pH reduction of vegetables by the application of the vacuum impregnation method

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ABSTRACT

The use of vacuum pressure to improve pH reduction of some vegetables was studied. In particular, vacuum acidification (VA) and pulsed vacuum acidification (PVA) on peppers, eggplants, mushrooms and zucchini in different operative condition were applied. The effects of the treatments on the pH reduction, kinetic, vegetable tissue structure changes and visual aspect of samples were studied. Results showed that both VA and PVA are useful methods to improve the rate of pH reduction of vegetables showing that in all cases pH values were significantly lower if compared to a conventional acidifying-dipping at atmospheric pressure. Vacuum pressure was the most important process variable able to affect the pH reduction of samples; nevertheless, the characteristics of fresh vegetable tissue structures such as porosity fraction, mechanical properties, etc., greatly affected the efficacy of the innovative treatments. For instance, in the case of zucchini slices due to the high rigidity of vegetable tissues, an increase of vacuum level of 200 mbar did not improve the pH reduction. Also, the importance of the effect of VI on visual aspect of samples was highlighted.

Keywords: vegetables; pH reduction; acid solution; vacuum impregnation; vegetable tissue structure

INTRODUCTION

pH reduction is one of the most important critical control points for the production of vegetable canned food. As reported from the Food and Drug Administration the term “acidified food” refers to foods with an $a_w$ value greater than 0.85 and a pH value at equilibrium of 4.6 or less [1]. This value allows to inhibit the germination of spores and the toxins production of Cl. botulinum. However, as reported from some authors [2] due to the high variability of pH values of fresh vegetables the limit of 4.6 is too general to guarantee the safety during industrial production. According to this idea, the New York State Agricultural Experiment Station (2005) stated that in practice the pH should be reduced at 4.2 or below for safety reasons. Several techniques are available to reduce the pH of vegetables. In particular, FDA summarized the following methods: 1) Blanching of the food ingredients in acidified aqueous solutions; 2) Immersion of blanched food in acid solutions; 3) Direct batch acidification; 4) Direct addition of a predetermined amount of acid to individual container during production; 5) Addition of acid food to low-acid food in controlled proportion to conform the specific formulations. It may be stated that all above methods are based on diffusion mechanism in which the driving force is the differences of hydrogen ions concentration between vegetable tissues and acid solution. In this way, pH reduction is affected by several variables such as shape and dimension of vegetable pieces, temperature, acid solution concentration, solution/product mass ratio, etc. Moreover, it should be considered that for heat sensitive vegetables, acidifying-dipping is the only way to reduce pH values; nevertheless, in this condition the diffusion rate may be very low significantly increasing the time length of the process. Under these consideration the development of innovative methods to increase the rate of pH reduction, also reducing the number of process variables, is very interesting for food industries. In the last years vacuum impregnation (VI) has been recognized as useful method to improve the rate of several infusion treatments [3]. VI treatments allow to increase the contact area between external solution and vegetable tissues as a consequence of capillary impregnation. The method is based on the application of two steps: 1. The immersion of vegetables inside aqueous solution and the application of a vacuum pressure for a time $t_1$ (vacuum period); 2. The restoration of the atmospheric pressure keeping the vegetables immersed in aqueous solution for a time $t_2$ (relaxation time). In the first step, gases and native liquids inside capillaries are expelled and the pores are expanded. During the second step, due to the gradient of pressure the impregnation of pores by external solution occurs and the capillaries relaxed. VI treatments have been extensively used to improve the rate of several treatments such as osmotic dehydration, the enrichment of firming agents, nutritional compounds, ingredients, etc. [3]. Nevertheless, few papers are available on the application of this method to improve the rate of pH reduction of vegetables. In this paper the application of innovative vacuum
acidification (VA) and pulsed vacuum acidification (PVA) at some vegetables also showing the response of vegetable tissues and the effects on visual aspect of samples was reported.

MATERIALS & METHODS

Zucchini, mushrooms, peppers and eggplants were purchased locally. Fresh vegetables were kept at refrigerated condition until the experiments were carried out. Vegetables were previously washed and cut in half for mushrooms and in slices for zucchini, peppers and eggplants. Acid solutions were prepared dissolving in tap water an amount of lactic acid enough to obtain a pH of 3.05±0.05. Vacuum acidification (VA) and pulsed vacuum acidification (PVA) at some vegetables also showing the response of vegetable tissues and the effects on visual aspect of samples was reported.

RESULTS AND DISCUSSION

In figure 1 the RpH values of pepper slices submitted to VA treatments at 200 and 400 mbar for a t1 of 5 minutes as a function of relaxation time are shown. It is possible to observe that RpH decreases with the increase of both vacuum level and relaxation time. Samples treated at 400 mbar showed RpH values ranged between 0.981±0.0078 and 0.929±0.00436 respectively for a t2 of 10 and 30 minutes; instead, samples treated at 200 mbar showed RpH values ranged between 0.951±0.009 and 0.894±0.007 which were equivalent at a pH values respectively of 4.95 and 4.30. According to literature these results stated that an higher vacuum level allowed to improve pH reduction as a consequence of an increase of surface contact area between vegetable tissues and acid solution which improved hydrogen ions diffusion. Also, it is possible to observe that samples submitted to acidifying-dipping for 30 minutes showed a RpH values of 0.968 which is equivalent to a pH of 4.81. An increase of vacuum period from 2 to 5 minutes significantly reduced RpH values only for samples treated at 200 mbar stating that vacuum pressure was the most important process variables able to affect acidification of peppers (data not shown). Figure 2 shows RpH values of eggplants

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\text{Eq. 2}
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\text{Eq. 3}
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\text{Eq. 4}
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slices submitted to VA with a vacuum pressure of 300 and 500 mbar and for a $t_1$ of 3 minutes as a function of relaxation time. Also in this case the use of a vacuum pressure allowed to improve the acidification rate in comparison with a conventional acidifying-dipping at atmospheric pressure. After a relaxation time of 5 minutes the samples showed average RpH values 0.86 and 0.84 respectively for samples submitted at a vacuum pressure of 500 and 300 mbar; instead, after 5 minutes of immersion at atmospheric pressure samples showed a RpH value 0.99. Also, the figure showed that no significant differences were observed as $t_2$ increased from 5 to 20 minutes probably because the complete filling of pores occurred for the lower relaxation time. Table 1 shows the kinetic parameters of “Peleg’s type model” used to fit experimental RpH values of zucchini slices submitted to PVA treatments at 200 mbar and 400 mbar. In all cases the empirical model well fitted the experimental data; in fact determination coefficient values were always greater than 0.966. In general, $K_1$ values were lower when a high vacuum level was used in accordance with an improvement of acidification rate when a high vacuum was used; nevertheless, the estimated confidence intervals showed that $K_1$ values were not significantly different. In general, this result is in disagreement with the improvement of pH reduction showed for peppers and eggplants but it may be explained taking into account the characteristics of the vegetable tissue structure. Zucchini slices showed a low porosity fraction (8.39 ± 2.22%) which could have reduced the effect of vacuum pressure on pH values. Also, taking into account that zucchini slices had two useful surface from which the impregnation occurred and that the thickness of samples was low (~ 1.5 cm), the more easy impregnation of void phase could have reduced the effect of an increase of vacuum pressure of 200 mbar on the vegetable tissue-solution contact area leading to similar acidification rate. Figure 3 shows the total volume changes of zucchini slice samples submitted to PVA at 200 mbar as a function of the liquid phase volume changes. It is possible to observe that volume liquid changes occurred with a very low total volume changes probably due to high rigidity of vegetable tissues; this could have minimized the effect of the increase of vacuum values as a consequence of a reduced deformation-relaxation phenomena during PVA treatments. However, must be considered that after 720 minutes, zucchini slices showed pH values of 4.50, 4.87 and 5.59 respectively for the treatments at 200 mbar, 400 mbar and at atmospheric pressure stating the efficacy of the innovative acidification method. Figure 4 shows the RpH values and the fit obtained by using the “Peleg type model” of mushroom samples submitted to PVA at 200 mbar and 400 mbar as a function of treatment time. The empirical model well fitted experimental data; in fact, correlation coefficients were always greater than 0.975. Samples treated at 200 mbar always showed RpH values significantly lower than those measured when a pressure of 400 mbar was used during the experiments, stating that vacuum level was directly correlate with pH reduction of the samples. For instance, after 720 minutes, RpH values of 0.818 ± 0.01 and 0.756 ± 0.02 respectively for samples treated at 400 mbar and 200 mbar were measured. In addition, when an acidification treatment at atmospheric pressure was performed for 12 h a RpH of 0.848 ± 0.041 was measured clearly showing efficacy of PVA in comparison with traditional acidifying-dipping (data not shown). Furthermore, fresh mushrooms showed a porosity fraction in the range of 35.37 ± 6.04 stating a high void phase available for the liquid impregnation. Also, liquid volume changes occurred in line with a significant total volume changes stating the low rigidity of vegetable tissues which allowed to improve the capillary impregnation as a consequence of an high deformation-relaxation phenomena (data not shown). According to these results, samples treated at 200 mbar showed a residual porosity fraction of 6% stating the high changes of the void phase as a consequence of liquid impregnation and structural compression of pores. Figure 5 shows the visual aspect of zucchini and eggplant slices submitted to PVA and VA treatments in different operative conditions. The images allow to highlight the effects of capillary impregnation and relaxation-deformation phenomena on the visual aspect of vegetable tissues. For zucchini slices impregnation occurred in the core of the slice probably due to the high porosity or low rigidity of vegetable tissues in that section of samples. Also, in terms of surface area interested by liquid impregnation, not significant differences were appreciable for samples submitted to 200 mbar or 400 mbar. Instead, for eggplant slices a higher effect of VI treatments on visual aspect was observed. The comparison of figure 5c and 5d allow to appreciate the effect of deformation-relaxation phenomena during treatment; in fact, it is easy to highlight to presence of capillaries on the surface of samples which on fresh vegetable were indistinguishable. So, for commercial reasons, the effects of VI on sensorial quality of vegetable should be considered in the vegetable food industries.
Figure 1 - RpH values of samples submitted to vacuum impregnation at 200 and 400 mbar for a vacuum period of 5 minutes as a function of relaxation time.

![Graph showing RpH values as a function of time for different vacuum pressures.]

Figure 2 – RpH values of eggplants slices submitted to VA treatments at 300 and 500 mbar for a vacuum period of 3 minutes as a function of relaxation time ($t_2$).

![Graph showing RpH values as a function of time for different vacuum pressures.]

Table 2 – Kinetic parameters of Peleg’s model used to fit RpH values of zucchini submitted to pulsed vacuum acidification at 400 mbar and 200 mbar.

<table>
<thead>
<tr>
<th>Vacuum pressure</th>
<th>$K_1$</th>
<th>Confidence interval</th>
<th>p-level</th>
<th>$K_2$</th>
<th>Confidence interval</th>
<th>p-level</th>
<th>$r^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>400 mbar</td>
<td>918.79</td>
<td>521.42 – 1316.17</td>
<td>0.0013</td>
<td>2.511</td>
<td>1.46 – 3.55</td>
<td>0.0118</td>
<td>0.9739</td>
</tr>
<tr>
<td>200 mbar</td>
<td>415.70</td>
<td>130.40 – 701.01</td>
<td>0.0010</td>
<td>3.87</td>
<td>2.80 – 4.95</td>
<td>0.0011</td>
<td>0.9666</td>
</tr>
</tbody>
</table>
**Figure 3** – Total volume changes ($\Delta V^0$) of zucchini slices submitted to PVA treatments at 200 mbar as a function of liquid phase volume changes ($\Delta V^L$).

**Figure 4** – Experimental RpH values data and “Peleg type model” of mushroom samples submitted to PVA treatments at 400 mbar and 200 mbar as a function of treatment time.
CONCLUSION

VA and PVA treatments may be considered as useful techniques to improve the rate of acidification treatments of vegetables. The impregnation of capillaries with the external solution significantly increases the surface contact area hence, the rate of hydrogen ions diffusion from acid solution to vegetable tissues. According to literature, vacuum pressure was the most important variable affecting pH reduction of samples. Nevertheless, it must be taken into account that the properties of vegetable tissues structure such as porosity fraction and its variability, mechanical properties as well as shape and size of pores and its connectivity greatly affect the efficacy of VA and PVA. Moreover, the effect of vacuum pressure on visual aspect of vegetables need to be considered because some of these could become not appreciable from the consumers.

REFERENCES