



Original article

## New “Acidic Acetatic Index” (AAI) Based on the Interrelationship “Volatile acids – Ethyl Acetate” for Determination of the Level of Wine Acidification

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### Abstract

New “Acidic Acetatic Index” (AAI) for determination of the level of acidification and acid stability of wines was proposed. AAI was the ratio between the amount of volatile acids (g.dm<sup>-3</sup>) and the amount of ethyl acetate (mg.dm<sup>-3</sup>) in the wine. A scale for determination the degree of acidification and acid stability of wine, based on the values of the new AAI indicator was proposed. The scale consists of four ranges formed by the AAI values: 1 (AAI ≤ 0.0002) - Poor acidic stability, high probability for apparent acidification due to spoilage; 2 (0.0002 < AAI ≤ 0.002) - Relatively good acidic stability but with possible acidification due to spoilage; 3 (0.002 < AAI ≤ 0.02) - Very good acidic stability; 4 (0.02 ≤ AAI) - Excellent acidic stability. An analysis of 12 red wines was carried out and the values of the new AAI indicator were determined. The lowest AAI was found in sample 11 (AAI = 0.007), and the highest in sample 9 (AAI = 0.078). Wine sample 11 was fell within range 3 of the suggested scale. This range characterized it as having a very good acidic stability. Wine sample 9 was fell within range 4 of the AAI scale. It was characterized by excellent acidic stability. The new AAI index and its scale can be used as an adequate indicator to characterize the acidification due to spoilage process. It can find application in research practice as well as a control indicator in wine production practice.

**Keywords:** Acidification Spoilage, Acidic – Acetatic Index (AAI), Acetic Acid Fermentation, Ethyl Acetate, New Indicator, Volatile Acids, Wine, Wine Quality.

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## INTRODUCTION

One of the most dangerous processes occurring in wines, defined as "wine disease", is acetic acid decay. It is mainly caused by acetic acid bacteria able to degrade alcohol to acetic acid formation (Yankov et al., 2000; Abrasheva et al., 2008). This process leads to a severe deterioration in quality with unpleasant acetic-acid taste of the wine, which is increased by increasing of the amount of synthesized acetic acid. The control indicator of this process in wine practice is the determination of "volatile acidity". It is the sum of all volatile monovalent fatty acids (Chobanova, 2012), 90% of their quantity being acetic acid (Velkov, 1996). The normal content of volatile acids in young wines is 0.30 - 0.50 g.dm<sup>-3</sup> (Chobanova, 2012) and the average is 1.00 - 1.30 g.dm<sup>-3</sup> (Velkov, 1996).

The ethyl acetate is one of the main quantitative dominant wine esters (Robinson et al., 2014; Meng et al., 2011). It is formed during the alcoholic fermentation from the yeasts *Saccharomyces cerevisiae*, as well as during the wine aging based on the esterification process (Nordest et al., 1975; Perestrelo et al., 2006; Chobanova, 2012). It is present in wines in quantities 30.00 - 300.00 mg.dm<sup>-3</sup>, and in young wines 50.00 - 80.00 mg.dm<sup>-3</sup> (Chobanova, 2012). When it is in normal quantities, it positively affects the aromatic perception, giving fruit flavors to the wines (Tao and Li, 2009). Exceeding the permitted concentrations, however, it deteriorates the quality of the wine and increases the acetic-acid aroma indicating spoilage (Gil et al., 2006). Wines can be evaluated with high degree of acetic acid decay at high levels of ethyl acetate and relatively normal volatile acidity (Chobanova, 2012).

*The aim of the present study is to propose new "Acidic Acetatic Index" (AAI) based on the interrelationship "volatile acids - ethyl acetate" for determination of the level of wine acidification.*

### **Materials and Methods**

#### ***Determination of volatile acids content***

The volatile acids (g/dm<sup>3</sup>) were determined by distillation method followed by titration with NaOH (Ivanov et al., 1979; Chobanova, 2012).

#### ***Identification and quantification of ethyl acetate by GC-FID***

The ethyl acetate content of the wines was determined on the basis of a stock standard solution obtained in accordance with method IS 3752:2005. 1-octanol was used as the internal standard.

The resulting standard solution was injected in a Varian 3900 gas chromatograph (Varian Analytical Instruments, Walnut Creek, California, USA) with a capillary column VF max MS (30m, 0.25mm ID, DF = 0.25µm) equipped with a flame ionization detector (FID). The carrier gas was He. The hydrogen for combustion was delivered to the chromatograph via a hydrogen bottle. The injection was manually by microsyringe.

The parameters of gas chromatographic determination were: injector temperature -220° C, detector temperature - 250° C, initial oven temperature - 35° C / 1 min, rising to 55° C in 2° C / min for 11 min, rising to 230° C in 15 ° C / min for 3 min. Total chromatographic time - 25.67 min.

After determination of the retention time of ethyl acetate in the standard solution, identification and quantification were performed. They were based on the injection of wine distillates. The samples were injected in 2 µl in the gas chromatograph and identification and quantification of ethyl acetate in the wine distillates were performed.

## Results and Discussion

The new Acidic Acetatic Index (AAI) is the ratio between the amount of volatile acids in the wine and the amount of ethyl acetate (equation 1).

$$AAI = \text{Volatile acids amount (g.dm}^{-3}\text{)} / \text{Ethyl acetate amount (mg.dm}^{-3}\text{)} \quad (1)$$

Data for AAI in different red wines are presented in Tables 1 and 2.

**Table 1.** Data for Acidic Acetatic Index (AAI) in analyzed red wines

| INDICATORS                         | RED WINES |          |          |          |          |          |
|------------------------------------|-----------|----------|----------|----------|----------|----------|
|                                    | Sample 1  | Sample 2 | Sample 3 | Sample 4 | Sample 5 | Sample 6 |
| Volatile acids, g.dm <sup>-3</sup> | 0.84      | 0.66     | 0.66     | 0.84     | 0.60     | 0.66     |
| Ethyl acetate, mg.dm <sup>-3</sup> | 46.15     | 30.03    | 25.34    | 55.44    | 30.92    | 41.74    |
| AAI                                | 0.018     | 0.021    | 0.026    | 0.015    | 0.019    | 0.015    |

**Table 2.** Data for Acidic Acetatic Index (AAI) in analyzed red wines

| INDICATORS                         | RED WINES |          |          |           |           |           |
|------------------------------------|-----------|----------|----------|-----------|-----------|-----------|
|                                    | Sample 7  | Sample 8 | Sample 9 | Sample 10 | Sample 11 | Sample 12 |
| Volatile acids, g.dm <sup>-3</sup> | 0.66      | 0.72     | 0.66     | 0.36      | 0.66      | 0.78      |
| Ethyl acetate, mg.dm <sup>-3</sup> | 24.25     | 52.80    | 8.38     | 42.90     | 85.60     | 43.63     |
| AAI                                | 0.027     | 0.013    | 0.078    | 0.008     | 0.007     | 0.017     |

The obtained results showed that AAI occupied values of 0.007 - 0.078 for the studied wines.

Based on the correlation between the amount of ethyl acetate (ester of ethanol and acetic acid synthesized by both yeasts during alcoholic fermentation and acetic acid bacteria) and volatile acidity (dominated by acetic acid) was proposed an Acidic Acetatic Index (AAI) scale to assess the level of wine acidification and its acidic stability (Figure 1).

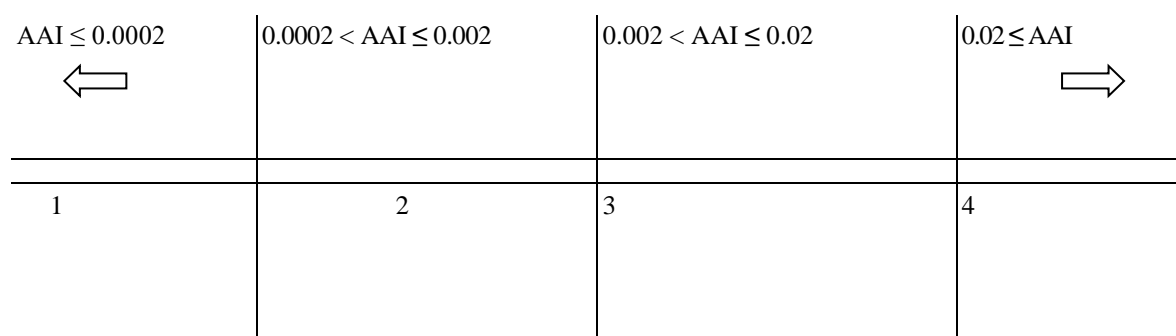


Figure 1. The AAI scale for determination of the level of wine acidification and its acidic stability  
 1- Poor acidic stability ( $AAI \leq 0.0002$ ), high probability for apparent acidification due to spoilage; 2 - Relatively good acidic stability but with possible acidification due to spoilage ( $0.0002 < AAI \leq 0.002$ ); 3- Very good acidic stability ( $0.002 < AAI \leq 0.02$ ); 4 - Excellent acidic stability ( $0.02 \leq AAI$ ).

The suggested scale (Figure 1) can define the acidic stability of the analyzed wines. It consists of 4 intervals fixed by the values of proposed new Acidic Acetatic Index (AAI).

Interval 1 had values of  $AAI \leq 0.0002$ . Wine fell within this range was define with poor acidity and high probability for apparent acidification due to spoilage, with a characteristic acetic-acid taste and aroma. The quality of the wine was degraded by the initiation of the acetic acid process.

Interval 2 had AAI values in the range of  $0.0002 < AAI \leq 0.002$ . Wine with values of AAI in this interval showed relatively good acidic stability but with possible acidification due to spoilage. The ratio between the amount of volatile acids and ethyl acetate was relatively good. There was not observed strong acetic flavor, but it is possible.

Interval 3 fixed AAI in the range  $0.002 < AAI \leq 0.02$ . Wine with such values of AAI was characterized by very good acidic stability. A very good balance between the amount of volatile acids and ethyl acetate was achieved. Stability without acetic acid spoilage.

Interval 4 fixed AAI in the range  $0.02 \leq AAI$ . Wine with AAI values in this range had excellent acidic stability. An excellent balance between volatile acids and ethyl acetate was observed. There was not observed the acetic acid spoilage.

Considering and analyzing the AAI results in the investigated wines, it was evident that the lowest AAI value was observed in sample 11. This value of the new indicator  $AAI = 0.007$ . This wine fell within range 3 from the scale -  $0.002 < 0.007 (AAI) \leq 0.02$ . The wine can be characterized by the proposed scale (Figure 1) as having a very good acidic stability. It can be seen that the volatile acidity has a very good quantitative dimension -  $0.66 \text{ g.dm}^{-3}$ . The ethyl acetate content was also in the norm ( $85.60 \text{ mg.dm}^{-3}$ ). In this quantity it exerted a positive influence on the wine aroma. The ethyl acetate gives a fruity aroma to wine at concentrations lower than  $150.00 \text{ mg.dm}^{-3}$  (Tao and Li, 2009).

The highest AAI value in the wines analyzed was reported in sample 9, namely  $AAI = 0.078$ . This wine fell within range 4 of the AAI scale (Figure 1) -  $0.02 < 0.078$  (AAI). The wine was characterized by the proposed scale as having excellent acidic stability. This was also apparent from the result of its volatile acidity ( $0.66 \text{ g.dm}^{-3}$ ) that absolutely met the normal volatile acidity criterion. The amount of ethyl acetate ( $8.38 \text{ mg.dm}^{-3}$ ) was also in the norm that provided a positive influence of the ester.

From the analysis of the results obtained, it can be seen that the new "Acidic Acetic Index" (AAI) and proposed scale can serve as adequate indicators for determining and characterizing the level of wine acidification and its acidic stability.

AAI and its scale can be used for:

- Adequate characterization of the wines with regard to their level of acidification;
- The indicator may be applied in scientific studies to better reflect the acid stability of the wines analyzed;
- AAI can be applied in wine-making practice, for more complex determination of acidity and acidic stability of wines.

## **Conclusion**

New indicator "Acidic Acetic Index" (AAI) for determination of the level of acidification and acid stability of wines was proposed. AAI was based on the ratio between the amount of wine volatile acids ( $\text{g.dm}^{-3}$ ) and the amount of ethyl acetate ( $\text{mg.dm}^{-3}$ ).

AAI scale for determination of the level of wine acidification and its acidic stability was proposed. The scale consists of four ranges formed on the base of AAI values: 1 ( $AAI < 0.0002$ ) - Poor acidic stability, high probability for apparent acidification due to spoilage; 2 ( $0.0002 < AAI < 0.002$ ) - Relatively good acidic stability but with possible acidification due to spoilage; 3 ( $0.002 < AAI < 0.02$ ) - Very good acidic stability; 4 ( $0.02 < AAI$ ) - Excellent acidic stability.

Twelve red wines were analyzed, with the lowest AAI found in sample 11 ( $AAI = 0.007$ ). The wine fell within the scale range 3, which characterized it as having very good acidic stability.

The highest AAI was found in sample 9 ( $AAI = 0.078$ ). The index of this wine fell within range 4 of the AAI scale. It was characterized by excellent acidic stability.

The new AAI index and its scale can be used as an adequate indicator to characterize the acidification due to spoilage process. It can find application in research practice as well as a control indicator in wine production practice.

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