Iranian Polymer Journal
14 (1), 2005, 5-13

Evaluation of Changes in Properties of PA/PE Film Used for Vacuum-packed Lactic Acid Cheese

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Received 2 January 2002; accepted 28 September 2004

ABSTRACT

The subject of research was to assess the changes of properties of PA/PE packaging film used for vacuum packing of lactic acid cheese. The research investigated the influence of storage time, low temperature and the presence of lactic acid produced by lactic fermentation bacteria on the packaging film features such as barrier properties towards water vapour and the degree of swelling. The assessment comprised period of 14 days of cheese storage at the temperature 4±2^{o}C. At the same time, the tests in model conditions were conducted, consisting of storage of the packaging film filled with solutions of lactic acid and distilled water at this temperature. The obtained results showed that during period of 14 days of lactic acid cheese storage, there had occurred the increase and decrease in the permeability of PA/PE packaging film coming from the stored cheese. The results achieved from the study revealed a difference in the permeability and the degree of swelling of the films coming from the cheese and from packaging of bags filled with lactic acid. The study revealed the influence of even a small fluctuation of acidity on the level of changes of permeability of the film regarding water vapour in the model conditions. In packages taken from the refrigerated lactic acid cheese a significant dynamic relation was discovered in the increased degree of swelling up to 7 days of storage in comparison to the results achieved in the model condition. The study shows that lactic acid is not the only factor determining the dynamics relation in the changes of permeability of the films during storage of the cheese.

Key Words:
PA/PE film;
permeability;
swelling coefficient;
lactic acid cheese.

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INTRODUCTION

The interaction between food products and their packaging determines the safety of the stored food product.

This problem becomes significant when the packaging used for the food products is made from polymers such as: PA/PE, LDPE, HDPE, LLDPE, PCV, and PVDC.

There are ways to define the interaction between a food product and its packaging [1-4]. The definitions concentrated more on the changes caused in polymers by the bi-directional contact than observing the multi-directionality of these effects.

Among the described interactions, an important role is played by the phenomena connected with the penetration of water phases into the polymers, which washes off the monomers, with their subsequent migration to the food product [5-11].

Research into the barrier features the diffusion of organic compounds through foil, laminates and single polymers and for the most part do not address the physico-biological interactions, but concentrate on the evaluation of the influence of temperature on said changes [12-15].

Frequently the topics of research concerning packaging involve the sorption of steams, gases and organic compounds through the polymers [16-21].

However, for the quality of the packaged product, there are important phenomena connected with the influence of the penetration of liquids into the polymers and the influence of said penetration on the properties of the packaging.

Polyethylene is a polymer used in food packaging and is a vital component in many laminates used for this purpose.

To secure the quality of the packaged food products, the most important property of polyethylene i.e., its ability to allow the penetration of water steam and gasses must be considered.

The permeability of the film used in packaging with regards to water vapour is an important parameter which influences the dynamics of the changes connected with microbiological processes occurring on the surface of the product. The changes in permeability can bring about the microbiological changes in the product. Our earlier studies [22-23] suggest a disparity emerging in the reaction of microflora on the surface of the product during the refrigeration storage between the vacuum packed cheese and those that are hermetically sealed. The research works of Deligvere et al. [24] and Nicolaci et al. [25] confirm the influence of both factors, i.e. microflora products and the packaging system, on the quality of stored food products.

Our study reports the influence of metabolism of the microflora in cheese. The changes in the permeability of the film point to an important difference in the permeability of carbon dioxide after 7 and 14 days of storage in hermetically sealed packages [26]. The stated changes in the permeability of the film after storing the product were several times higher than the results found in the model condition by Lewicki [27]. This confirms the clear influence of the metabolites, produced by the surface of microflora that are dissolved in the water phase, on the properties of the film under study.

Among the metabolites present in the first phase of the hermetically packing cheese of PA/PE film, lactic acid dominates as a product of metabolism of the surfaces of microflora. At the same time, one can also confirm the presence of other organic acids such as acetic acid which is metabolized by bacteria of the E coli and staphylococci group. However, the evaluation of the influence of lactic acid on the changes of permeability of water vapour through the packaging is highly significant due to its high initial contact with the product.

In order to investigate the changes in the packaging resulting from the changes in the water phase of the cheese we made a denotation of the degree of swelling and permeability of water vapour in the model environment and the real environment to determine the critical point which may influence the development of the cheese s microflora when hermetically packaged in PA/PE film.

EXPERIMENTAL

The study was conducted in a model condition and the same type of film used to package and store the cheese passed our confirmation. The packages used in the study were in the shape of a bag with multi-layer PA/PE barrier film with a thickness of 80 μm. PEFLEX ANP 80 M-PA/PE 2060 is used to package lactic acid cheeses. This is an extruded multi-layer polyamide-polyethylene laminate.

The packaging is characterized by the following qualities connected with the permeability of gasses and steam:

- water vapour permeability: 1,7 g/m^2 × 24 h × bar (23ºC,75% r.h.)
- oxygen permeability: 56 cm^3/m^2 × 24 h × bar (23ºC,75% r.h.)
- carbon-dioxide permeability: 168 cm^3/m^2 × 24 h × bar (23ºC, 75% r.h.)

The laminate is allowed to come in contact with the food products by the FDA-BGA-EC 90/128/EEC.

Lactic acid cheeses are packaged by removing 95%

of the air using 0.80-1.00 Pa with an Henkelman-vacuum system type 600 packing machine.

The materials studied were in the form of PA/PE film taken from vacuum packed cheese and film from hermetically closed bags containing solutions of lactic acid with 4.3, 4.7 and 5.2 pH levels. The selected pH levels matched the average level of acidity obtained in the cheese stored in a refrigerator [22].

Samples of the packages were selected for the study from each batch of cheese stored in a refrigerator for 24 h as well as those stored for 7 and 14 days.

The bags were filled with solutions of lactic acid in amounts equal to those, which are produced by lactic acid cheese at different pH values of the environment. These concentrations were equal to adequately 0.55% v/v , 0.24 % v/v, and 0.08%v/v .

The bag filled with distilled water constituted the control sample while assessing the degree of swelling of PA/PE film.

The bags filled with lactic acid and distilled water were also chilled at a temperature of 4 –2 C and were selected for study after 7 and 14 days of storage.

In the studied packaging film, the permeability of water vapour, q, was denoted as well as the level of swelling. The denotation of the permeability of water vapour was done using the bag method with dried silica gel as a filler [28]. A bag was prepared for the studied film, which was stored in cellular thermostat at 38 C (–0.5 C) with a relative humidity of 90% (–2%) for 24 h. For the triple measurement of the mass, scales with a sensitivity of 0.0001 g. were used. The permeability of the water vapour through the foil was calculated using the following equation:

$$q= 240 \times \Delta m/S \times \Delta t \qquad (1)$$

where Δm- the growth of the mass of vessel with film over a given time (mg);

Δt- the time during which the growth of the vessel occurred (h);

S- the surface of the tested sample (cm^2).

To determine the level of swelling the value of the coefficient α was calculated using eqn (2):

$$\alpha= (m_x - m_0/m_0) \times 100\% \qquad (2)$$

where m_x is the mass of the laminate sample with an area of 25 cm^2 taken from the cheese packaging or the PA/PE bag after storage in a refrigerator for time x (7 or 14 days); and m_0 is the mass of the film sample taken from the cheese package on the day the cheese was purchased.

In lactic acid cheese the amount of water was noted. The amount of water was denoted on the basis of the denotation of the dry product by weighing it on a drying-scale. Samples of lactic acid cheese with a mass of 3 g were dried with sand at a temperature of 102 C (–2 C) with a precision of 0.0001 g. The results are given in grams of water per 100 g of cheese.

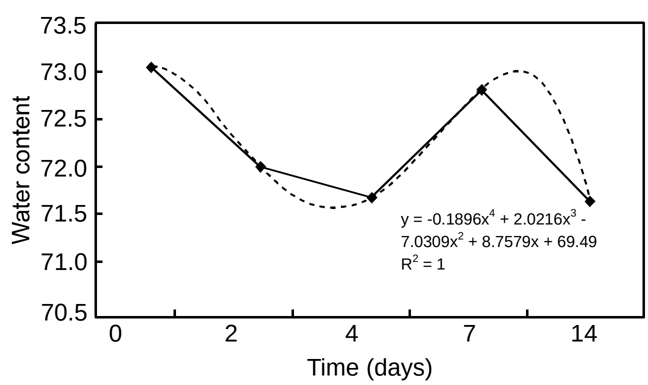
RESULTS AND DISCUSSION

The amount of water in the product vacuum packed in PA/PE film in the refrigeration storage period showed significant variability. A drop in the amount of water was observed in the cheese after 4 days of storage, and then an increase after 7 days. After 14 days of storage, a renewed drop was observed in the amount of water in the tested cheese.

After 4 days of storage, a decrease in the amount of water of 1.34 g was measured, later a rise of 1.15 g and then a renewed decrease of 1.17 g (Figure 1).

The decrease in water content in lactic acid cheese in the first storage period could result from the sorption of water vapour by the packaging.

Further fluctuation of the water phase occurring between 4 and 14 days of cheese storage resulted probably from the metabolism of lactic fermentation bacte-



(◆) water content; (----) polymer (water content).

Figure1. Changes in the liquid phase of vacuum-packed lactic acid cheese under refrigeration.

ria constituting the main microflora of the products. During reactions connected with functioning of cells of these bacteria, there occurred a release of water in the fermentation processes, and its absorption during hydrolysis of lactic acid cheese ingredients.

The change in the amount of water phase in the vacuum packed cheese can be described using the eqn (3):

$$y = -0.1896x^4 + 2.0216 - 7.0309x^3 - 7.0309x^2 + 8.7579x + 69.49 \quad (3)$$

where y- water content (g) and x- time (days)

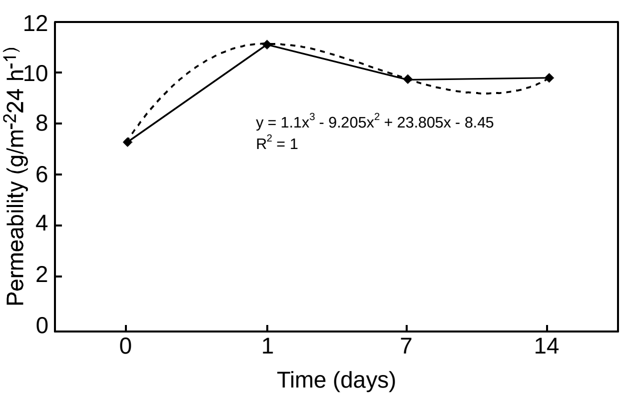
The curve of the fourth order describes the trend showed by the liquid phase of lactic acid cheese while stored in vacuum.

The variability of the water phase in the tested cheese was close to the results obtained by Smietana [29] who observed a similar character in the fluctuation of water phase removed from the packaging.

The packaging (PA/PE film) from the stored cheese showed significant differences in permeability from 1 to 14 days of storage.

A rise in permeability of 3.8 $g.m^{-2}.24h^{-1}$ was observed with regard to water vapour over the 24 h of storage in a refrigerator (Figure 2). The permeability of packaging film, where lactic acid cheese is stored, may be described by the eqn (4):

$$y= 1.1x^3 -9.205x^2 + 23.805x - 8.45 \quad (4)$$



(◆) permeability of water vapour; (----) poly (permeability).

Figure 2. Changes in the permeability of water vapour of PA/PE film (samples originating from lactic acid cheese packaging).

where y- permeability [$g \times m^{-2} \times 24\ h^{-1}$];

x- time (days).

The producer suggests that the studied film should fluctuate between 2-3g $\times\ m^{-2} \times 24\ h^{-1}$ at a temperature of 37 C [30].

Our experience shows that the permeability of water vapour reached the suggested levels despite the low storage temperature. After 7 days of storage in a refrigerator a drop of 1.32 g in the permeability of water vapour was observed, while the cheese was characterized by a fall of 0.019 g in the amount of water in the product relative to the initial amount.

The accumulation of water may be explained only through the condensation of water vapour on the surface of the packaging or penetration through the surface structure of the packaging polymer. The film showed a growth in swelling of 1.22%. For every 1 cm^2 of film, 0.002541 g of water vapour remained over the product. The change in mass of the tested film at the same time, came to an average of 6.0×10^{-5} $g.cm^{-2}.\ 24\ h^{-1}$ of film. This is the result of the fact that 2.36% of the water vapour that should penetrate to the surface of the package can be held back by the polymer. The fall in permeability of the film at this time came to 0.000972 $g.cm^{-2}.24\ h^{-1}$ which means that about 3.19% of the water vapour emitted by the product cannot get out of the package.

Changes in the swelling coefficient suggest that the water is held back by the packaging polymer. That the water is held in by the film is confirmed by the tests conducted by Doroszewicza [31], showing the sorption of water through polymer in the first 40 h of holding the polymer in contact with a stream of water vapour.

The fall in the concentration of the water phase of the product for up to 4 days can be explained by the process of the swelling of the material caused by trapping the water vapour molecules which are passing through packaging material. As a result, the saturation of the polymer causes the halting of the absorption of water and a drop in the permeability of water vapour through the packaging. The polymer's solubility coefficient is actually different for each type of foil and it is correlated with the temperature. Significant water absorption is characteristic of the polyamide PA which is used as an ingredient in PA/PE foil. This fact is probably quite influential in the processes occurring between the surface of the cheese and the tested packaging.

From 7 to 14 days of storage, the cheese does not undergo any significant changes (0.07 g) in the permeability of water vapour through the polymer. During this time, the cheese showed a drop in the amount of water (Figure 1). The water remained condensed on the inside surface of the packaging and also passed through the outside surface of the packaging. The results obtained assumption about the amount of water vapour sorption through the polymer, causing a fall in the concentration and the further absorption of water by the polymer as well as its passing through the outside. These factors also add to the increased condensation of vapour on the inside surface of the packaging and falling onto the surface of the product (Figure 2).

After 14 days of refrigeration storage of the cheese in PE/PE packaging film further growth was observed in the swelling of the packaging material, clearly differentiating the mass at the level of 0.000068 $g.cm^{-2}$.

However, in addition to water vapour on the inside of the cheese's hermetic packaging we also found organic acids dissolved in water. Over the surface of the cheese one can also verify the existence of vapour from lactic acid solution, acetic acid and formic acid which makes it significantly more difficult to interpret the results simply on the basis of the retention of water vapour.

From the results presented by Peiper [1] we see that polyethylene shows good barrier properties against the penetration of acetic acid. Peiper's research also shows that if the product comes into contact with LDPE, then the surface of the polymer absorbs mainly non-polar elements. However, it is obvious that this does not have to be true for PA/PE foil.

The collection of water inside the packaging film, connected with the drop in the foil's permeability, is a significant problem. The gathering of water and water solutions allows for intense development of micro flora, the growth of which was stopped in the first stage of storage as the result of the interaction with milk fermentation bacteria and by the initial lowering of the water s activity in the product.

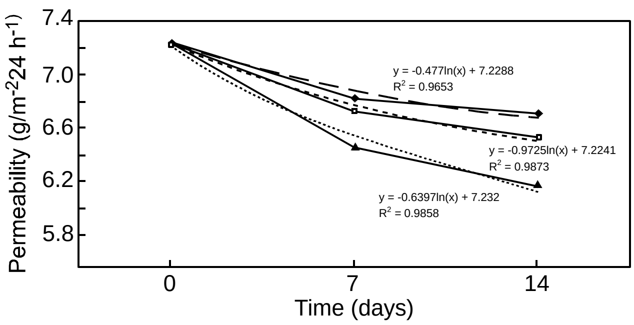
Our earlier research [21-23] confirmed the intense growth of microflora after 7 days of storing the lactic acid cheese vacuum packed in PA/PE film in a chilled environment.

Hilldingsson [32] showed that the permeability of LPDE increases together with the concentration of acetic acid and this process is dependent on the temperature. So whatever rise which occurs in the concentration of lactic acid as a result of the microflora metabolism in the cheese during the initial storage phase that we may observe, the concentration of acetic acid and the low storage temperature of the cheese may influence the process of permeation and the holding of acid solutions on the inside of the packaging and over the inside surface of the packaging film. Perhaps the slow production of acetic acid in the PE phase is a factor influencing the retention of water and other solutions of bacterial metabolites between the surface of the cheese and the surface of the packaging film.

The adhesion of acetic acid on the surface of the polymer in a model condition reaches 370 N.m relative to LDPE is shown by Hilldingsson [32].

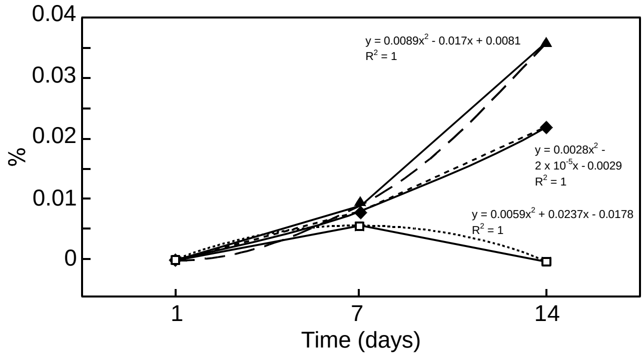
In a model condition, the changes in permeability of PA/PE film in terms of water vapour in the presence of lactic acid of variable acidity is more distinct than in reality. In foils tested in a model environment a fall in the permeability of water vapour is observed regardless of the acidity of the solution used (Figures 3-5). The growth in the permeability levels off at 0.41 to 0.77 $g^{-2}.24\ h^{-1}$ by the first week of storage. The largest drop in permeability for the model solution with a pH value of 5.2 after 7 days reaches -0.77 $g.m^{-2}.24\ h^{-1}$ and 1.05 $g.m^{-2}.24\ h^{-1}$ after 14 days (Figure 5).

The drop in the permeability of the packaging film after 14 days using a model solution with a pH value of 5.2 testifies to the fact that the processes of the actualization of the product during the extended storage



(◆) pH 4.3; (□) pH 4.7; (▲) pH 5.2; (— —) log (pH 4.3); (- - -) log (pH 4.7); (........) log (pH 5.2).

Figure 3. Changes in the permeability of water vapour for PA/PE film (samples from lactic acid solution pH 4.3, 4.7 and 5.2).



(◆)pH 4.3; (□) pH 4.7; (▲) pH 5.2; (▬ ▬) poly (5.2); (- - -) poly (4.3); (........) poly (4.7).

Figure 4. Changes in the swelling coefficient for PA/PE film stored under model conditions (lactic acid solution pH 4.3, 4.7 and 5.2).

process lead to the collection of water between the product and the packaging.

We can conclude, that in a solution with such pH the intense processes lead to building of water molecules into the packaging polymer.

Changes in the PA/PE film s permeability under the influence of low temperatures and lactic acid with a pH of 4.3-5.2 can be described using the following equation:

$$y = ax^2 - bx + c \qquad (5)$$

where y- permeability (g.m^{-2}.24 h^{-1}), and x- time (days)

The permeability of the films tested in a model condition can even be twice as high as the values presented by other authors [30].

Testing the level of swelling of the film in lactic acid solution showed that the PA/PE film in storage for 7 to 14 days shows a growth in the coefficient α. Changes in the swelling coefficient α in lactic acid solution can be described with the help of the following polynomial:

$$y = ax^2 - bx + c \qquad (6)$$

where y-swelling coefficient, and x- time (days).

A film which was removed immediately from the cheese packaging also showed a dynamic growth in the coefficient during the initial storage period. The curve describing the tendencies of a change in the level of swelling in PA/PE film coming off the cheese takes the form of:

$$y = -a \ln(x) - b \qquad (7)$$

where y-swelling coefficient, and x- time (days).

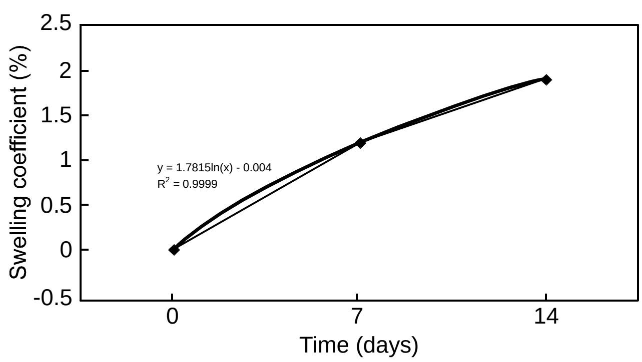
The swelling coefficient for the model environment and the real environment differed significantly up to 7 days of storage.

This shows that during the storage of the cheese it is possible that some of the water vapour will be absorbed by the packaging material. The results obtained suggest that the stopping of water with dissolved metabolites is more intense in case of about 7 days of storage.

The film coming from the model environment shows a tendency to change the level of swelling described using the different formula that was used to describe lactic acid solution.

The observed differences in the form of formula can testify to the significance of the metabolites coming from the product and produced by microorganisms as the result of contact between the film and the surface of the food product.

Changes in the permeability of the film, however, in relation to the water vapour produced in the model condition and with packaging taken off from the food product testifies to the type of metabolites dissolved and the volatile with water vapour determines the level of sorption and the permeability of the PA/PE film. The results obtained from the model condition tests also suggest that lactic acid is not the most important metabolite which decides as to the holding of water inside the packaging and limiting its passage into the



(◆) swelling coefficient; (▬) log swelling coefficient.

Figure 5. Changes in the swelling coefficient for PA/PE film originating bags filled with lactic acid cheese.

outside environment.

The literature recognizes interactions between food products or their ingredients and the polymers as packaging films.

Briston et al. [33] have divided these interactions into 3 groups of phenomena connected with direct contact between the polymers and the food products. This classification distinguishes, among others, migration of ingredients of the polymer to the food products as a result of the penetration of the food product into the polymer.

Migration was not, however, the subject of the current research, but the swelling and permeability of the polyamide-polyethylene laminate for steam signified the possibility of interactions specified by Briston et al.

The interphasal occurrence of penetration of the polymer by liquid food particles is also described by Feingenbaum [2].

According to Johansson [34] the type of interaction between the air-borne food particles and the polymer is a 'sorptive' type of interaction that involves the absorption of aromas.

Whatever interactions between the food-products and the packaging described by Johansson [34] there are a different type of change, still of interaction type and it is most like those observed in our experiment.

Among the microflora in the lactic acid cheese the dominating microorganisms are lactic acid bacteria and enterococci. From publications presented by Paleari et al. [35] we are able to conclude that the mutual influence of these bacteria on each other stimulates the emission of larger amounts of concentrations of air-borne capric, caprylic and butyric acids and gasses with steam. The increased concentration of these acids and of lactic acid during the storage of the lactic acid cheese causes changes to develop in the pemeability of the tested polymer for gasses and steam. The growth of the factor of pemeability in proportion to the growth of the concentration was observed for acetic acid by Hildingsson et al. [31]. The authors also observed the influence of another acid such as oleic acid with the change of the coefficient. Olafsson [36] has shown, however, that acids of lower polarity (lactic acid and citric acid) do not have a significant influence. This would indicate a weak penetrability of these acids through polyethylene.

In the present research, however, multi-directional interactions were observed between the biogenic factors (the metabolites of bacteria and the water phase of the packaged product) and the PA/PE laminate used.

The causes behind the observed changes in the properties of the tested laminates were:

- The reactions between the functioning groups of acids and polymers after the penetration of water solutions of these acids into the interior side of the polymer.
- Insignificant changes in the crystalline structure.
- Holding the steam and air-borne acids with steam inside the polymer.
- The penetration of the small amounts of steam and acid solutions to the outside of the packaging.

CONCLUSION

1. The permeability of PA/PE film for lactic acid cheese packaging changes under the influence of the metabolites produced by the microflora present on the surface of the cheese. Changes in permeability of film regarding water vapour between the lactic acid cheese surface and the packaging vary from the changes caused by lactic acid solution in model condition.
2. The study of changes occurring in film properties in model conditions shows a significant influence of even small fluctuations of environmental acidity on the permeability of tested packaging
3. Changes of permeability of PA/PE film towards water vapour are the result of release of lactic acid from the cheese during its contact with the packaging, and the sorption of its solutions by the film.
4. The magnitude of sorption and desorption of water, lactic acid and other metabolites during storage of lactic acid cheese indicates that these processes are based on the phenomena of saturation of polymer network with solutions and the clusters formation.

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