



Short communication

Crystallization and jamming in the traditional balsamic vinegar

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ABSTRACT

High-resolution light microscopy (HR-LM) and X-ray diffractometry (XRD) were used in a non-destructive way to study the bulk structure of the Traditional Balsamic Vinegar of Modena (TBVM), an Italian specialty produced from cooked grape must and through a long aging process at room temperature. Data showed that TBV is a viscous food dispersion that may undergo solidification through equilibrium and out-of-equilibrium phase transitions including crystallization and jamming of amorphous colloids. Crystallization is due to the rearrangements of the α -D-glucose monohydrate molecules; while, jamming is caused by the crowding of amorphous long-living colloids of unidentified substances that eventually separated into a space-limited solid-like phase or into a gel network spanning the available volume. Due to their natural ability to self-associate into amorphous colloids, a role of polymeric melanoidins was also supposed into the development of vinegar bulk structure and, eventually, into its solidification.

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1. Introduction

TBVM is an Italian special vinegar which has recently earned protection of their appellation by the European Community (G.U. n.124 del 30/05/2000). It is produced starting from the grape juice that is cooked at atmospheric pressure using an open vessel directly heated by flame. During cooking, both the composition and physical properties undergo profound changes, the main of which are the solute concentration due to the water vaporization as well as sugar conversion, polyphenol degradation and formation of new compounds having radical-scavenging activity (Falcone & Giudici, 2010). After cooking, the reducing sugars present into the concentrated must undergo fermentation by yeasts with the production of ethanol; therefore, the ethanol is oxidized into the acetic acid by acetic acid bacteria. Finally, fermented and oxidized vinegar is held for a long time within a series of wooden barrels having a scalar size, allowing some key chemical and physical changes to progress to a great extent. Time plays the central role in the overall production of TBVM requiring an aging period, 12 years at least according to the law, during which new chemical–physical changes take place giving to the vinegar its final rheological (Falcone, Verzelloni, Tagliazucchi, & Giudici, 2008) and sensorial properties (Giudici et al., 2009). In the last years, unexpectedly, undesired events of solidification of the TBVM are becoming frequent too either inside the wooden barrels

during aging as well as, after the aging period, inside the hermetic glasses. In these cases, the separation of a solid-like phase or of a full-space spanning gel may happen into the vinegar bulk, resulting in the loss of the sensorial quality of this precious vinegar.

In this work, either light microscopy and X-ray diffraction analyses were performed with the purpose to study the bulk structure in high quality and partially solidified TBVMs.

2. Materials and methods

2.1. Samples retrieval

Some aliquots of TBVM, produced according to the specific disciplinary (GURI n. 124 del 30-5-2000) and for which vinegar producers claimed an aging period of 25 years at least, were kindly supplied as of low quality samples by the Consortium for the protection of the TBVM designation. As a function of their visual appearance and of their ability to flow under gravity conditions, the samples have been grouped into three groups: (i) “vinegar flowing with constrained dynamics” because of their partial solidification (into the text these vinegar samples were labeled as “TBVM[1]” and “TBVM[2]”); (ii) “dynamically arrested vinegars” because of the full-space solidification (into the text these vinegar samples were labeled as “TBVM[3]”, “TBVM[4]” and “TBVM[5]”); (iii) “freely flowing vinegars” (i.e. TBVM[6]), high quality vinegars appearing into the desired state of homogeneous liquid. While, TBVM[1], TBVM[2], TBVM[3] and TBVM[5] were withdrawn from hermetic bottles; TBVM[4] and TBVM[6] were withdrawn directly from two different sets of barrels in which the vinegar reached 25-years of aging.

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3. Physical structure

3.1. High resolution light microscopy (HR-LM)

Vinegar samples were imaged by HR-LM at 400 and 1000 level of magnification using a Nikon Eclipse 80i fluorescence microscope equipped with DS5-M CCD camera (Nikon Instruments Inc., Melville, NY, U.S.A.). High-resolution details were obtained by an oil-immersion standard technique.

3.2. X-Ray diffractometry (XRD)

X-ray diffraction spectra were obtained with an X'Pert Pro Bragg–Brentano diffractometer (PANalytical Ins., Milan, Italy) operating in isothermal mode (25 °C) and with Ni-filtered Cu K α radiation at 40 kV and 30 mA. Samples were scanned with a step size of 0.017° (2 θ) from 3° to 80° and a time per step of 24.76 s. Pattern recognition was made

to identify the diffractive species according to the 2008 Joint Committee on Powder Diffraction Standards, JCPDS.

3.3. Soluble solids

According to Falcone and Giudici (2008), enzymatic kits coupled with a spectrophotometer UV/VIS (Jasco mod. V-550, Tokyo, Japan) were used to determine the concentration of the major solids of TBVM, i.e. D-glucose (Glu), D-fructose (Fru) and acetic acid (AcH) and their concentration was expressed as percent by weight. Standards were supplied by Roche (Darmstadt, Germany).

4. Results

Fig. 1 shows high-resolution details imaged by HR-LM at 400 (middle column) and at 1000 (right column) HR-LM magnification level. Dynamically arrested vinegars, i.e. TBVM[1] and TBVM[2],

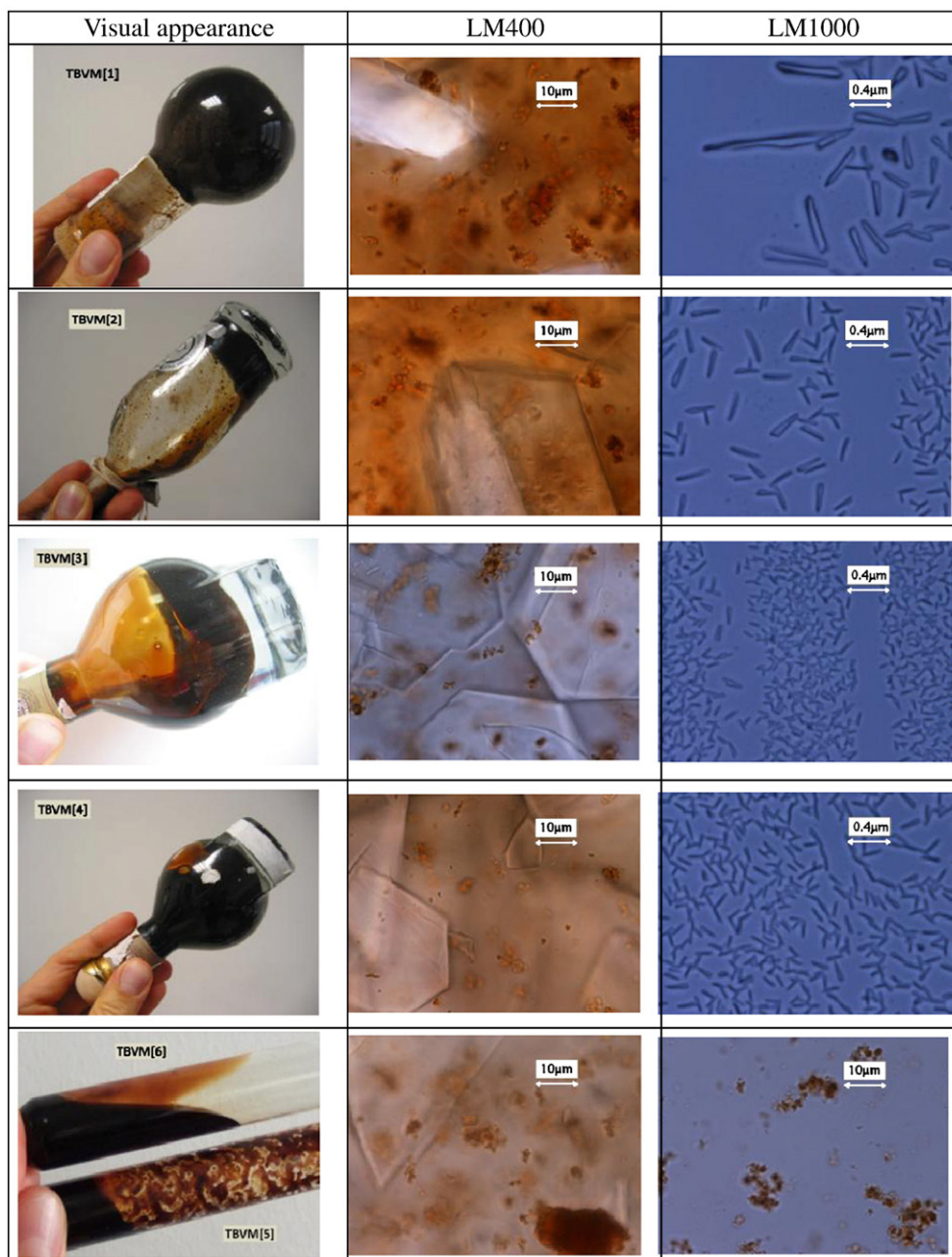


Fig. 1. Arrested structures in solidified TBVM as detected by visual inspection and High-Resolution Light Microscopy at 400 (LM400) and 1000 (LM1000) magnification level.

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