



Oil emulsion stability in electrolyzed water solutions

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Abstract:

Metastable electrochemically-activated water solutions possess unique properties that make it possible to modify food emulsions. This comparative analysis featured the stability of model oil-in-water emulsions with anolyte or catholyte as a dispersion medium, as well as the physical and morphometric profile of the emulsion system.

The research involved emulsions based on anolyte and catholyte. They consisted of refined sunflower oil, emulsifier (lecithin), and stabilizers, which were represented by sodium alginate, sodium carboxymethylcellulose, pectins, and agar. The study also covered such parameters as aggregative stability, viscosity, morphometry, oil particle size, and zeta potential.

Anolyte and catholyte affected the process of separation in the model emulsions. The samples stabilized with alginate and sodium carboxymethylcellulose proved to be the most stable emulsions while agar triggered gelation. The effect of substituting tap water with metastable electrolyzed water solutions depended on the oil proportion in the emulsion. Catholyte destabilized the samples with 20% of oil and liquified gel in the samples stabilized with agar. Anolyte was more aggressive in destabilizing emulsions with 30% of oil. The effective viscosity of these emulsions correlated with the stable phase fraction. The anolyte-based samples had low effective viscosity. The opposite results for emulsions with different oil fractions may have been caused by interface changes, i.e., surface tension, adsorption, coalescence, etc. In the emulsions with 46% of oil and animal origin emulsifier, neither anolyte nor catholyte had any significant effect on the aggregative stability of the system.

The revealed patterns can be used to control the properties of emulsion products with oil phase $\leq 30\%$, e.g., low-fat mayonnaises, sauces, emulsion drinks, etc. Metastable electrolyzed water solutions may provide a reagent-free control of properties and patterns of finished or semi-finished foods and biological raw materials.

Keywords: Electrolyzed water, anolyte, catholyte, oil-in-water emulsion, aggregative stability, scanning electron microscopy

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INTRODUCTION

The structure and properties of food products depend on such technological factors as temperature, pressure, mixing rate, etc., as well as on the interaction between ingredients, including water. Logically, modified water can change the properties of semi-finished or finished product. The method of electrolyzed water solution is quite promising in this respect. Its metastable fractions accumulate in the anode or cathode chamber of the electrolyzer [1, 2]. The resulting electrolysis produces anolyte and catholyte. These aqueous solutions possess high oxidizing or reducing abilities, respectively.

Water nanoclusters based on catholyte or anolyte were reported to show a correlation between the number of bound water molecules and the energy of hydrogen bonds between them. Experimental studies proved that water surface tension depends on the energy of hydrogen bonds [3]. Mathematical modeling demonstrated a decrease in the number of clusters in catholyte and an increase in the number of water molecules per cluster in anolyte [4]. Compared to water, catholyte has greater average energy of hydrogen bonds. For anolyte, however, the average energy is lower. This phenomenon indicates an increase or decrease in energy density, respectively.

It depends on the physiological effects produced by electrolyzed water solutions. Anolyte has acidic pH and an anomalous positive redox potential while catholyte has alkaline pH and a unique negative redox potential [5, 6].

Electrolyzed water solutions owe their useful properties to hydrogen and oxygen bubbles. Bubbles are stable due to the uncompensated electric charges on the interface between liquid and gas [7, 8]. During electrolysis, bubbles appear, grow, and detach themselves from the electrode surface [7]. Their growth rate depends on the current density and water flow rate. For instance, high current density increases gas bubble diameter while low flow rate reduces it.

Electrolyzed water solutions were reported to have a lower surface tension on the interface between liquid and air than the initial water solution. In catholyte, the surface of hydrogen bubbles started to adsorb atomic hydrogen and H_3O_2^- ions when the surface tension on the liquid-air interface went down [9]. Anolyte accumulated hypochlorite ions and hypochlorous acid molecules on the surface of oxygen bubbles. During open storage, electrolyzed water solutions interacted with air, causing spontaneous restoration of the water solution. Catholyte demonstrated a correlation between the surface potential difference and the tension [8–10]. This effect was probably brought about by the neutralization of negatively charged hydroxyl ions on the solution surface. The fact that ions accumulate on the interface and reduce the surface tension could also explain the surface-active properties of catholyte and anolyte. As a result, electrolyzed water solution may obtain emulsifying properties.

Such dispersed systems as emulsions, suspensions, and foams are the main objects of directed modification in food production. Food producers control the water phase of dispersed systems by introducing food additives with certain functions and applying various physical and physicochemical methods, including the electrolyzed water solution technology [11, 12]. Ultrasound treatment was reported to facilitate the process in emulsions [12, 13]. Related studies usually feature such physicochemical properties as pH, redox potential, water activity, and color, as well as such technological parameters as yield, emulsion stability, and consistency. As a rule, the resulting emulsions prove stable and more abundant while the texture profile remain the same. For instance, ultrasound treatment combined with electrolyzed water was able to boost the extraction efficiency and solubility of krill proteins. Their emulsifying properties, foaming ability, and foam stability improved while the particle size decreased [14]. Other studies reported other factors that affected the conformation of molecules, their volume in the solution and, eventually, the rheological properties of the biomolecular solution. These factors include polyelectrolyte concentration, temperature, pressure, low molecular weight substances, pH, and electric field [15–20].

Emulsions with a surfactant, or emulsifier, are the most common dispersed systems in the food industry. By changing the physicochemical properties of the aqueous

phase, food producers can adjust the emulsion, while giving it additional stability by applying protein or polysaccharide food thickeners. If a direct emulsion contains a high proportion of the hydrophilic phase and the emulsifier has an amphiphilic chemical structure, the system can become sensitive to such factors as pH, redox potential, electrical conductivity, ionic impurities, etc. [21]. As a result, dispersed systems based on electrolyzed water solutions have anomalous physicochemical parameters [22–25]. Their properties may differ from those of dispersed systems that were prepared using ordinary water.

Therefore, anolyte and catholyte require a deeper scientific insight into the way they affect emulsions. The current trends in fatty products dictate that food emulsions, e.g., mayonnaise and sauces, should have low energy value or that some animal ingredients should be replaced by their plant analogues [26–28]. This article introduces a comparative analysis of the aggregative stability of oil-in-water model emulsions with anolyte or catholyte as a hydrophilic dispersion medium.

STUDY OBJECTS AND METHODS

The experiment involved softened tap water (pH 7.2, redox potential +340 mV). We used a diaphragm modular electrochemical cell (Delfin Aqua, Russia) to obtain metastable fractions of electrolyzed water solution, an HI98120 device (Hanna, Germany) to measure pH, and a ST20R meter (Ohaus, China) to define redox potential. The anolyte had pH 4 and ORP +800 mV while the catholyte had pH 9 and ORP –400 mV.

Liquid fat-soluble soy lecithin (DENLEC, Louis Dreyfus Company, Brazil) served as emulsifier. To create the oil phase, we dissolved the lecithin in sunflower oil until 0.2% concentration (by weight). To obtain the aqueous phase, we dissolved plant polysaccharide in water/anolyte/catholyte until 0.5% concentration (by weight). This plant polysaccharide was a standard stabilizer for an oil-in-water emulsion. For comparison, we used several polysaccharides, namely agar (GELAGAR IT+, B&V S.R.L., Italy), sodium carboxymethylcellulose (Acucell AF2985, Akzo Nobel Chemicals A.G., Netherlands), sodium alginate (Shandong Jeling Group Corporation Inspection Report, China), as well as low-methoxylated apple pectin and apple fruit pectin (APA105, Yantai Andre Pectin Co. Ltd, China). We produced direct emulsions by pouring the oil phase into the water phase while stirring the mix with a mechanical stirrer at 1200 rpm for 5–10 min. All the samples were stored at room temperature.

To study the separation process, we left the emulsion to settle at room temperature for 6, 7, and 13 days. The stable component (V_{st}) was determined as a percentage from total emulsion volume. Emulsion stability depended on how well the polysaccharides were dissolved in water. The effective viscosity was measured using an RVDV II+Pro rotational viscometer (Brookfield, Spain). The morphological studies involved a light microscope (Altami, Russia) and a JSM-6390A scanning electron microscope (JEOL, Japan). The droplet size distribution

(diameter, μm) was obtained by morphometric analysis of microphotographs of the emulsion monolayer using the ImagoJ software. We appealed to the dynamic light scattering method to study the hydrodynamic radius of droplets in a Zetasizer Nano ZS analyzer (Malvern, UK). The same device made it possible to define the zeta potential of emulsion droplets. The experimental data were processed using MS Excel and graphs.

RESULTS AND DISCUSSION

Aggregative stability of 20% oil emulsion. We used polysaccharides because polar groups could respond to changes in the redox potential of the water solution. The hypothesis was that by replacing water with anolyte or catholyte with a modified redox potential value we would be able to affect the consistency, stability, and gelation of the resulting emulsion. However, unique properties of electrolyzed water solutions do not depend on the chemical composition of the water solution. As a result, they gradually return to the initial values of softened tap water.

The water emulsion samples (redox potential +340 mV, pH 7.2) contained alginate, sodium carboxymethylcellulose, or pectins. They were initially homogeneous, but the liquid part eventually became separated. The emulsion stabilized with agar formed a stable gel that did not separate even after 6 days of storage. Table 1 illustrates the comparative analysis of model emulsions stabilized by several different polysaccharides.

Sodium carboxymethylcellulose yielded the most stable model emulsion. Pectins, however, were responsible for the least stable emulsions, especially apple pectin, so we excluded them from further studies. Figure 1 demonstrates a comparative analysis of the experimental data.

Anolyte and catholyte had different effects on the stability of the oil-in-water emulsion. However, the severity and direction of this effect depended on the polysaccharide.

The emulsion samples stabilized with sodium carboxymethylcellulose initially looked like homogeneous thick liquid. The samples with water or catholyte released the liquid component after 7 days of storage. Their aggregative stability dropped by 20% for the water sample and by 40% for the catholyte sample. The emulsion that contained anolyte maintained its initial state. These differences may be attributed to the chemical structure of sodium carboxymethylcellulose, which is an anionic polyelectrolyte with a functional acidic carboxymethyl group.

All the sodium alginate emulsions eventually lost their aggregation stability by approximately 20% (Fig. 1). The liquid volume was the same in all the samples.

The agar-stabilized emulsion exhibited two states. If the aqueous phase was water or anolyte, it was a stable gel. If the aqueous phase was represented by catholyte, the emulsion split into dense and liquid components. In the latter case, the aggregative stability decreased by 25% by the end of the experiment (Fig. 1). This effect took place because agar gel develops together with hydrogen bonds between agarose molecules, a process that depends entirely on electrostatic interaction, not chemical one [29]. Probably, the negative redox value of catholyte reduced the number of hydrogen bonds, which, in its turn, reduced the stability of agar gel. In terms of emulsion stability, the sample with sodium carboxymethylcellulose and anolyte demonstrated the best results, provided that the purpose was to maintain the liquid state of the emulsion.

Table 1 Stable component vs. total emulsion stabilized with different polysaccharides stored at room temperature, %

Storage time	Polysaccharide	Alginate	Sodium carboxymethylcellulose	Apple pectin	Fruit pectin	Agar
0 days		100	100	100	100	100
6 days		85	98	48	73	100

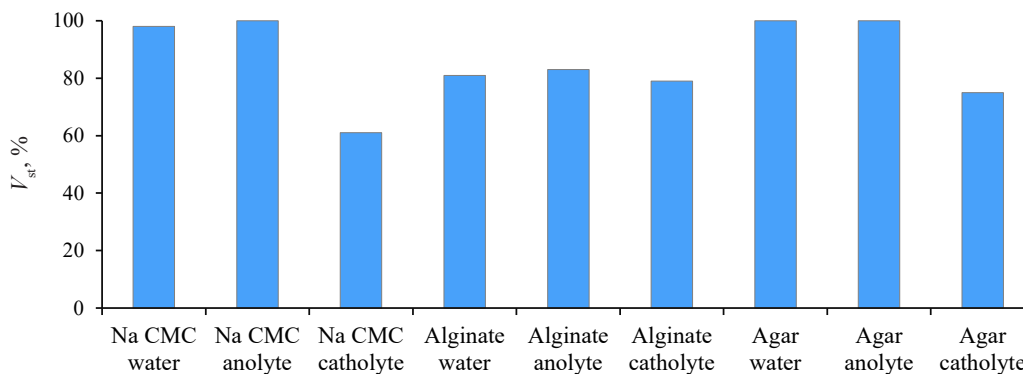


Figure 1 Stable component (V_{st} , %) vs. total emulsion (20% oil) stabilized with sodium carboxymethylcellulose, alginate, or agar after 7 days at room temperature: water – softened tap water (redox potential +340 mV, pH 7.2); Na-CMC – sodium carboxymethylcellulose; anolyte – oxidized fraction of electrolyzed water solution (redox potential +800 mV, pH 4); catholyte – reduced fraction of electrolyzed water solution (redox potential –400, pH 9)

Anolyte and catholyte failed to increase the stability of the sodium alginate emulsion.

Figure 2 demonstrates the state of the stable component in these two emulsions for 13 days, which gives an idea of how the composition stability may change during longer storage.

The curves in Fig. 2 clearly demonstrate that the stability trends for 7 days of storage persisted for 13 days. Catholyte triggered separation in both types of emulsions. This feature was most pronounced for the sample stabilized with sodium alginate. The samples with anolyte and softened water demonstrated the same changes. The emulsions stabilized with sodium carboxymethylcellulose had the best results.

In this experiment, we studied the effect of electrolyzed water solution on emulsions in a model oil-in-water system with 20% of oil and 0.2% of liquid lecithin as emulsifier. Catholyte, being the reduced fraction, accelerated separation, destroyed the emulsions stabilized by sodium carboxymethylcellulose or sodium alginate, and prevented gelation in the agar emulsion.

Aggregative stability of 30% oil emulsion. Low-calorie mayonnaises and sauces have the mass fraction of fat between 15 and 40%. We raised the fatty phase proportion to 30%. The hypothesis was that, by lowering the aqueous phase, we could affect the emulsion properties. This experiment involved only sodium carboxymethylcellulose because this polysaccharide had proved more active in stabilizing the oil-in-water system. The oil phase was prepared in line with the same method, and the mixing procedure also remained the same as described above, but the storage time was as long as 16 days (Fig. 3).

The catholyte-based sample had the best preservation effect on the emulsion with 30% of oil and sodium carboxymethylcellulose: no signs of separation were registered during the entire storage period. This effect was opposite to the one we observed when the oil phase was 20%. In that case, catholyte was more destructive. Anolyte also produced a different effect (Fig. 3), reducing the stable fraction to 30% of the total emulsion volume. The samples with water demonstrated the same kinetics at both oil concentrations.

By bringing up the oil phase proportion from 20 to 30%, we changed the conditions on the interfacial surface, i.e., surface tension, adsorption, coalescence, etc. Our results were consistent with the effect of anolyte and catholyte on the structure and pattern of proteins and polysaccharides in water solutions reported in [30, 31].

A relatively high content of the dispersed medium might have affected the electrostatic interactions between the molecules of stabilizer biopolymers, their aggregation, and solvation. Probably, it also changed the properties of the hydrophilic molecules of the emulsifier on the interface between oil and water.

The first three days of storage saw no changes in the emulsion stability (Fig. 3) but the effective viscosity was significantly different. This indicator was 3706 mPa·s for the catholyte sample, 3544 mPa·s for the water sample,

and 3316 mPa·s for the anolyte sample, respectively. The stability of each sample depended quite strongly on its effective viscosity. Therefore, the higher the initial effective viscosity value, the greater the stable component volume during storage.

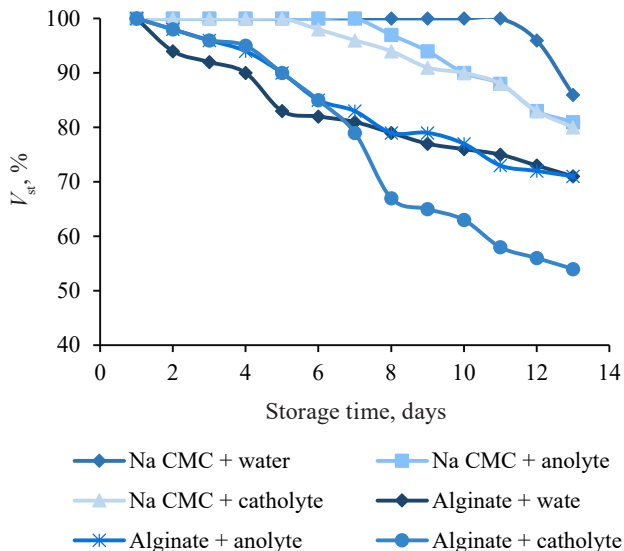


Figure 2 Stable component (V_{st} , %) vs. total emulsion (20% oil) stabilized with sodium carboxymethylcellulose or sodium alginate during 13 days at room temperature: water – softened tap water (redox potential +340 mV, pH 7.2); Na-CMC – sodium carboxymethylcellulose; anolyte – oxidized fraction of electrolyzed water solution (redox potential +800 mV, pH 4); catholyte – reduced fraction of electrolyzed water solution (redox potential –400, pH 9)

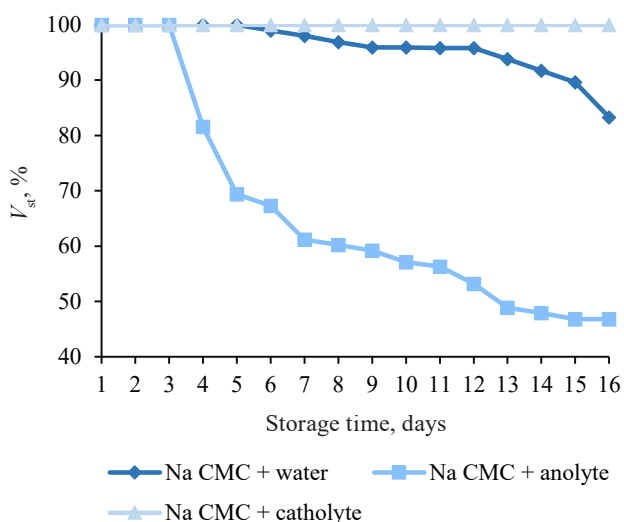


Figure 3 Stable component (V_{st} , %) vs. total emulsion (30% oil) stabilized with sodium carboxymethylcellulose or sodium alginate during 16 days at room temperature: water – softened tap water (redox potential +340 mV, pH 7.2); Na-CMC – sodium carboxymethylcellulose; anolyte – oxidized fraction of electrolyzed water solution (redox potential +800 mV, pH 4); catholyte – reduced fraction of electrolyzed water solution (redox potential –400, pH 9)

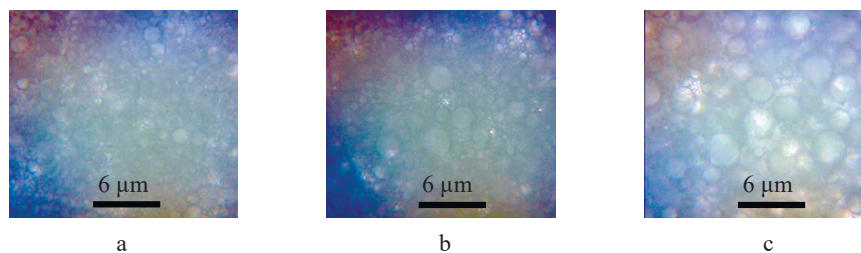


Figure 4 Emulsion (30% oil with lecithin) stabilized by sodium carboxymethylcellulose after 3 days of storage. The microphotographs were obtained in a light microscope at a magnification of 200X: A) emulsion based on softened water (redox potential +340 mV, pH 7.2); B) emulsion based on anolyte (redox potential +800 mV, pH 4); C) emulsion based on catholyte (redox potential –400, pH 9)

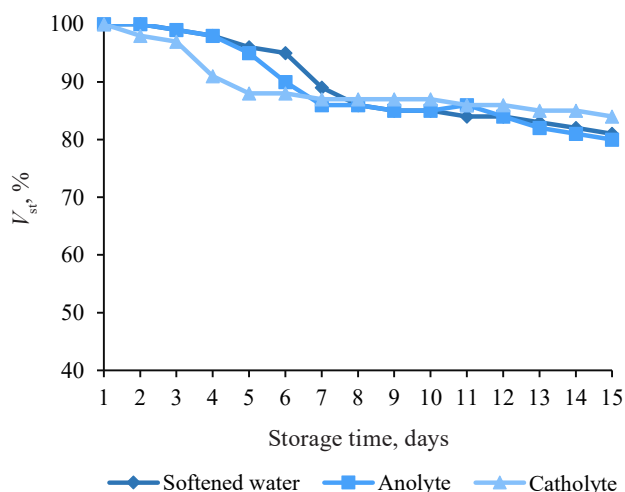


Figure 5 Stable component (V_{st} , %) vs. total emulsion (traditional mayonnaise) during 15 days at room temperature: softened tap water (redox potential +340 mV, pH 7.2), anolyte (redox potential +800 mV, pH 4), catholyte (redox potential –400, pH 9)

Under a light microscope, the samples with catholyte and anolyte demonstrated larger particles in the emulsion, compared to water-based emulsions. This trend was the strongest in the catholyte sample (Fig. 4).

This comparative experiment studied aggregative stability on model emulsions with 20 and 30% oil which were stabilized with plant polysaccharides. The aqueous phase consisted of softened water, anolyte, or catholyte.

Anolyte or catholyte affected the stability of the model system. The effect was multidirectional and depended on the concentration of oil.

Aggregative stability of mayonnaise. This part of the research featured emulsions that simulated low-energy mayonnaise or sauces and contained only plant ingredients. Such formulations follow the current trends in functional nutrition, e.g., developing new vegetarian fatty products. However, traditional mayonnaise is still in great demand.

Here, we studied the effect of replacing water with anolyte or catholyte on the aggregative stability of the emulsion system. Its composition was the same as in the target product, e.g., a popular Lyubitelsky mayonnaise (oil

content of 46%) [32]. The samples were prepared according to the previously described procedure, which started by mixing together water-soluble ingredients.

The obtained dependence (Fig. 5) made it possible to evaluate the stability of mayonnaise prepared with anolyte or catholyte during 15 days of storage.

The similarity between the aggregation stability curves (Fig. 5) indicates that the fractions of electrolyzed water solution had almost no effect on the multicomponent mayonnaise. The mayonnaise was a complex system with a high oil concentration (46%) and an emulsifier in the form of animal phospholipids. All the mayonnaise samples remained highly and equally stable throughout the entire storage period.

The zeta potential of mayonnaise particles confirmed this conclusion. This parameter characterizes the stability of dispersed systems. The method of electrophoretic light scattering showed the following values: -40 ± 3 mV for the mayonnaise with softened water, -42 ± 3 mV for the mayonnaise with catholyte, and -38 ± 2 mV for the mayonnaise with anolyte.

Figure 6 shows microphotographs of emulsion particles in the traditional mayonnaise.

Figure 7 illustrates the morphometric analysis of the microphotographs in Fig. 6. The mayonnaise monolayer revealed that the particles were distributed heterogeneously. However, we detected no significant difference between the samples. These data indirectly confirmed the results of the stability experiment. The particles could be divided into three size groups. The most representative group consisted of droplets with the diameter of 0.6–0.9 μm .

The dynamic light scattering method confirmed the morphometric results. Figure 8 shows that each sample contained several groups of particles. The largest group consisted of droplets with the hydrodynamic diameter of 300–700 nm. The fraction of micron particles was also quite large, which also confirmed the morphometric data (Fig. 7). In addition, the research revealed an insignificant number of nanometer-sized particles that the light microscope could not identify because the method was limited in spatial resolution.

Considering that the anolyte or catholyte effect occurs at the interface between oil and water, we had to study the fine surface structure for an individual oil drop using the method of scanning electron microscopy.

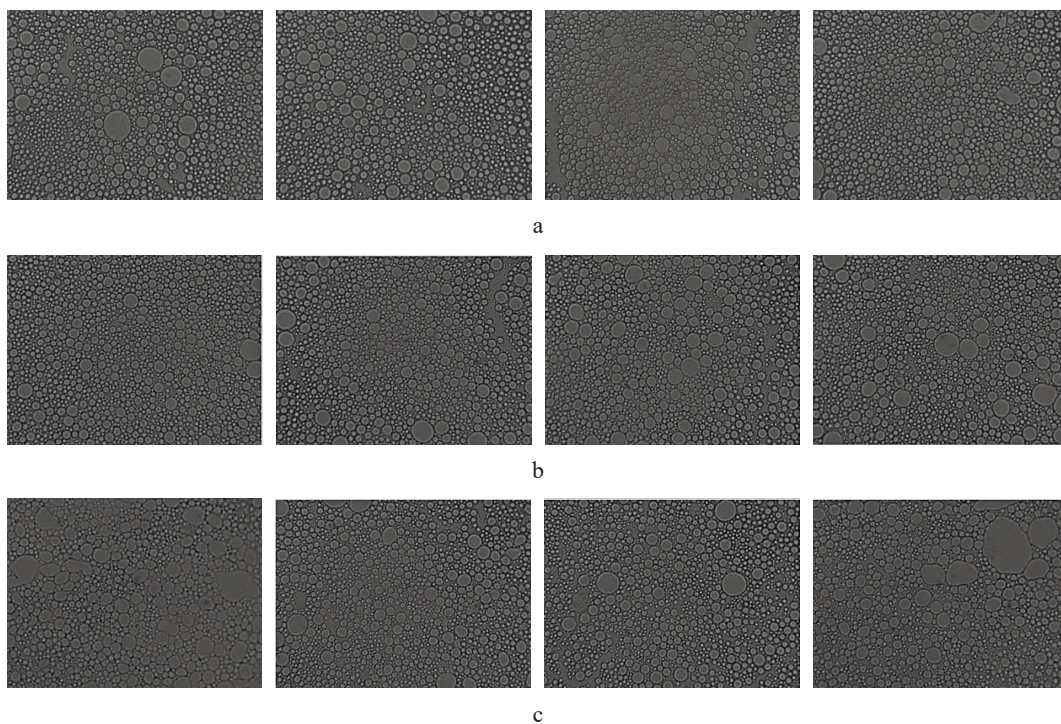


Figure 6 Monolayer of mayonnaise particles, microphotographs: samples prepared with water (a), anolyte (b), catholyte (c). The top side of all frames is 50 μm

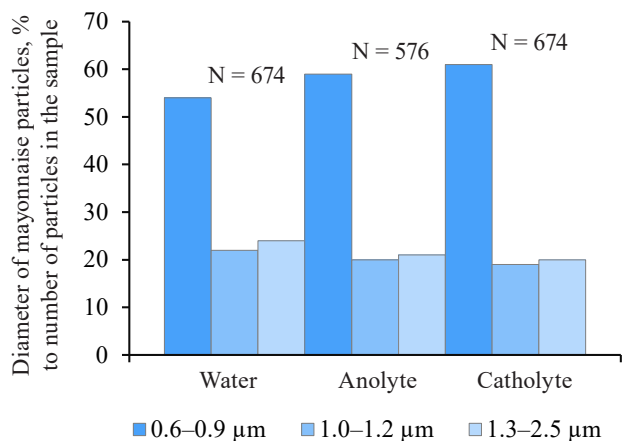


Figure 7 Diameter of mayonnaise particles after ImagoJ processing, % to number (N) of particles in the sample: softened water (redox potential +340 mV, pH 7.2), anolyte (redox potential +800 mV, pH 4), catholyte (redox potential –400, pH 9)

Figure 9 shows the results of the ultrastructural study. The microphotographs demonstrate the characteristic features of each mayonnaise sample.

The mayonnaise emulsion with water contained micron, submicron, and nano-sized particles. The high magnification revealed cavities that may point at fragmentation.

The ultrastructure of the emulsion particles with anolyte was similar to that of the previous sample but for larger particles of several microns in size. The catholyte sample also had similar visual characteristics but contained some large fat droplets which exceeded several microns.

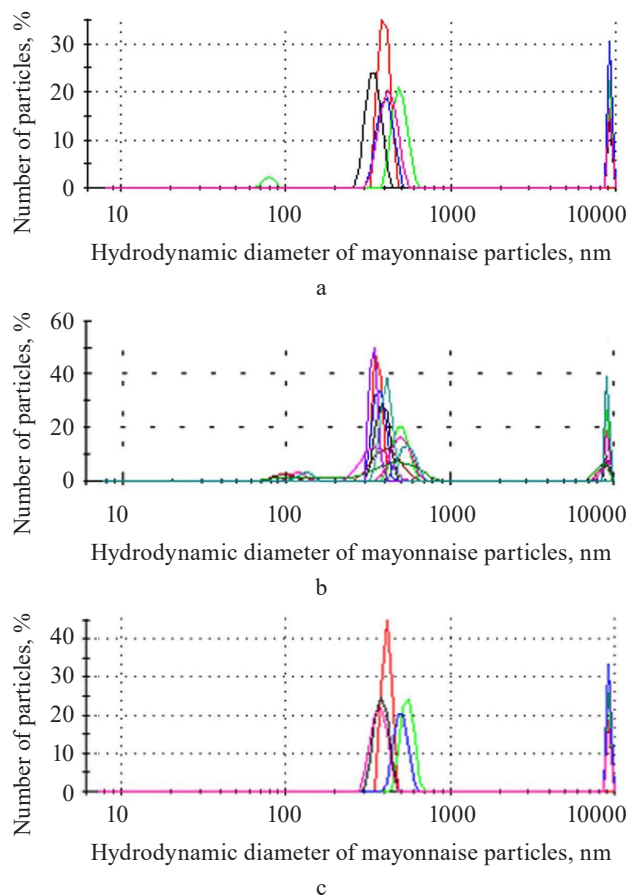


Figure 8 Hydrodynamic diameter (nm) of particles: samples prepared with softened water (a), anolyte (b), catholyte (c). The data were obtained by dynamic light, mead value from multiple tests for each sample

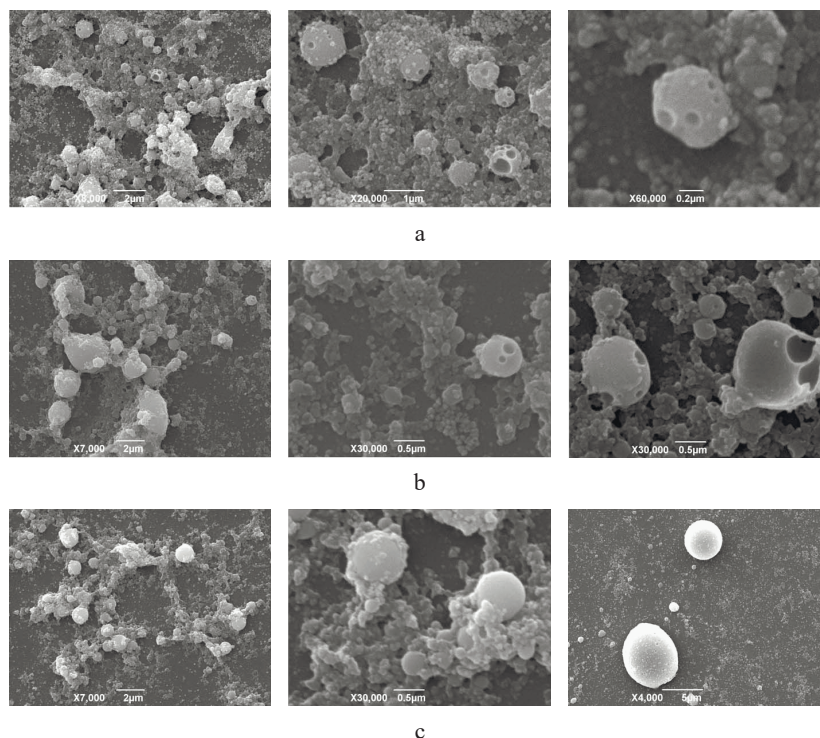


Figure 9 Mayonnaise emulsion obtained by scanning electron microscopy in the secondary electron mode. Microphotographs samples prepared with water (a), anolyte (b), catholyte (c)

These results were consistent with the micrographs and the dynamic light scattering method. Apparently, the pronounced effect of anolyte or catholyte on the aggregative stability of mayonnaise emulsion disappeared when the oil content was high and/or the mix involved a surfactant represented by phospholipids of animal origin. The effect of anolyte/catholyte on phospholipid emulsifier requires a more comprehensive study. Prospective research might include phosphatidylcholine or its derivatives, both fat-soluble and water-soluble. In addition, catholyte exhibited some antioxidant properties, which suggests that electrolyzed water solutions can protect emulsion-based products from oxidative processes during storage.

CONCLUSION

Electrolyzed water solution was able to serve as a hydrophilic phase in an oil-in-water model emulsified with fat-soluble lecithin and stabilized with polysaccharides of plant origin. It demonstrated a multidirectional effect depending on the oil phase proportion.

The catholyte fraction with a negative redox potential caused gradual separation in the emulsions with a 20% share of sunflower oil stabilized by sodium carboxymethylcellulose or sodium alginate. A similar composition, but stabilized with agar, resulted in gelation, which did not occur when softened water was replaced with catholyte.

The samples with 30% of oil demonstrated an opposite effect: it was anolyte with high redox potential that destabilized the emulsion. After separation, the volume

of the stable phase correlated with its initial effective viscosity. In other words, catholyte provided a more stable emulsion whereas anolyte lowered aggregation stability. Perhaps, the opposite results for the sample with 20 and 30% of oil could be explained by the changes in the interface conditions, i.e., surface tension, adsorption, coalescence, etc.

To sum it up, anolyte and catholyte fractions of electrolyzed water solution did not affect the stability of traditional high-fat mayonnaise emulsified by animal phospholipids.

Therefore, the practical application of anolyte and catholyte as means of reagent-free control of oil-in-water food emulsions is limited to low-fat products, e.g., mayonnaise sauces, emulsion drinks, and cocktails.

CONTRIBUTION

A.G. Pogorelov developed the research concept and wrote the manuscript. L.G. Ipatova interpreted the obtained data, designed the experiment, and performed the experimental studies. A.I. Panait prepared the samples for optical microscopy and processed the obtained results. A.A. Stankevich was responsible for electron microscopy. V.N. Pogorelova provided the physicochemical measurements and obtained the electrolyzed water solutions. O.A. Suvorov processed the materials and proofread the manuscript.

CONFLICT OF INTEREST





The authors declare no conflict of interests regarding the publication of this manuscript.

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