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IMPACT OF CAUSTIC SODA SOLUTION TEMPERATURE ON EFFICIENCY OF ITS CLEANING ON SOLID SEDIMENTS AFTER COMPLETED PROCESS OF CLEANING IN CIP SYSTEM OF BREWING UNIT

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ABSTRACT

The paper presents the results of analysis concerning the impact of temperature on sedimentation in the caustic soda solution that constitute a contamination after the process of cleaning utensils and pipes for hopped wort transport in the brewery. The solution was collected from the production plant after the process of cleaning and subjected to 12-hour sedimentation and changes of the solid particles participation, their size and percentage share in the solution was determined. The study was carried out with the Shadow Sizing method. The results were subjected to statistical analysis and the surface area of the response of the relation between the time and temperature of sedimentation and the number of particles which stay in the solution was calculated. The research results proved that the temperature significantly affects the cleaning degree of solutions by sedimentation and its duration. After sedimentation in solutions, particles with the surface area from 0.001-0.003 mm^2 remain. Those particles are not subject to sedimentation and constitute a coloidal suspension in the solution.

Introduction

CIP cleaning is a basic method of cleaning devices applied in many food processing industries, inter alia, in dairy, fat, fruit and vegetable and brewery industry. The principle of that method is based on the flow of chemical cleaning solutions by production devices without the need to de-assembly them (Chung and Lai, 2008; Goode et al., 2010; Pettigrew et al., 2015). The conditions of washing and cleaning solutions as well as their concentrations depend on the cleaned device and the type of removed contaminations (Chen et al., 2012). In breweries, the so-called brewing utensils, i.e. a mash kettle, boiling pot, hop containers, sediments containers along with a cooler of hopped wort and pipes that transport require two-stage alkaline-acidic cleaning (Wawrzacz, 2006). Solutions returning after the cleaning process have a high concentration of organic compounds (ChZT and BZT_5) and general phosphorus (Janczukowicz et al., 2013) which before their exchange in the CIP station containers should be subjected to chemical degradation (Biń and Zieliński, 2000;

Krzemińska et al., 2013). Cleaning solutions are kept in CIP station containers for the period of even up to six months, which makes it possible to use many times in the cleaning processes. This solution is one of advantages of this cleaning method because it reduces the costs of chemical substances purchase and naturalization of sewage directly after the cleaning process (Olajire, 2012). On the other hand, however, it requires regeneration of solutions after the cleaning process, constant monitoring of their quality and also raises the costs related to their maintenance in high temperature reaching even 95°C (Simate et al., 2011; Wojdalski et al., 2013; Muster-Slawitsch et al., 2011).

Microbiological and physico-chemical properties of solutions decide on their quality and application for cleaning the apparatus. Firstly, the solutions must be free from microorganisms and solid sediments (Blel et al., 2015; Chen et al., 2012). Additionally, they must meet numerous requirements related to, inter alia, relevant concentration of active cleaning substances included therein and temperature, in which they are used in the cleaning process (Gésan-Guiziou et al., 2007). These parameters decide on the efficiency of cleaning of devices, affect the wettability of the cleaned surfaces, reduction of the surface tension, penetration of the solution in the structures of sediment and sediment solution ability (Goode et al. 2010). In order to meet those requirements, solutions are regenerated after each use, which consists in their cleaning from solid sediments, filling up to a determined volume and increase of their concentration by adding suitable substances (Merin et al., 2002).

Purification of the cleaning solutions after the cleaning process is carried out by gravitational sedimentation of solid sediments and their introduction to the sedimentation basin CIP before another use (Simate et al., 2011; Gönder et al., 2010; Dif et al., 2013). Popularity of this method results from low costs. Other, rarely applied methods with regard to economy are: membrane filtration and centrifugation or related methods (Kaya et al., 2009; Judd and Hillis, 2001; Räsänen et al., 2002). Studies carried out with regard to regeneration of cleaning solutions by sedimentation proved that this method does not ensure their complete cleaning. In the NaOH sodium hydroxide solution used for malt filter in the brewer after 48 hours of sedimentation still solid sediments constituting approx. 14% of the solution volume, were present. While, in the NaOH sodium hydroxide solutions and the phosphorus and nitrogen acids mixture (H_3PO_4+HNO_3) used for cleaning containers, coolant of hopped wort and transporting pipes in the brewery, approx. 2-3% of sediments remained. They constituted a suspension with the particle sizes of approx. 0.001 mm^2, which was hard to remove in the sedimentation process (Piepiórka-Stepuk, 2018). Moreover, it was proved that the contamination degree of cleaning solutions with solid sediments increases along with time of their storage in the CIP station containers (Piepiórka et al., 2014). Thus, it is justified to search for solutions which support the regeneration process of chemical solutions used many times for cleaning in the CIP systems.

Objective and the scope of research

The objective of the research was to determine the impact of the temperature of the sodium hydroxide solution on its cleaning from solid sediments cleaned from the brewery apparatus and hopping wart transport conduits. The presented tests constitute a fragment of works related to optimization of conditions and time of storing cleaning solutions in the CIP cleaning containers in breweries.

Methodology of research

Research material

Research material consisted of the NaOH (T) sodium hydroxide solution used in the brewing of the brewery for cleaning the apparatus (a mash kettle, wort boiler, hops tanks, sediments tanks, heat exchanger and pipes transporting the wort). Solution was three times collected from the CIP station container after the cleaning process (with sediment before cleaning) in the volume of approx. 2 litres and subjected to sedimentation within 20-80°C increasing the temperature every 10°C. Sedimentation was carried out in Imhoff cone with the cubic capacity of 1000 ml. The sedimentation process was carried out for 12 hours. In order to obtain variable thermal conditions, cones were soaked in water bath and kept for the duration of tests. Tests were initiated in the moment the temperature of the solution got stable on the level assumed in the research program. During the process, approximately 5 ml of the solution was collected in order to determine solid particles remaining therein, their size and amount and their distribution in the sample volume. The place of sampling was set out at a fixed height (5 cm above the solution surface). Read outs were made after 1, 2, 4, 6 and 12 hours. The reference sample consisted of a solution subjected to the assessment before the sedimentation process. Research was carried out in three iterations. Therefore, 6 readouts for the solution were obtained. The obtained results were averaged by determination of the random error expressed as a standard deviation of spread with respect to the mean. Due to coloidal properties of sediments and their thermolabile properties, the measurement of the sediments capacity after the process of sedimentation above the temperature of 50°C was impossible.

Measurement of the size and amount of particles and their distribution in the solution

Measurements were carried out with the use of the Shadow Sizing method on the DynamicStudio platform. The samples of the analysed solution were placed in 2.5 mm cuvette made of optic glass (97.40/G, Starna Scientific) and lighted with uniform dispersed laser light with the wave light of 532 nm (green light dispersed with a diffusion filter). The light source with the power of 1000 mW was behind the cuvette and in front of the cuvette there was a FlowSence 2 M camera. The camera was equipped with a matrix with a resolution of 1600×1200 pixels and a manual macro objective (Nikkor 50 mm f/1.8 macro lens) along with the set of three medium rings (12, 20 and 36 mm Kenko, DG). The idea schematic representation of the measurement and real stand for measurement with the Shadow Sizing method with the system of positioning was presented in figure 1.

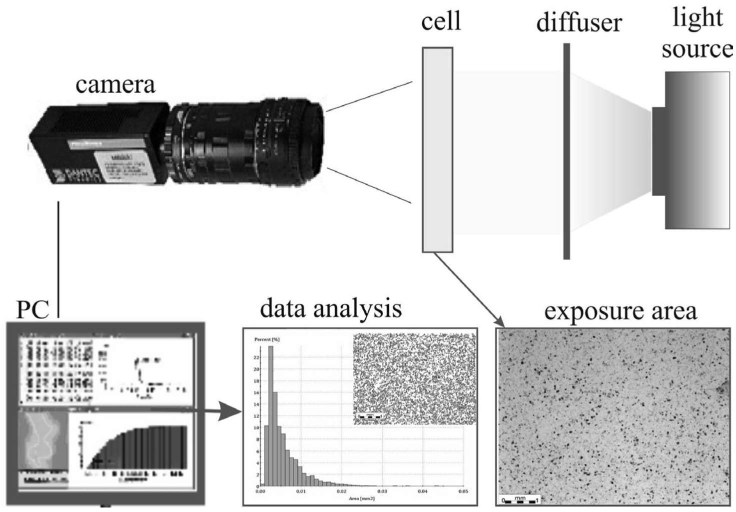

Figure 1. Stand for measurement with Shadow sizing method with positioning system

The tests were carried out after the solution was cooled down. The total capacity of the sample subjected to the assessment resulted from the cuvette capacity and was 1.4 ml. The surface area of the exposition of a single measurement was 0.1875 cm^2 which proves that the measurement capacity of the tested solution was approx. 0.5 ml. The tests were carried out in 6 replications but for each, 15 images with the frequency of recording of 1 Hz (frame per second) were taken). Thus, a measurement series of 90 images was obtained, which were analysed according to the method presented by Piepiórka - Stepuk et al., (2014) and Jakubowski et al., (2016).

Statistical analysis

The obtained results were averaged and set on diagrams (Figure 2÷5) in the form of the distribution of the particles participation in the solution with the specific surface area and the final amount of particles in the volume of the analysed sample of the solution after 12 hours of sedimentation. For the obtained results, standard deviations of the distribution in comparison to the obtained means were determined. To check out the significance of the impact of temperature on the final cleanness of the solution in Statistica 13, **Fisher-Snedecor test that verifies the zero hypothesis H_0 was carried out:** ($\mu=\mu_0$) **and alternative H_1:** ($\mu\neq\mu_0$)**, with F^{kryt}=2.3718.** Moreover, the Tukey test of multiple comparisons at the level of significance of α=0.05 was carried out. Additionally, based on the curves of the distribution, a character of the impact of particular independent variables on the amount of particles that remain in the solution after the sedimentation process was determined. The surface area of the response of the relation between the sedimentation and the temperature time and amount of particles remaining in the solution was calculated. Adequacy of the assumed model was verified through the goodness of fit to the observed ones.

Research results

Analysis of the obtained data indicates that for each temperature value in which the sedimentation process was carried out along with the lapse of the sedimentation time in solutions, the participation of big particles changed in comparison to small particles (Fig. 2). In the case of the analysed results, an increase in participation of particles of the surface area from 0.001-0.003 mm^2 out of approx. 40% in the zero sample to approx. 90% after 12 hours of sedimentation and the decrease in participation of big particles with the surface area of 0.004-0.016 mm^2 out of approx. 20% in the zero sample to approx. 5% takes place. Similar observations were presented also in the paper by Piepiórka-Stepuk (2018). Moreover, it was observed that the solution of the sodium hydroxide considerably faster cleans itself in the temperature of 60°C. For those conditions the participation of particles with the surface area of 0.001-0.003 mm^2 as early as in the first hour of the process, it raised above 70% while in the remaining temperature conditions participation of these particles was at the level of 40-60%. Moreover, it was observed that participation of small particles (approx. 95%) occurred in the solution with the temperature of 50°C.

Number of particles that remain after the sedimentation process in the analysed NaOH solution (T) indicates their final cleanness (figure 3). Outcomes of Fisher test for the analysed experimental results enable rejection of the zero hypothesis H_0: ($\mu=\mu_0$) on the equality of the average number of particles after the sedimentation process in various conditions and assumption of an alternative hypothesis H_1: ($\mu\neq\mu_0$), but the value of F statistics is F=39.4696 which meets the irregularity ($F^{kryt} < F$). The least number of contaminations after 12 hour of sedimentation was obtained for solutions with the temperature of 50°C and 60°C. For those sedimentation conditions the number of solid particles identified in 0.05 ml of the solution was at the level of 713±53 particles (for 50°C) and 738±109 particles (for 60°C). Tukey test (Table 1) does not show statistically significant differences between those two means, they differed statistically significantly from the remaining obtained results. The biggest number of particles after the sedimentation process was obtained in the solution cleaned in the temperature of 30°C and 40°C (respectively 1512 particles and 1488 particles). A big number of particles was also obtained for all high temperatures: 70°C and 80°C. It is an essential technological information because in the plant where samples were taken, solution of sodium hydroxide was kept is the CIP station container in the temperature of 80°C. Due to its big capacity which it took in the container (approx. 1000 l) the solution did not cool down in natural conditions between another cleaning processes. As a result it could have affected the low efficiency of regeneration of this mean. The presented assumption confirms the tests included in the paper by Piepiórka-Stepuk (2018).

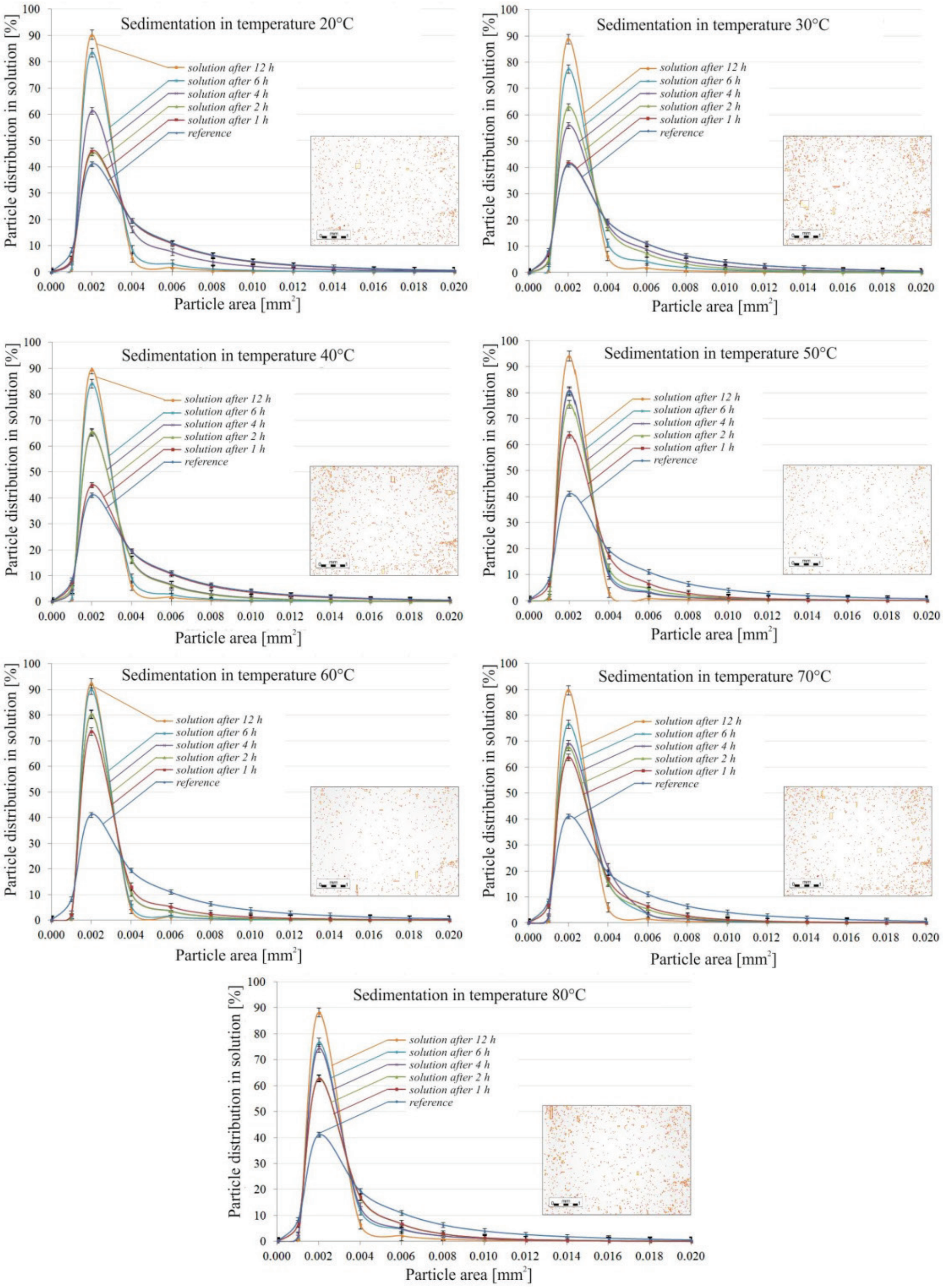

Figure 2. Changes in distribution of particles size during their sedimentation in the sodium hydroxide NaOH (T) in relation to solution temperature

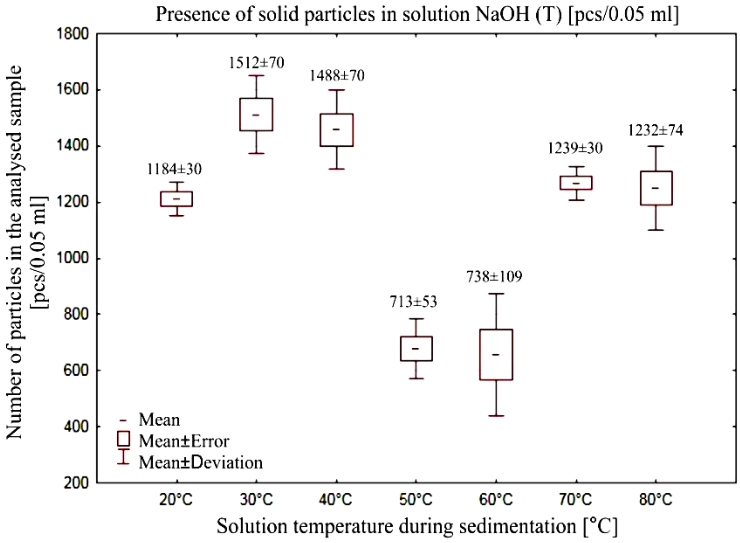

Figure 3. Number of solid particles that remain in NaOH (T) solution after 12 hours of sedimentation in various temperature conditions

The impact of the temperature on the level of purification of NaOH solution from solid particles through sedimentation may result from many factors. Reduction of the solution temperature affects, inter alia, changes of density and viscosity of the sodium hydroxide solution causing at the same time the increase of the centre resistance, where sediment particles are suspended and extension of the duration of their gravitational sedimentation. Therefore, for the temperatures of the solution within 30-40°C, the number of solid particles present in the solution after the sedimentation process is higher. Analogous analysis of those impacts would make us expect that reduction of the solution temperature during sedimentation to 20°C will cause further deterioration of the process efficiency. The obtained results, however, indicate occurrence of additional favourable impacts which would result in the reduction of participation of solid particles in the solution after sedimentation. In this case, it may result from occurrence of the so-called cold cloudiness in the sodium hydroxide solution that is characteristic for brewer's wort. Occurrence of this phenomenon results in combination of particles of sediments in bigger agglomerates and in the increase of their weight and density, which facilitates sedimentation. While, in the temperature exceeding 60°C, along with the decrease of density of the sodium hydroxide solution and sediments particles which constitute a suspension, increase of the centre internal energy takes place activating Brown movements. The solution takes up a coloidal nature. Particles of sediments start affecting each other being a subject of intense self-mixing in the solution which impedes their gravitational sedimentation. Extension of the solution regeneration results from those impacts. Dif et al., (2013) observed similar effects of the temperature impact in the process of regeneration of cleaning solutions (2013).

Table 1.
Results of significance of temperature impact on final cleanliness of solution

Temperature		HSD Tukey's test; variable number of particles (particles in NaOH solution (T); Similar probabilities for post hoc tests; Error: MS between groups = 18210, df = 35						
		20°C	30°C	40°C	50°C	60°C	70°C	80°C
1.	20°C		0.008136*	0.047421*	0.000131*	0.000131*	0.990626	0,999014
2.	30°C	0.008136*		0.992071	0.000131*	0.000131*	0.049972*	0,028062*
3.	40°C	0.047421*	0.992071		0.000131*	0.000131*	0.216275	0,136425
4.	50°C	0.000131*	0.000131*	0.000131*		0.999960	0.000131*	0,000131*
5.	60°C	0.000131*	0.000131*	0.000131*	0.999960		0.000131*	0,000131*
6.	70°C	0.990626	0.049972*	0.216275	0.000131*	0.000131*		0,999983
7.	80°C	0.999014	0.028062*	0.136425	0.000131*	0.000131*	0.999983	

* samples that differ statistically significantly

Further analyses aimed at determination of the function of simultaneous influence of time and temperature on the level of cleaning of the NaOH (T) solution in the sedimentation process. Based on the distribution of results, it was assumed that the impact of time on sedimentation may be approximated with the exponential function (Fig. 4 a) while the impact of temperature with the second degree polynomial which has its extremum within temperatures 50- 60°C (Fig. 4b). While, the Pearson correlation coefficient of the linear relation (significant for $\alpha=0.05$) for the influence of the sedimentation time on the number of particles in the solution is r = -0.8349. It indicates a strong negative relation for those variables which should be understood that along with the extension of the sedimentation process duration the number of particles present in the solution decreases. Thus, the impact of the sedimentation time on the level of the solution cleanness meets the expectations. In case of correlation of the solution temperature with the final number of particles after the sedimentation process, the Pearson correlation coefficient of the linear relation (significant for $\alpha=0.05$) is low, at the level of r = -0.2447.

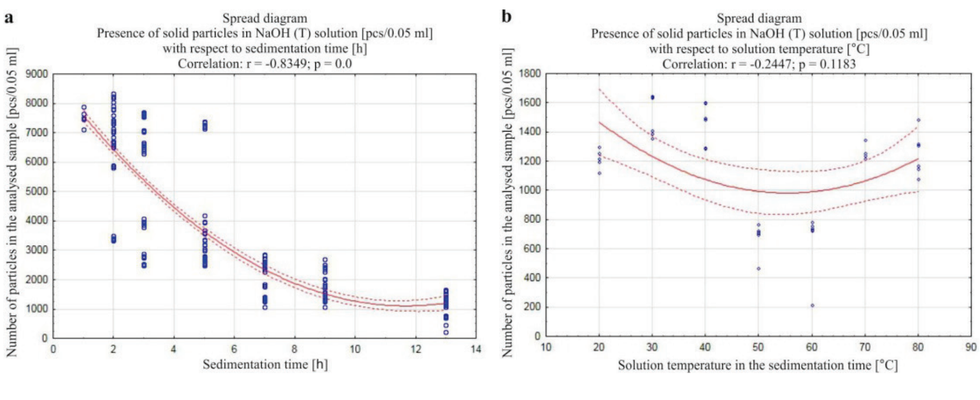

Figure 4. Spread of solid particles in NaOH solution (T) towards: a – sedimentation time (exponential distribution); b – temperature of solution in sedimentation time (polynomial)

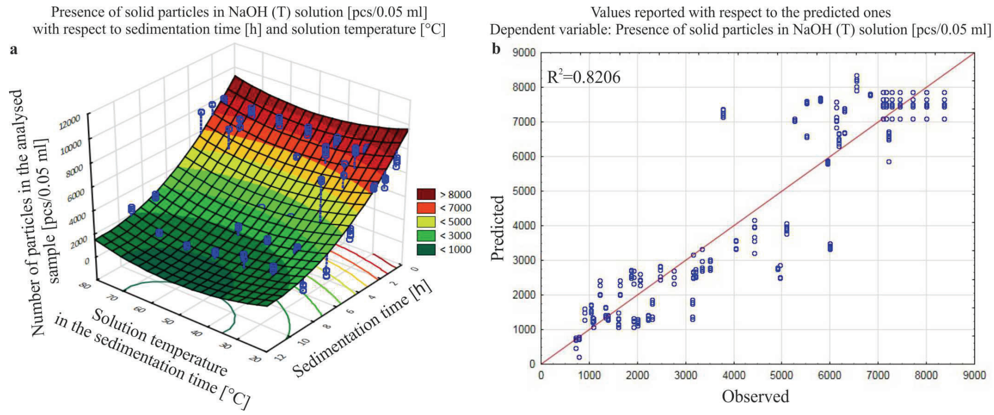

Figure 5. Relation of the number of solid particles in NaOH (T) solution with sedimentation time and solution temperature: a- surface of response of the values observed towards the predicted ones; b - diagram of spread of predicted values towards the observed ones

Figure 5a presents a spatial diagram of the observed number of solid particles which remain in the sodium hydroxide solution with respect to the predicted number of particles after the sedimentation process in the various temperature and time. Based on the obtained distribution it was read out that within temperatures 50-60°C the sodium hydroxide solution cleans up the best (the least from solid particles) and in the shortest time. In case of the tests that were carried out, for those conditions, it enables shortening of the sedimentation time by approx. 30% (approx. 9 hours). The diagram of spread (Fig. 5b) indicates adequacy of the assumed model for the obtained data which confirms the value of the coefficient of adjustment (R^2=0.8206) and location of the points of the values of remainders predicted towards the observed ones. Reduction of temperature of the sodium hydroxide solution on the time of its regeneration enters the concept of "Green Brewer" concept that assumes introduction of strategic actions in the production process which will favour reduction of the thermal energy consumption in breweries and finally will affect the reduction of CO_2 emission (Muster-Slawitsch et al., 2011; Rivera et al., 2009).

Conclusions

1. During cleaning of the sodium hydroxide solution, particles with the surface area above 0.004 mm^2 are subject to sedimentation. On the other hand, in the solution, particles with the surface area of 0.001-0.003 mm^2 remain. Increase of their participation during the experiment suggests that they are not subject to sedimentation and constitute suspension in the solution.
2. Time and temperature in which sedimentation is carried out, affect in a non-linear manner the final efficiency of cleaning the solution of sodium hydroxide of solid particles. But, the influence of time in the sedimentation process in the sodium hydroxide solution after cleaning the apparatus of the brewery has an exponential nature and depends on the solution temperature.

3. The most favourable temperature conditions for cleaning the sodium hydroxide solution from solid particles washed out of the production apparatus (brewery utensils, heat exchanger and hopped wort transport pipes) are 50-60°C. In such temperature and in the assumed technical conditions, the minimum required sedimentation time was 9 hours.

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WPŁYW TEMPERATURY NA SKUTECZNOŚĆ OCZYSZCZANIA ŁUGU SODOWEGO PO PROCESIE MYCIA W SYSTEMIE CIP NACZYŃ WARZELNICZYCH I DROGI BRZECZKI

Streszczenie. Przedstawiono wyniki analiz dotyczące wpływu temperatury na sedymentację osadów w roztworze wodorotlenku sodu stanowiących zanieczyszczenie po procesie mycia naczyń i przewodów do transportu brzeczki w warzelni zakładu piwowarskiego. Roztwór pobierano z zakładu produkcyjnego po procesie mycia i poddawano 12 godzinnej sedymentacji oznaczając zmiany udziału cząstek stałych, ich wielkość i procentowy udział w roztworze. Badania prowadzono metodą Shadow Sizing. Wyniki poddano analizie statystycznej wyznaczając powierzchnię odpowiedzi zależności między czasem i temperaturą sedymentacji a ilością cząstek pozostających w roztworze. Wyniki badań wykazały, że temperatura w sposób istotny wpływa na stopień oczyszczania roztworów przez sedymentację oraz na czas, w jakim to następuje. W roztworach po sedymentacji pozostają cząstki o powierzchni od 0,001-0,003 mm^2. Cząstki te nie ulegają sedymentacji i stanowią koloidalną zawiesinę w roztworze.

Słowa kluczowe: system CIP, ług sodowy, regeneracja, sedymentacja