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JOURNAL OF WATER AND LAND DEVELOPMENT

e-ISSN 2083-4535

ITP-PIB

Polish Academy of Sciences (PAN) Institute of Technology and Life Sciences – National Research Institute (ITP – PIB)

JOURNAL OF WATER AND LAND DEVELOPMENT
DOI: 10.24425/jwld.2022.141564
2022, No. 54 (VII–IX): 131–137

Technological improvement of the *Sorghum saccharatum* syrup production by membrane technologies

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RECEIVED 23.09.2021 ACCEPTED 08.03.2022 AVAILABLE ONLINE 28.09.2022

Abstract: Easy-to-handle and effective methods of juice clarification and concentration by membrane technologies are still under exploration. The current article presents results of research on the technological development of an alternative natural sweetener of high biological value and improved organoleptic properties. Sorghum saccharatum stem juice is used in research. It is pre-clarified enzymatically with α-amylase and glucoamylase, clarified by ultrafiltration, and concentrated by the direct contact membrane distillation in various temperature ranges. The study shows the efficacy of membrane methods for improving juice purity, total soluble solids (*TSS*), and total sugar (*TS*) content in the syrup obtained. Clarification depends on membrane characteristics at the beginning of the process, as there are no differences at the end of it. Juice concentration at high-temperature differences allows to accelerate the process by approx. 60% comparing to low-temperature differences. A lower temperature difference (ΔT = 20–30°C) in the concentration process results in a longer process and syrup acidisation, whereas a higher temperature difference (ΔT = 70°C) affects physicochemical properties of syrup due to local overheating and formation of Maillard reaction products. The juice concentration at ΔT = 50–60°C allows to obtain high values of total soluble solids without significant degradation of physicochemical and organoleptic properties.

Keywords: juice clarification, juice concentration, membrane distillation, *Sorghum saccharatum*, sweet sorghum syrup, ultrafiltration

INTRODUCTION

The concept of high-quality and biologically safe food products which contain vitamins, macro- and microelements and other biologically active nutrients is among the current leading trends in the food industry. Nowadays, the market of sweeteners is represented mainly by natural and highly processed products, like stevia, palm and coconut sugar, and molasses [Kim *et al.* 2016]. Unfortunately, the number of metabolic-related diseases has increased dramatically all over the world during the past years. Common examples include obesity, diabetes, and cardiovascular diseases [Swaminathan 2011]. Changes in diet habits, connected with the replacement of natural products with refined or canned ones, which are almost deprived of biological nutrients, can be considered as the primary reason for the health disorders. Therefore, the development of natural origin alternative sugar substitutes that increase the biological value and diversity of food products is an essential focus of modern research [Singh, Kashyap 2020]. The establishment of alternative sugar-containing products, which in addition to sucrose, fructose and glucose, contain other naturally occurring biologically active substances, such as tagatose and psicose (allulose) is one of promising areas for

further development [Saraiva *et al.* 2020]. One of potential strategies for such studies is related to the expansion of sugar-containing functional, therapeutic and dietary food products [Au-Yeung *et al.* 2018].

A promising crop that can be an alternative source for natural sugar-containing products is *Sorghum saccharatum*. It is characterised by a short ripening period, well tolerates droughts, and provides high and stable yields of grains and green biomass. All the attributes of *Sorghum saccharatum* make it possible to grow it successfully in climatic conditions of middle and semitropical latitudes. Apart from cellulose, hemicellulose and lignins, above-ground parts of *Sorghum saccharatum* contain 8–30% wt. of sugars, 48–101 $g \cdot kg^{-1}$ dry matter (DM) of soluble carbohydrates, 40–106 $g \cdot kg^{-1}$ DM of total proteins, 24–73 $g \cdot kg^{-1}$ DM of soluble proteins, 2.0–8.3 $g \cdot kg^{-1}$ DM of free amino acids, 100–380 $mg \cdot dm^{-3}$ of ascorbic acid, 1.5–9.4 $mg \cdot dm^{-3}$ of carotene, 0.45–1.25% wt. of calcium, 0.035–0.15% of phosphorus, 16.42 $g \cdot kg^{-1}$ DM of potassium, 1.9 $g \cdot kg^{-1}$ DM of magnesium, and 2.13 $g \cdot kg^{-1}$ DM of sodium. This makes it possible to consider *Sorghum saccharatum* as an alternative raw material to sugar beet [Faheed *et al.* 2005; Kozłowski *et al.* 2009; Rakhmetov *et al.* 2018]. Another beneficial feature of *Sorghum saccharatum* is the possibility to produce syrups and molasses as liquid sweet products of high biological value. Such products contain a significant percentage of glucose and fructose which interfere with sucrose crystallisation [Dar *et al.* 2018].

Sorghum saccharatum is grown in more than 85 countries in geographical regions in Western Africa (Nigeria), Eastern Africa (Ethiopia), Central Africa (Sudan), Eastern Asia (China), Southern Asia (India), North America (USA, Mexico), South America (Argentina, Brazil), and Australia. According to data provided by the United States Department of Agriculture, the top five countries growing *Sorghum saccharatum* are the USA, Nigeria, Ethiopia, Sudan, and Mexico [Dukhnytskyi 2019]. Several countries, including the USA, China, India, and Australia, use *Sorghum saccharatum* successfully to produce syrups.

Apart from the production of liquids for food purposes, *Sorghum saccharatum* biomass and residues are used in the production of fermented products (biofuel), and industrial products from lignocellulosic waste (alcohol, bioplastic food films and coatings) [Aruna, Visarada 2018; Boboescu *et al.* 2019; Umakanth *et al.* 2018].

According to the policies on the use of Sorghum saccharatum syrup, European countries may be divided into three categories: those with the capacity to grow and produce *Sorghum saccharatum* syrups (Russia, Bulgaria), importers of *Sorghum saccharatum* syrups (Germany, France), and countries where the consumption of *Sorghum saccharatum* syrup is not reported (Ukraine, Spain, Greece).

Currently, the vast majority of countries in the world do not report on industrial production of sugar-containing *Sorghum saccharatum* syrups and juices and scientific exploration underpinned by the interest of agrarians in the future syrup production [Ratnavathi *et al.* (eds.) 2016].

Traditional technologies of sugar syrup production include thermal evaporation subject to different hardware aspects (open/vacuum/film evaporators) [Shishir, Chen 2017]. Thermal evaporation is a process that involve heating of raw juice to evaporate its solvent (water) and its further condensation. The simplicity of installations, high productivity, and sterility of the process are among the main advantages of this method. The main drawback of high-temperature concentration is partial oxidation of the heat-sensitive components (vitamins, proteins, enzymes). The decomposition of juice odorants necessitate to operate additional modules to recover odorants separately, which significantly increases energy cost [Bagger-Jørgensen *et al.* 2011]. The most considerable drawback of the thermal evaporation process is the local juice overheating which leads to the caramelisation of sugars and the formation of coloured substances, also known as the Maillard reaction. Products of this reaction cycle detract organoleptic characteristics of juice, change its appearance, induce bitter taste, and introduce juice turbidity and discolouration. They also influence the chemical composition of the final product due to reducing the content of monosaccharides and heat-sensitive substances. Therefore, to ensure the quality of final products, evaporation of juices should be carried out at lower temperatures and its duration should be shorter [Lu *et al.* 2018].

Alternative to thermal evaporation, the membrane distillation (MD) method involves reduced energy cost and maintains nutritional profile of the final syrup. The process consists in passing juice through an only-water-permeable porous material [Qtaishat *et al.* 2008]. The driving forces behind the process are determined by differences in concentrations/pressures/temperatures on both sides of the membrane. By applying a low driving force difference, the MD allows to obtain juice concentrates with total soluble solids (*TSS*) content of 60–70% and high biological value. Thus, compared to other concentration technologies, advantages of the MD are improved quality of juice concentrate, ability to achieve high *TSS* content, and the reduction of energy cost due to low driving force differences [Bahçeci *et al.* 2015; Julian *et al.* 2020].

The simplicity of installations, possibility to carry out the process at low temperatures, which ensures the preservation of odorants and heat-sensitive substances of juice, modular facilities and the ability to adjust operating parameters of the process are among the major advances of the MD compared to traditional concentration methods. A wide range of commercial membranes is available on the global market. Parameters of membranes vary and the main features of distillation membranes include hydrophobicity, monodisperse porosity, small pore tortuosity, low thermal conductivity, high thermal stability, and chemical and mechanical resistance. Additionally, a number of strategies are developed by the membrane community to improve the distillation process, such as different distillation techniques (contact distillation, vacuum, air gap), use of different membrane polymers (tetrafluoroethylene, polyvinylidene difluoride, polypropylene or use of copolymers), supporting layers and/or reinforcement agents (biodegradable cellulose fillers, nanotubes, non-woven scrims), surface modification (antimicrobial agents, selective sorbents), etc. [Khayet 2011]. Thus, the scaling up of membrane production depends on the membrane practical application only.

In the traditional technology, filtration is an essential process to clarify juices before their concentration. Basic methods use bentonite, SiO_2, gelatine, ceyssatite, and randanite filters. The other option to apply membrane techniques in the food industry is preliminary clarification of sugar-containing juices [Vaillant *et al.* 2005]. The purpose of preliminary clarification is to remove macromolecular substances (MMS, such as pectins, saponins, melanoidins) as much as possible, which further increases the purity of the final product and reduces its colour. Clarification of

juices is mainly provided by means of ultrafiltration, where the main purpose of the process is cold sterilisation. It allows for longer juice storage without the use of preserving agents.

Easy-to-handle and effective methods of juice clarification and concentration may be achieved by utilising membrane techniques to offer an alternative natural sweetener of high biological value and improved organoleptic properties on the global market. Therefore, the purpose of the work is to offer a two-step method of *Sorghum saccharatum* juice clarification and concentration with the view of increasing the quality of the syrup and the possibility of its use in various foods as a sugar-containing component or sweetener.

STUDY MATERIALS AND METHODS

JUICE EXTRACTION

Sorghum saccharatum, provided by the Institute of Bioenergy Crops and Sugar Beet of National Academy of Agrarian Sciences of Ukraine (Ukr. Instytut Bioenerhetychnykh Kultur i Tsukrovykh Buryakiv), was chosen as a pilot plant. The plant was grown on an experimental field in Ksaverivka, Kyiv District, Ukraine. Ten kilograms of stems were cleaned, grinded, and then juice was extracted by pressing and separating from the pulp to obtain 4.3 kg of juice.

JUICE PRE-CLARIFICATION

The process involved thermal coagulation of non-sugars and starch pasting. Reduction of macromolecular substances (MMS) content in the solution was performed by flocculant-antiseptic "Valeus-D" with subsequent filtration of the precipitate [Khyzniak *et al.* 2011]. Enzymatic hydrolysis of starch was conducted in two steps. Dextrinisation and simultaneous dilution with the addition of α-amylase were performed at the very beginning of the process, followed by the next stage of saccharification of dextrins to glucose with the addition of glucoamylase. The precipitate was separated by filtration. The juice was preserved and stored at –18°C for further studies [Koo *et al.* 2019].

JUICE CLARIFICATION

Juice clarification was performed by ultrafiltration (UF) to remove MMS using a dead-end type laboratory baromembrane installation (Amicon 8200 Stirred Ultrafiltration Mini Cell). Several membranes were tested to compare working parameters. Polyester membranes with a pore diameter of 0.08–0.15 μm (PET0125100) were chosen according to monodisperse pore diameter distribution, pH stability, and the capacity of ultrasound cleaning/autoclaving. The juice clarification process was conducted at pressure 0.005–0.015 ±0.001 MPa and temperature of 10 ±1°C ensured by the thermostatic cooler (Julabo F12-MA) [Zhu, Mhemdi 2016]. Membrane performance and permeate flux J_V (in $dm^3 \cdot m^{-2} \cdot h^{-1}$) was calculated using the Equation (1):

$$J_V = \frac{\Delta V}{S \cdot \Delta t} \quad (1)$$

where: ΔV = permeate volume, penetrated through a membrane of an area S during time Δt [Vu *et al.* 2020].

JUICE CONCENTRATION

The pilot direct contact membrane distillation installation (DCMD) was used to carry out the concentration of clarified juice using a membrane cell (UM – 0.2) consisting of two chambers, separated by a hydrophobic microporous membrane [Gunko *et al.* 2006]. The installation layout is described in Figure 1. A hydrophobic polytetrafluoroethylene (PTFE) microfiltration membrane with a pore diameter of 0.45 μm was chosen. The membrane was chosen according to its improved porosity (8–10%), thermal stability (up to 140°C), defined pore diameter and low thickness. The membrane was placed vertically with its active layer oriented in the direction of the hot circuit (Fig. 1).

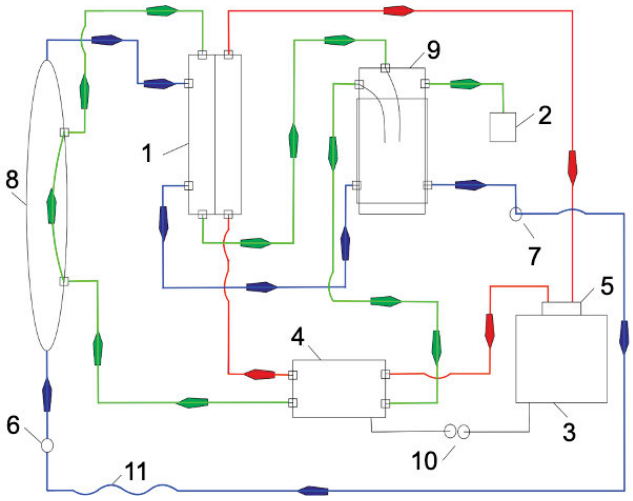

Fig. 1. Installation scheme of juice concentration process by direct contact membrane distillation; *1* = membrane cell, *2* = permeate vessel, *3* = heating block, *4* = peristaltic pump, *5* = initial solution vessel, *6* = cooler tap, *7* = recirculation valve of a cooling circuit, *8* = heat exchanger, *9* = distribution vessel, *10* = utility outlet, *11* = a circuit of cooler regeneration; hot circuit with juice is marked on red; cold circuit with cooler marked on blue; permeate circuit is marked on green; source: own elaboration

PA peristaltic pump ensured the circulation of the working solution with a volume flow of 5.76 ±0.06 $dm^3 \cdot h^{-1}$. To determine the optimal technological values of the process, the temperature of the hot circuit was regulated in the range of 40–90°C according to experimental conditions. The heating agent temperature was maintained by a heating block (WiseCircu WCB – 11). The cold circuit was cooled by glycerin from the thermostatic cooler (Julabo F12-MA). The temperature of the cooling agent was set at 10 ±1°C [Gunko *et al.* 2006].

METHODS OF ANALYSIS

The effectiveness of identified methods of clarification and concentration was confirmed by tracking changes in the content of total soluble solids, total sugars, reducing substances, sucrose, macromolecular substances, purity, colourity and pH.

Total soluble solids (*TSS*) content was measured by refractometry, whereas total sugars (*TS*) and reducing sugars by the Luff–Schoorl method [Dekker 1950]. MMS and colloids were determined by the Dumansky–Harin method in the Korolkov–Silin modification [Husiatynska *et al.* 2021]. The method is based on the properties of hydrophilic colloids to coagulate in the

solution after the addition of ethanol, and followed by the weight quantitative analysis. Syrup purity was determined by the ratio of total sugars to the *TSS*. The calculation of syrup purity allowed to evaluate the content of non-sugars in the syrup. Colourity changes were detected by photocolorimetry at 560 nm in units adopted by the International Commission for Uniform Methods of Sugar Analysis (ICUMSA). The determination of juice colourity allowed to control the visual characteristics of the final product (uniform colour, presence of colloids) and indirectly determine the presence of macromolecular substances and approximate content of total soluble solids in the solution. This shows direct relation to juice colourity. The evaluation of pH by potentiometric measurements at 20°C was also conducted to track the degree of alkalisation, whereas pH growth might have promoted the formation of colloids and degraded the quality of the final product.

RESULTS AND DISCUSSION

JUICE PRE-TREATMENT AND CLARIFICATION

The flow rate of pre-clarified juice during the clarification process showed time dependencies and reflected in Figure 2. The parameter that denoted the quality of the membrane used during clarification is water flow rate J_V. The value of flow productivity on the membrane was from 18.3 ±0.3 to 15.3 ±0.15 $dm^3 \cdot m^{-2} \cdot h^{-1}$ for enzyme-treated juice. The juice flow rate showed a significant drop for 120 min from the start, which indicated that the process was limited by the concentration polarisation process on the membrane surface. It resulted in the formation of a gel layer of juice proteins and polysaccharides on the membrane surface. This led to a secondary selectivity for the water permeation process. Statistically insignificant flow rate changes were reported for the time intervals from 120 to 150 min and from 180 min up to the end of clarification. However, no statistically significant differences could be observed in these timespans, and a moderate pore-clogging might be defined in these time intervals. Thus, clarification depended on membrane characteristics at the beginning of the process and did not show any differences at the end. Moreover, juice pre-treatment provided higher productivity of UF by decreasing the gel layer formation on the membrane during clarification. Regeneration of the UF membrane (NaOH, C = 1 M, T = 60°C, t = 60 min) allowed to increase the flow rate from 15.3 ±0.15 to 16.9 ±0.2 $dm^3 \cdot m^{-2} \cdot h^{-1}$, which enabled its secondary use for juice clarification after autoclaving.

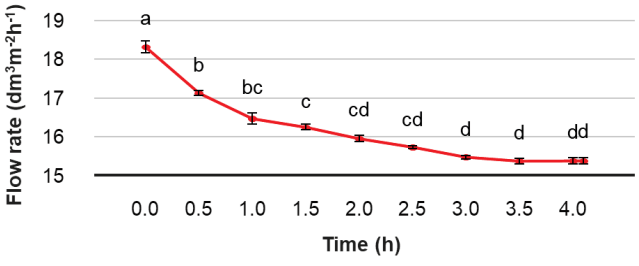

Fig. 2. Flow rate of pre-clarified juice during the clarification process; elimination ratio for juice = 50%, membrane – PET0125100, p = 0.015 ±0.001 MPa, J_V = 28.8 ±0.2 $dm^3 \cdot m^{-2} \cdot h^{-1}$; error bars are standard errors; superscript letter shows analysis of variance; the same superscript letter above bars means no significant difference at p = 0.05; the effect is significant with $p < 0.05$; source: own study

Juice pre-treatment allowed to improve its physicochemical parameters. Specific parameters are provided in Figure 3. Pre-clarification of the *Sorghum saccharatum* juice led to a significant drop in the MMS content (Fig. 3E) due to the deposition in interaction with flocculants. The enzymatic treatment of juice with α-amylase and glucoamylase and the coagulation of colloids reduced the MMS content of pre-treated juice by a quarter to the initial amount due to dextrinisation of starch with its subsequent breakdown into glucose. This led to an increase in the content of reducing substances by 88.4% to the initial amount and the establishment of an equilibrium sucrose ratio: reducing substances ≈ 35:65, characteristic of inverted syrups (Fig. 3D). This assumption is confirmed by the values of active acidity (Fig. 3G), which for invert syrups is in the range of pH ≈ 5.5. A slight improvement in growth colourity and purity enhances overall organoleptic characteristics of juice (Fig. 3F, H).

Juice clarification by ultrafiltration has a positive influence on its physicochemical parameters. Statistically significant differences, compared to pre-clarified juice, were observed in *TSS*, *TS*, reducing substances and MMS content (Fig. 3A, B, D, E), whereas the sucrose content decreased insignificantly (Fig. 3C). The purity and colourity of juice also improved (Fig. 3F, H). Differences in pH were observed neither on the pre-clarification step nor during clarification (Fig. 3G). Such dependencies show that there was no juice contamination with bio-contaminants, juice acidisation and fretting. Such relations support the hypothesis of the effectiveness of ultrafiltration treatment, since syrup's advantageous characteristics for further use include pleasant aroma, dim colour, soft viscosity, absence of residuals, and high purity.

JUICE CONCENTRATION

The main operating parameters of juice concentration are provided in Figure 4. Dependency of temperature difference on the water elimination ratio showed significant impact of the water flow rate and duration of the concentration process. According to flow rate values, several separate zones of the process may be observed. Temperature differences of ΔT = 20°C and ΔT = 30°C did not show statistically significant differences in the water flow rate, as well as in the process duration (20.1 5±0.41 h and 19.56 ±0.37 h for ΔT = 20°C and ΔT = 30°C, respectively). The same dependency on the duration was observed for ΔT = 50°C and ΔT = 60°C (9.13 ±0.23 h and 9.09 ±0.14 h for ΔT = 50°C and ΔT = 60°C, respectively). The highest water flow rate was observed for ΔT = 70°C (Fig. 5). Analysis of variances showed, that for concentration at ΔT = 20°C and ΔT = 30°C, the flow rate did not depend on the process duration. Concentration at ΔT = 40°C showed no statistically significant changes within elimination ratios of 7.5 and 75%. The other three temperature differences (50, 60, 70°C) showed no statistically significant flow rate changes only from the 45% elimination ratio up to the end of the concentration process. These data show that higher differences in the driving force of the concentration process mainly contribute to membrane fouling, hydrophilisation, pore-clogging and gel layer formation, which adversely affect the concentration process. Apart from the exploration of the concentration process flow rate, membrane regeneration and the presence of bio-contaminants after the concentration was also discovered (data not included). The analysis showed no amounts of bio-contaminants, whereas membrane regeneration (NaOH,

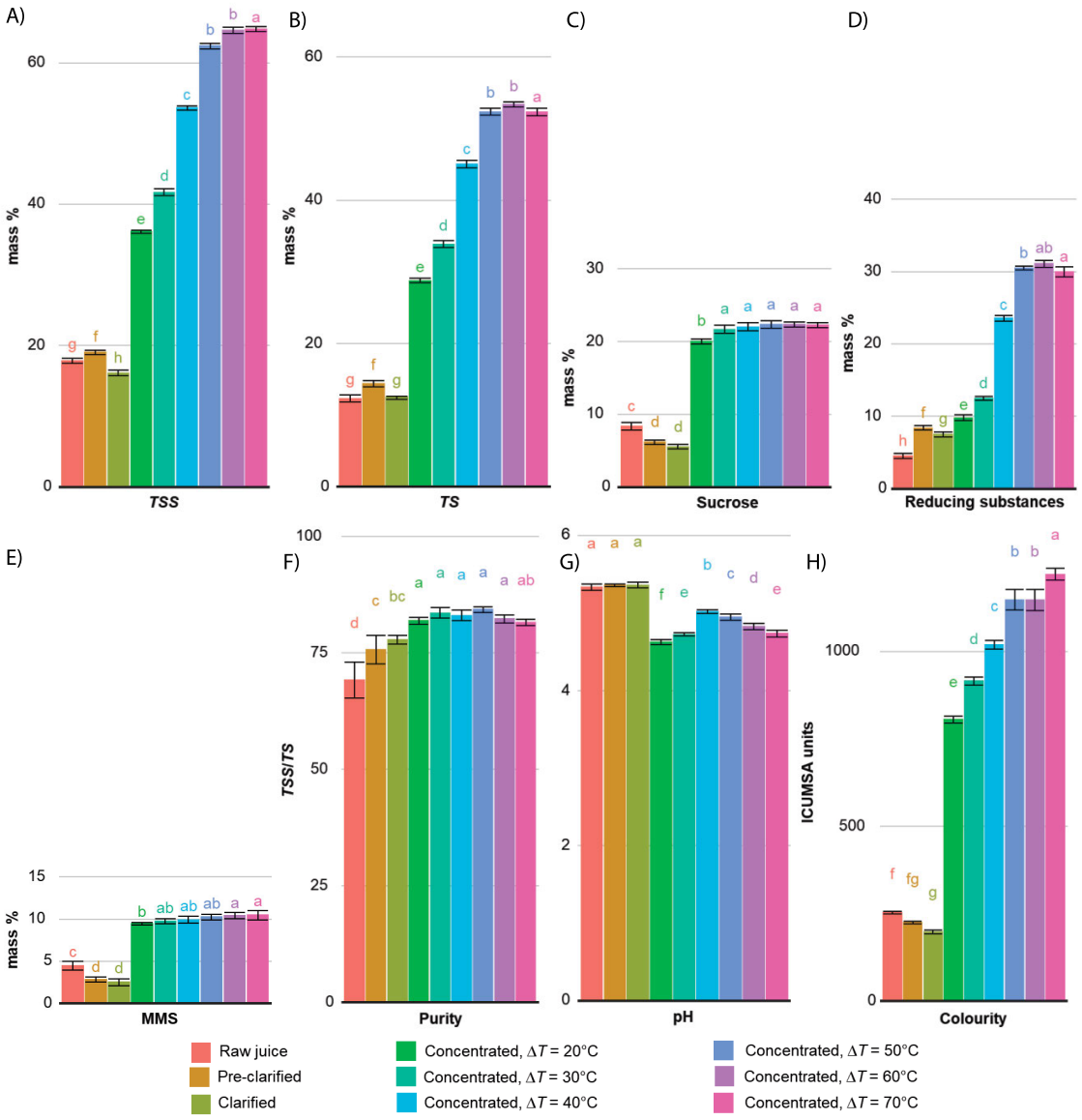

Fig. 3. Physicochemical parameters of raw, pre-clarified, clarified and concentrated juice in its quantitative characteristics: A) *TSS* = total suspended solids, B) *TS* = total sugars, C) sucrose, D) reducing substances, E) MMS = macromolecular substances, F) purity, G) pH, H) colourity; error bars are standard errors; superscript letter shows analysis of variance, the same superscript letters above bars mean no significant difference at $p = 0.05$, the effect is significant with $p < 0.05$; source: own study

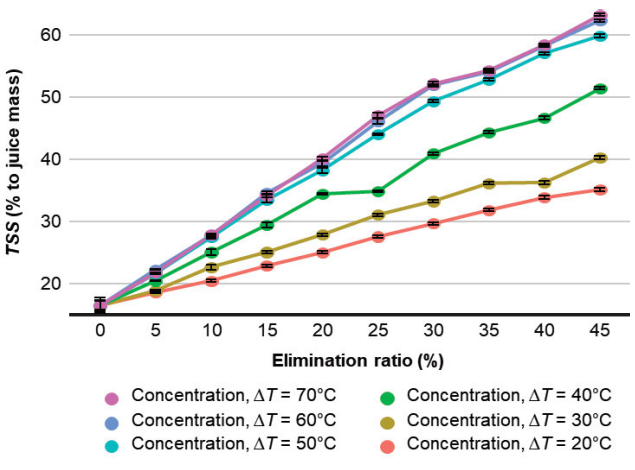

Fig. 4. Total soluble solids (*TSS*) function to elimination ratio; membrane – PTFE, J_V = 2.0–6.8 ±0.2 $dm^3 \cdot m^{-2} \cdot h^{-1}$; source: own study

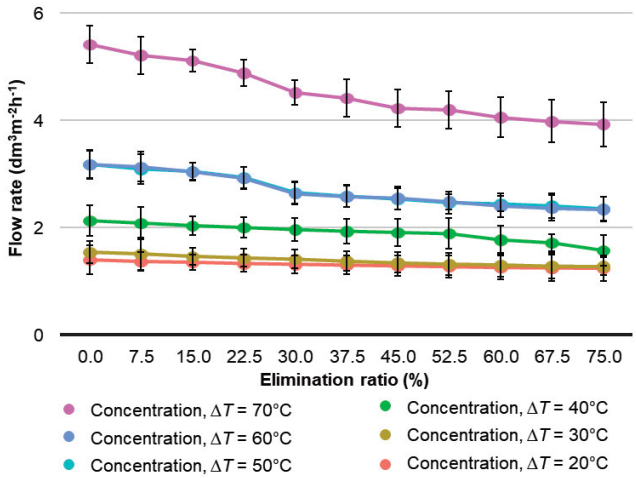

Fig. 5. Juice flow rate function to elimination ratio; membrane – PTFE, J_V = 2.0÷6.8 ±0.2 $dm^3 \cdot m^{-2} \cdot h^{-1}$; error bars are standard errors; source: own study

C = 1 M, T = 60°C, t = 60 min) allowed to restore the water flow rate by 7÷10 ±0.5%, compared to the flow rate after concentration.

The concentration process significantly affected physicochemical properties of juice. Total soluble solids increased with a growing temperature difference of process, whether at ΔT = 60–70°C no significant differences in *TSS* were observed (Fig. 3A). Total sugars value shows the same behaviour to the content of reducing substances (Fig. 3B, D). It constantly grew with the increasing temperature difference. However, at ΔT = 70°C, the total sugar content decreased, compared to lower temperature differences ΔT = 50–60°C. The sucrose content in the juice concentrate did not show significant variations with temperature differences (Fig. 3C). The content of MMS showed almost no variations with the temperature difference, which indicated that its content was the only issue in water removal (Fig. 3E). The reaction expressed by pH was highly sensitive to the conditions of concentration. The closest values of pH to raw juice were observed for ΔT = 40–50°C, whereas increasing/decreasing of the temperature difference resulted in a pH drop (Fig. 3G). Colourity was in direct dependence with the temperature difference, whereas at ΔT = 50–60°C no significant differences were observed (Fig. 3H).

Since the *Sorghum saccharatum* juice is a heterogeneous system which consists of sucrose, monosaccharides, as well as amino acids and other organic substances, irreversible changes of its composition occur over a long-term effect of elevated temperature. While analysing physicochemical parameters of juice concentrates, obtained at ΔT = 20–40°C, several drawbacks may also be observed. The low driving force of the concentration process does not allow to obtain *TSS* and *TS* values comparable to the higher one. Such operation conditions may significantly increase the primary cost of concentration, whereas organoleptic characteristics of juice will be high due to the avoidance of the saccharose thermal degradation without the growth of MMS. The other key point is that underconcentrated juices still contain water, which may promote the growth of bio-contaminants. It differs from the main hypothesis of juice concentration to enhance its storage time without the use of preservatives. Moreover, changes in the pH value for juice concentration show that juice acidisation takes place at ΔT = 20–30°C. It can be explained by the prolonged duration of the process. It adversely affects syrup quality, mainly by its inhomogeneous structure and appearance of crystallisation centres.

Under conditions of local juice overheating, the decomposition occurs of amino acids and reducing sugars that contain carbonyl groups. Thus, amino acids form aldehydes, ammonia and CO_2 from reducing sugars may form furfurol or oxymethylfurfurol. They may interact with the colouring melanoidins, enhancing juice colourity, and adversely affecting the resulting colour and taste. Such a dependency may be observed for the concentrate obtained at ΔT = 70°C, for which a slight decrease of total sugars, reducing substances, and sucrose were observed due to juice overheating. A decrease in the pH value may indirectly related to the presence of products of the Maillard reaction in concentrate.

High temperatures adversely affect juice, both in terms of changed organoleptic properties of the syrup and the safety of its consumption. The product of the first step of the Amadori reaction – N-substituted glycosylamine, is unstable. It degrades in aqueous media with the formation of dark brown Maillard reaction products. The stability of Amadori rearrangement reaction products at pH close to neutral suggested that the intermediate aminophenol was unstable, and its further conversion might occur with the formation of furfural derivatives. Arguments in favour of this were clarified in the study of Mayar's reactions [van Lancker *et al.* 2011]. The formation of furan derivatives was confirmed for fragmentation and recombination of carbohydrates during juice concentration by thermal evaporation [Seok *et al.* 2015].

CONCLUSIONS

This study shows the impact of pre-clarification, clarification and concentration on physicochemical properties of the *Sorghum saccharatum* juice depending on its conditions. Pre-clarification and clarification allowed to decrease the MMS content from 4.36 mass % to 2.86 and 2.64 mass %, respectively, with juice sterilisation. The concentration of juice by a membrane distillation at lower temperature differences allowed to increase total soluble solids and total sugars up to 50 mass %, whereas a prolonged duration of the concentration process promoted acidisation. The juice concentration at high-temperature differences allowed to shorten the process by approx. 60%, compared to low-temperature differences. However, physicochemical properties of syrup were affected by overheating and the formation of Maillard reaction products. Juice concentration at ΔT = 50–60°C allowed to obtain high values of total soluble solids (62 mass % and 65 mass %, respectively) without significant degradation of physicochemical and organoleptic properties.

ACKNOWLEDGEMENTS

For all brave defenders of Ukraine who helped to complete this publication.

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