</td>
<td>+</td>
</tr>
<tr>
<td>Combination ratio 2 powder</td>
<td>+ +</td>
<td>+ + +</td>
<td>+ + +</td>
<td>+ + +</td>
</tr>
<tr>
<td>Combination ratio 3 powder</td>
<td>+ + +</td>
<td>+</td>
<td>+ + +</td>
<td>+ + +</td>
</tr>
<tr>
<td>Combination ratio 4 powder</td>
<td>+ +</td>
<td>+ +</td>
<td>+ + +</td>
<td>+ + +</td>
</tr>
<tr>
<td>Combination ratio 5 powder</td>
<td>+ +</td>
<td>+ + +</td>
<td>+ + + +</td>
<td>+ + +</td>
</tr>
<tr>
<td>Combination ratio 6 powder</td>
<td>+</td>
<td>+ + + +</td>
<td>+ +</td>
<td>+ + + +</td>
</tr>
<tr>
<td>Combination ratio 7 powder</td>
<td>+</td>
<td>+ + + +</td>
<td>+ +</td>
<td>+ + + +</td>
</tr>
</tbody>
</table>

* quality of powder: + : very bad, ++++: very good
humidity absorption: + : very little, ++++: very much
volume of molecules: + : relatively light, ++++: relatively heavy
sour taste: + : very little, ++++: very strong

After evaluating the sour taste, the absorption of humidity, the volume of each molecule, and overall quality of powder of each persimmon vinegar powder produced through spray-drying, with combination ratios number 1 to 7, the powder obtained from spray-drying number 1 ratio absorbed the humidity too much and showed too high a tendency to stick to the walls of the spray-dryer that it was not properly powdered, as shown in Table 6.

Also, some of the spray-dried powder absorbed the humidity of the air immediately after exposure and had a strong burnt taste or smell. Thus, the combination ratio number 2 was spray-dried, adding more gum arabic and cyclo-dextrin to prevent the absorption of humidity and to capture sufficiently the refreshing taste and smell of persimmon vinegar. The powder obtained from number 2 - combination ratio had a good flowing quality due to the additional Arabic gum, which prevented most of the absorption of humidity, and did not have any burnt smell or taste due to the additional cyclo-dextrin. Also, the powder had quite a refreshing smell and taste. However, the concentration rate of the solid matter was 52.9° Bx, and the liquid had a high viscosity, that when sprayed using an atomizer, it formed a long string-like shape when drying and the powder molecules formed a net-like shape, which has a relatively low volume. On the other hand, lactose, Arabic gum, and cyclo-dextrin is expensive compared to other ingredients, so in order to lower the use of the three ingredients and the concentration rate of the solid matter, combination ratio number 3 was spray-dried. Number three showed less string-like formation when sprayed with an atomizer and it powder also had a very good quality.

Combination number 4 was spray-dried to find out the cyclo-dextrin's capturing effect of taste and smell, and when compared to combination number 2, its powder had a lower quality and the significantly less flavor of persimmon vinegar when examined with senses.
However, we were able to confirm that cyclo-dextrin has to be added in order to preserve adequately the taste and smell of persimmon vinegar. Also, as a way to prevent the net-like molecule formation of combination number 2, we tried spray-drying combination number 5, 6, and 7 after adding more persimmon leaf extract. As a result, we found out that as the content of persimmon leaf extract increased, the powder absorbed less humidity and had an improved flowing quality, which lead to an overall improvement in the quality of the powder. The reason that the powder has a higher quality when more persimmon vinegar extract is added seems to be because the persimmon leaf extract contains a significant amount of powder-like high molecule materials which help the liquid to spray-dry easily.

Also, when adding 30% of 15° Bx persimmon concentrate, the unique flavor of persimmon leaves were sensed in the powder, including the bitter taste of persimmon leaf extract. This lead to confirm that the adequate amount of addition was about 20%.

In conclusion, when the powder is considered better when it absorbs less humidity, and has bigger volume, good flowing quality, and adequately sour taste, the best combination to make good persimmon vinegar powder turned out to be combination number 6 and 7. However, combination number 7 had the bitter taste of persimmon leaves, so the best combination seemed to be combination number 6.

Example 4: The production of persimmon vinegar powder using the best combination

Persimmon vinegar powder was produced using the best combination ratio found in Example 3, and the physiochemical quality of the powder was evaluated and a sense test was performed: 15° Bx persimmon leaf extract 20g and cyclo dextrin 7.5g was added to 80g of 15° Bx persimmon vinegar concentrate and stirred in a mixer for 2 hours at 20°C, then lactose 2.5g, malto-dextrin 32.5g, and Arabic gum 7.5g was added and dissolved in the stirred liquid. Next, the liquid was homogenized in a homogenizer and made into powder using a spray-dryer.

Above is the detailed explanation of a certain aspect of this invention, and it is clear to all that these examples are only descriptions of the actual experiment which were done during the invention, and that this invention is not to be limited by these examples. Thus, the actual limit of this invention is to be defined by the attached claims and their equivalents.

[The effect of the invention]

As it is explained above, this invention provides the method of producing persimmon vinegar powder using the technique of adding persimmon leaf extracts and other ingredients to persimmon vinegar concentrate and powderizing the mixture. According to this invention, the acidic taste of of persimmon vinegar can be eliminated and persimmon vinegar powder with the unique flavor of persimmon leave which better appeals to the senses can be produce. Also, through using concentrates, the process of making persimmon vinegar powder can be standardized and economic.
[Claims]

[Claim 1]
The method of producing persimmon vinegar powder through:

(i) The process of getting persimmon vinegar concentration through concentrating persimmon vinegar with a vacuum concentrator to 12~18° Bx,

(ii) using the weight of the concentrated persimmon vinegar as a standard, adding and stirring in persimmon leaf extract 15~30% and cyclo-dextrin 5~9% to the persimmon vinegar concentrate,

(iii) Using the weight of the persimmon vinegar concentrate as a standard, adding and dissolving lactose 1~5%, malto-dextrin 25~35%, gum 5~9% in the stirred liquid, and afterwards, homogenizing the liquid.

(iv) Spary-drying the homogenized liquid and producing the powder.

[Claim 2]
in the method of producing persimmon vinegar powder as described in Claim 1, the technique of obtaining persimmon leaf extract through the following process:

(i) Washing the persimmon leaves and steaming it for 1~10 minutes at 100~200°C, then drying it for 1~2 hours using a 35~50°C hot wind.

(ii) Adding 8~12 liters of water per 1kg of dried persimmon leaves and boiling for 10~30 minutes at 90~100°C, then filtering with compressing filter and obtaining just the liquid.

(iii) Using a vacuum concentrator, coconcentrating this liquid to 12~18° Bx to obtain persimmon leaf extract.

[Claim 3]
in the method of producing persimmon vinegar powder as described in Claim 1, the technique of stirring which is done in a mixer for 30~180 minutes at 0~30°C.

[Claim 4]
in the method of producing persimmon vinegar powder as described in Claim 1, the technique of adding more than one of the following ingredients: black carrageenan, agar, alginate, guar, gum, arabic gum, rocust bean gum, xathan gum, pectin, and carboxymethyl cellulose.

[Claim 5]
in the method of producing persimmon vinegar powder as described in Claim 1, the technique of homogenizing using a homogenizer for 2~10 minutes at 0~30°C.

[Claim 6]
in the method of producing persimmon vinegar powder as described in Claim 1, the technique of spray drying using a spray-dryer, at 150~180°C inlet air, 80~100°C outlet air, and 15,000~20,000 rpm speed of the atomizer while adding the homogenized liquid to the dryer at a rate of 20~40ml/min.

[Claim 7]
The persimmon vinegar powder manufactured using the method described Claim 1.
[Fig. 1]

[Shear rate (Pa) vs. shear stress (i/s)]
[Fig. 2]

Persimmon leaves

↓

Washing

↓

Steaming

100~200°C
1~10 mins

↓

Hot air dry

35~50°C
1~2 hours

↓

Extract

90~100°C
10~30min

↓

Compressed filtering

↓

Persimmon leaf concentrate

8~12 liter/kg

Water
[Fig. 3]

Persimmon vinegar

Concentrate

Persimmon leaves

Extract

Persimmon leaf extract

Cyclodextrin

lactose / malto-dextrin / Gum

Stir

Homogenize

Spray-dry

Persimmon vinegar powder
A. CLASSIFICATION OF SUBJECT MATTER

IPC7 A23F 3/14

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7 A23L 1/10, A23L 1/218, A23L 2/04, A23L 2/38, A23B 7/10, C12J 1/08

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Korean Patents and applications for inventions since 1975

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

NPS, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
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Date of the actual completion of the international search

20 JUNE 2001 (20.06.2001)

Date of mailing of the international search report

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Name and mailing address of the ISA/KR

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Facsimile No. 82-42-472-7140

Authorized officer

KWON, Oh Hee

Telephone No. 82-42-481-5629

Form PCT/ISA/210 (second sheet) (July 1998)
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