Effect of Ageing on Rheological Properties and Quality of Shanxi Aged Vinegar

Hong Zhu1, Pasquale M. Falcone3, Ju Qiu1, Chang-zhong Ren4 and Zai-gui Li2*

1 Institute of Food and Nutrition Development, Ministry of Agriculture and Rural Affairs, Beijing, 100081, China
2 College of Food Science & Nutritional Engineering, China Agricultural University Beijing, 100083, China
3 Department of Agricultural and Food Science, University of Modena and Reggio Emilia, Reggio Emilia, 42100, Italy
4 Engineering and Technology Research Center of Oats, Baicheng Academy of Agricultural Sciences, Baicheng City, 137000, China
*Corresponding author’s e-mail: zhuhong@caas.cn

Abstract. The quality of Shanxi aged vinegar (SAV), a famous Chinese vinegar, was improved during its special long-time ageing. SAV samples of different ageing time were analyzed by composition analysis, rheometer, and principal component analysis (PCA). Three main quality indicators of SAV, namely total acidity, soluble solids content and R ratio, and most of composition, including glucose, acetic acid, lactic acid and total polyphenols increased when ageing process prolonged. Newton function and Power law model were fitted to rheological data to investigate the rheological characters: shear viscosity, consistency coefficients and flow behavior index ($n$). PCA showed the viscosity of SAV had positive relationship with ageing time as well as all composition parameters. Quality including main quality indicators, composition and rheological characters was significantly different ($p<0.05$) among the SAV in different phases. Thus, the rheological method was a very useful tool to classify the quality of SAV.

1. Introduction
Shanxi aged vinegar (SAV) was one of the most important traditional Chinese vinegar originated in Shanxi province, China, which was certificated as product of geographical indication (PGI) by Chinese national standard (GB/T 19777-2013). Authority certifies the vinegar authenticity and quality according to this national standard as well. The main raw material of SAV is cereal, such as sorghum, buckwheat, millet and wheat. Manufacturing processes of SAV has mainly four steps including alcoholic fermentation, acetic fermentation, thermal processing and ageing. The long-time ageing process of SAV took place in big open jar exposed to sunlight and air. Ageing plays a dominant role in the overall production of SAV, especially for its final rheological and sensorial properties. SAV becomes more brown and viscous over long-time ageing. The long-time ageing leads to high quality and appreciable transformations for vinegar by sensorial analysis [1].

The commercial value of SAV was closely related to its age, and special prices are requested for very old vinegar. So it is important to classify the quality of SAV of different ageing time. However,
current legislation of SAV in China only set a basic quality standard of composition indicators for market access. Some composition indicators were used to assess the quality of vinegar during ageing. The previous study showed that the sugar content, total acidity and R ratio (°Brix/total acidity) were good reference parameter to control crystallization in homemade balsamic vinegar during ageing[2]. Thus, a fast and precise method is still needed to classify the quality of SAV during ageing. Fortunately, some authors concluded that rheological measurements could be a simple and reliable method. In a pioneering work, Newtonian behavior was observed in high quality traditional balsamic vinegar samples appearing liquid at a visual inspection; shear-thinning behavior was observed in defect samples[3]. Further investigation demonstrated the difference in structure, such as molecular size distribution, between these two groups of traditional balsamic vinegar [4; 5]. Therefore, rheological method was a potential approach to classify SAV of different ageing time according to their quality.

In this research, the quality transformation of SAV during ageing was determined using composition analysis, rheometer and electronic-nose firstly. Then SAV of different ageing time was classified according to their quality by a rheological method.

2. Materials and methods

2.1. Samples and chemical agents
Detailed information of SAV sample was listed in Table 1. The SAV samples were obtained from three producers in Shanxi province, China, i.e. Donghu (DH) Co., Ltd., Ninghuafu (N) Co. Ltd. and Zilin (Z) Co., Ltd. The labeled ageing years were claimed by the producers, which ranged from 3 months to 20 years. Standards of glucose, fructose, acetic acid and lactic acid were obtained from Sigma-Aldrich (Shanghai, China).

2.2. Composition analysis
The soluble solids content in vinegar was measured using Leica temperature compensating oBrix scale (0-30) refractometer (Leica Inc., Buffalo, NY). Total acidity was measured by titration with 0.5 M sodium hydroxide solution as proposed by China national standard (GB/T 19777-2013). The R ratio was calculated as oBrix/total acidity. Sugar content (glucose and fructose) was determined on an ion chromatograph; model ICS-5000 (Dionex, USA). The content of total polyphenols was measured by the Folin-Ciocalteu method [6].

2.3. Rheological measurement
Rheological behavior of vinegar was studied by a controlled-strain rotational viscosimeter (ARES model, TA Instruments, USA, New Castle) equipped with a force rebalance transducer (model 1KFRTN1, 1-1000 g cm, 200 rad/s, 2-2000 gmf) and a couette tool, a concentric cylinder geometry (diameter of cup and bob, 34 mm and 32 mm respectively). The steady temperature was ensured with an accuracy of ±0.1 ºC using a controlled fluid bath unit and an external thermostatic bath. Three replicates of flow experiment were performed for each sample at 20 ºC. Steady shear stress over a range of shear rates of 0.1–500 s-1 was measured. A suitable cover tool was used to seal the top of the couette tool during tests to prevent acetic acid evaporation.

Newton function and Power law model in Eqs. (1)-(2) were fitted to the rheological data obtained for SAV of different ageing time. The Origin 8.0 was used for the calculation.

\[
\text{Newton: } \delta = \mu \cdot \gamma \\
\text{Pow law: } \delta = K \cdot \gamma^n
\]

Where \(\delta\) is the shear stress (Pa); \(\gamma\) is the shear rate (s-1); \(\mu\) is the shear viscosity (Pa s), and \(K\) is the consistency coefficient (Pa sn). \(n\) was flow behavior index. For a Newtonian behavior, \(n=1\). If \(n<1\), the flow behavior is called shear-thinning; if \(n>1\), the flow behavior is shear-thickening.
2.4. Electronic nose analysis
All samples were analyzed on a FOX 4000 from Alpha-MOS (Toulouse, France) combined with a headspace auto sampler. The FOX 4000 instrument includes 18 different metal oxide gas sensors (i.e. LY2/LG, LY2/G, LY2/AA, LY2/GH, LY2/gCTl, LY2/gCT, T30/1, P10/1, P10/2, P40/1, T70/2, PA/2, P30/1, P40/2, P30/2, T40/2, T40/1, TA/2). Before testing, the electronic nose system should be warmed up and calibrated with processed pure air (filtered with activated silica and charcoal, 99.99%), until the data are stable. In the experiment, 4.0 g of vinegar sample was placed in 20 mL vials with silicone caps and then settled into the automatic sampling carousel. After 10 min equilibration at 60 °C under agitation (350 rpm), the measurement started. The headspace was pumped over the sensor surfaces for 120 s. The internal for data collection was 1 s. The maximum response points automatically recorded for each of 18 sensors were used as the electronic noise response. Each of the 14 vinegar samples was prepared four times in random order, and the average of the results was used for data analysis.

2.5. Statics analysis
Statistical comparison of the mean values was performed by one-way ANOVA (p<0.05) by SPSS 18.0 software (SPSS Inc., Chicago, IL, USA). The multivariate statistical analysis of composition, rheological and electronic nose data in terms of principal component analysis (PCA) was carried out using STATISTICA (Statsoft, Inc., Tulusa, USA).

3. Results and discussion

3.1. Composition analysis
Three quality indicators of vinegar, namely total acidity, soluble solids content and R ratio, increased when ageing process prolonged (listed in Table 1). The total acidity of SAV is required to be above 6 g/100 mL according to Chinese legislation. Table 1 shows the total acidity of SAV samples at the beginning of ageing were around 7 g/100 mL. SAV with longer ageing time had much higher total acidity, indicating higher quality. The R ratio (ratio of oBrix and total acidity) was also a marker used in practice for the assessment of the sensorial properties of vinegar, such as traditional balsamic vinegar from Modena and Reggio Emilia, and expressed the balance between sweet and sour taste [7]. Higher R ratio indicated better sensorial properties, which referred to the shifting from sour to sweet taste. Previous research valued the ideal R ratio for traditional balsamic vinegar, which ranged from 6.74 (ideal sour taste boundary) to 9.26 (ideal sweet taste boundary) [2]. The R ratio of SAV ranged from 2.3 to 5.1. The trends of R ratio from each SAV producer during ageing were increased, demonstrating that the sour and sweet taste of SAV became more balance and acceptable. Thus, the overall of SAV quality has been improved during ageing.

| Sample | Ageing time (year) | Total acidity (g/100mL) | oBrix | R ratio|
|--------|-------------------|-------------------------|-------|--------|
| DH-young | 0                 | 7.32±0.04<sup>a</sup> | 18.3±0.1<sup>b</sup> | 2.5    |
| DH-3y  | 3                 | 8.07±0.21<sup>b</sup>  | 21.9±0.1<sup>c</sup> | 2.7    |
| DH-5y  | 5                 | 8.63±0.02<sup>c</sup>  | 26.2±0.0<sup>d</sup> | 3.0    |
| DH-8y  | 8                 | 8.97±0.55<sup>c,d</sup>| 25.7±0.4<sup>e</sup> | 2.9    |
| DH-10y | 10                | 10.29±0.04<sup>e</sup>| 37.1±0.0<sup>f</sup>| 3.6    |
| DH-16y | 16                | 10.38±0.21<sup>f</sup>| 43.2±0.0<sup>f</sup>| 4.2    |
| DH-20y | 20                | 13.44±0.33<sup>f</sup>| 47.4±0.0<sup>f</sup>| 3.5    |
| N-3y   | 3                 | 7.26±1.70<sup>e</sup>  | 16.8±0.0<sup>e</sup>| 2.3    |
| N-10y  | 10                | 12.09±0.21<sup>f</sup>| 53.5±0.1<sup>f</sup>| 4.4    |
| N-16y  | 16                | 12.78±0.12<sup>f</sup>| 59.7±0.0<sup>f</sup>| 4.6    |
The contents of most major constituents, including glucose, acetic acid, lactic acid and total polyphenols, increased during ageing (Table 2). Fructose was only detected in relatively younger SAV under five years but disappeared after that. The increasing of major constituents and quality indicators indicated SAV was condensed during ageing, mainly owing to the remove of water. This result was in agreement with a previous study of SAV [8]. Furthermore, condensing was a common characteristic of ageing for several well-known vinegars. The sugar content of traditional balsamic vinegar of Modena can reach up to 72 oBrix, because of evaporation of water during ageing [2]. The dry extract and total acidity of Sherry vinegar were almost twice the initial value due to ageing [9].

### Table 2 Chemical composition of Shanxi aged vinegar.

| Sample   | Glucose (g/L) | Fructose (g/L) | Acetic acid (g/L) | Lactic acid (g/L) | Total polyphenols (g/L) |
|----------|---------------|----------------|-------------------|-------------------|-------------------------|
| DH-young | 3.10±0.14ª    | 8.60±0.69ª     | 89.92±2.70ª       | 30.96±2.17ª       | 7.61±0.42ª              |
| DH-3y    | 11.50±0.42ª   | 6.90±0.99ª     | 89.93±3.32ª       | 35.73±0.75ª       | 12.45±0.34ª             |
| DH-5y    | 24.70±2.40ª   | 7.92±0.78ª     | 89.35±5.36ª       | 35.23±2.11ª       | 12.61±0.87ª             |
| DH-8y    | 34.63±4.20ª   | n.d.           | 75.11±5.26ª       | 51.97±4.16ª       | 18.98±1.06ª             |
| DH-10y   | 47.20±3.39ª   | n.d.           | 91.05±3.64ª       | 48.67±0.97ª       | 23.82±0.53ª             |
| DH-20y   | 54.40±2.26ª   | n.d.           | 138.09±2.76ª      | 67.00±4.02ª       | 25.94±0.27ª             |
| N-3y     | 11.04±0.45ª   | 9.68±0.91c     | 61.69±3.70ª       | 14.62±1.17ª       | 6.21±0.34ª              |
| N-10y    | 44.24±1.13ªf  | n.d.           | 83.10±6.65ª       | 58.62±1.76ª       | 23.84±3.47ª             |
| N-16y    | 44.88±1.13ªf  | n.d.           | 100.42±3.01ª      | 72.05±2.88ª       | 34.19±0.91ª             |
| N-20y    | 69.68±0.59ª   | n.d.           | 96.00±3.84ª       | 61.09±1.83ª       | 20.08±0.95ª             |
| Z-5y     | 12.28±0.40ª   | n.d.           | 111.96±3.36ª      | 40.05±2.00ª       | 11.10±0.72ª             |
| Z-8y     | 25.32±0.85ª   | n.d.           | 77.60±1.55ª       | 19.02±1.14ª       | 10.56±0.34ª             |
| Z-10y    | 61.12±2.72ª   | n.d.           | 43.70±1.31ª       | 24.58±0.49ª       | 12.93±0.11ª             |

Different lowercase letters indicate significance differences (p <0.05).

#### 3.2. Rheological properties

The rheological properties were analyzed by fitting the Newton function to experimental data over a range of shear rates varying from 0.1 to 500 s⁻¹ in the Eq. (1). The suitability of fit was proved by adjust $R^2$ (listed in Table 3), which was greater than 0.95 which was chosen as the threshold for checking linearity [3]. The results showed all of the fourteen SAV exhibited Newtonian behavior. Fitting results of Pow law model were listed in Table 3. The rheological data was fitted to Pow law model successfully because the adjust $R^2$ were all above 0.99. The values of K increased with ageing, implying the viscosity of SAV increased during the ageing process. The values of n decreased from 1.52 to 0.98, demonstrating reduction in shear-thickening behavior and transformation to Newtonian behavior. Twelve samples of 14 SAV were shear-thickening fluids (n>1) and the rests were Newtonian fluid (N-16y, n=1) and shear-thinning fluid (N-20y, n<1). All SAV samples were identified as Newtonian fluids by Newton model, while some younger SAV were considered as non-Newtonian fluids according to Pow law model. Obviously, there was a conflict in the fitting results between Newton function and the Pow law model. A similar conflict was also found in research on carrot juice [10]. It could be caused by the different flow behavior between middle shear rates and lower or higher
shear rates. As Fig. 1 shown, younger SAV samples only exhibited Newtonian behavior at middle shear rates (1 to 300 s⁻¹), but exhibited non-Newtonian behavior at lower (0.1 to 1 s⁻¹) or higher shear rates (>300 s⁻¹). Shear stress measured at low shear rates (i.e. below 60s-1) plays a key role in sensory perception of the rheological properties [11]. It will be of more usefulness to understand the physical origin of the non-Newtonian behavior of the younger SAV. Compared to Newton function, the Pow law model showed higher values of adjust $R^2$ (>0.99), because it could describe flow behavior at lower (0.1 to 1 s⁻¹) or higher shear rates (>300 s⁻¹) better. Thus, Power law was the best model to SAV.

Table 3 Parameters of Newton function and Power law model of SAV of different ageing time.

| Sample | Newton ($\mu=\mu\cdot\gamma$) | Power law ($\bar{\delta}=K\cdot\gamma^n$) |
|--------|-------------------------------|------------------------------------------|
|        | $\mu$ (Pa•s)                  | $K$ (Pa•s^n)    | $n$ | $R^2$ |
| DH-young | 0.00393 0.9579 | 2.14e^-4 1.49 | 0.9961 |
| DH-3y    | 0.00414 0.9538 | 1.86e^-4 1.52 | 0.9956 |
| DH-5y    | 0.00448 0.9633 | 3.74e^-4 1.42 | 0.9928 |
| DH-8y    | 0.00446 0.9635 | 3.75e^-4 1.42 | 0.9929 |
| DH-10y   | 0.00738 0.9990 | 0.00516 1.06  | 0.9999 |
| DH-16y   | 0.01155 0.9995 | 0.00903 1.04  | 1.0000 |
| DH-20y   | 0.01658 0.9997 | 0.01379 1.03  | 1.0000 |
| N-3y     | 0.00367 0.9512 | 1.33e^-4 1.56 | 0.9980 |
| N-10y    | 0.03209 0.9999 | 0.02985 1.01  | 1.0000 |
| N-16y    | 0.10130 1.0000 | 0.10099 1.00  | 1.0000 |
| N-20y    | 0.33970 0.9999 | 0.38159 0.98  | 1.0000 |
| Z-5y     | 0.00430 0.9571 | 2.48e^-4 1.48 | 0.9936 |
| Z-8y     | 0.00535 0.9870 | 0.00175 1.19  | 0.9950 |
| Z-10y    | 0.00475 0.9758 | 7.36e^-4 1.31 | 0.9948 |

Figure 1. Rheological data representative of non-Newtonian behavior of younger SAV at low and high shear rates.

The transformation of flow behavior of SAV during ageing was unique, indicating profound composition and structure change occurred. Traditional balsamic vinegar exhibited two flow
behaviour, namely Newtonian and shear-thinning behaviour [3]. Composition and structure analysis demonstrated significant difference in sugar content, oBrix, microstructure image and molecular size distribution between these two kinds of traditional balsamic vinegar [3; 5]. Increasing of viscosity could be one reason that leaded transformation of soursop juice from shear-thickening to Newtonian behavior [12]. The research on traditional balsamic vinegar shown that the n values decreased with the increasing of viscosity, which caused by increasing of glucose, fructose and oBrix [3]. The increasing of viscosity of SAV could be evidenced by the increasing of K value (Table 3). However, the change from the Newtonian to shear-thinning behavior was not only caused by increasing of viscosity, but also forming of some polymers, which resulted in the structure change of the system. Traditional balsamic vinegar changed from Newtonian to shear-thinning behavior due to the presence of nitrogen-free polymers (melanoidin) [5]. Furthermore, a deviation from the Newtonian to the shear-thinning behavior of vinegar was proved in a synergic way by adding minor constituents affecting viscosity [13]. Theoretically, some polymers, such as melanoidin, could be formed in the ageing process of SAV as well, led to the shear-thinning behavior of N-20y. The brown-colored product, namely melanoidin, formed through Maillard and caramelization reactions [14]. Low molecular weight compounds, such as 5-hydroxymethyl-2-furfural (HMF) and furfurals, formed and then were progressively incorporated into the melanoidin skeleton during ageing [15; 16]. It was reported the formation of HMF and furfurals is strongly dependent on the amount of water [17; 18]. In vinegar with a high sugar concentration and low water content, the formation of HMF and furfurals take place even at room temperature. It was further evidenced by detection of HMF, furfurals and melanoidin in final products of SAV [19; 20].

3.3. PCA analysis of composition, rheological and electronic nose data of SAV

Principal component analysis was performed treating composition, viscosity at different shear rates (1 s-1, 10 s-1, 50 s-1, 100 s-1 and 500 s-1) and response of 18 gas sensors of electronic nose as independent variables. The result highlight a clear picture of the vinegars distribution with respect to the investigated parameters as well as to the vinegar producer and aging time (Fig. 2). In addition to the K and n, some targeted viscosities measured at 1 s-1, 10 s-1, 50 s-1, 100 s-1 and 500 s-1 was used, in order to simulate the shear rates that are widely believed occurring under tasting of food beverages. As showed in Fig. 2, the Factor 1 explains more than 42% of the experimental variance, with all composition parameters increasing together with all targeted viscosities as well as with aging time (vinegars concentrated over decade becoming more dense and viscous). Factor 2 (explaining more than 39% of the variance) discriminated the investigated SAV samples mainly according to the noise-metal-oxide responses and aging time. As can be inferred, more younger vinegars are characterized by greater levels for the cluster of the noise-metal-oxide responses referred as to "LY_"; whereas, more aged vinegars are characterized by pronounced levels of the cluster of noise-metal-oxide responses referred as to "T_" and "P_".

The PCA plot demonstrated the positive correlations between viscosity, ageing time and all composition parameters. Similarly, positive correlations were found between K values and oBrix in traditional balsamic vinegar [3]. Therefore, the rheological properties were not only related to the texture of SAV but also related to composition of SAV.
Figure 2. PCA plot of factor loading showing the correlations between the composition, rheological and electronic nose data of SAV and the distribution of vinegar samples with respect to PC1 and PC2. GLU, FRU, AcH, LacAc, TotPolyph, TitAc, Brix and V1-V500 indicated glucose, fructose, acetic acid, lactic acid, total polyphenols, total acidity, °Brix and viscosity at 1 to 500 s⁻¹.

4. Conclusions
In summary, the quality variation of SAV during the long-time ageing process (from 3 months to 20 years) was assessed by composition analysis, rheometer and electronic nose, and PCA was performed on the experimental data. Three quality indicators increased when ageing process prolonged, namely the total acidity, soluble solids content and R ratio. The content of most of major constituents increased during SAV ageing, including glucose, acetic acid, lactic acid and total polyphenols. These results showed the quality and sensorial properties of SAV has improved during ageing. Newton function and Power law model were fitted to the rheological data to calculate the rheological parameters: µ, K and n. Power law was the best fitted model to the experimental data of SAV, because it could describe flow behavior at lower (0.1 to 1 s⁻¹) or higher (>300 s⁻¹) shear rates better. PCA showed viscosity of SAV was increased together with ageing time as well as all composition parameters, except for fructose. In addition, younger vinegars are characterized by greater levels for the cluster of the noise-metal-oxide responses referred as to "LY_". Thus, we thought rheological method was a very useful tool to class quality of SAV during ageing.

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