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A study on the migration of organic pollutants from recycled paperboard packaging materials to solid food matrices

V.I. Triantafyllou [a], K. Akrida-Demertzi [b], P.G. Demertzis [b,*]

[a] Department of Molecular Biology and Genetics, Democritus University of Thrace, GR-68100 Alexandroupolis, Greece
[b] Department of Chemistry, University of Ioannina, Panepistimioupolis-Dourouti, GR-45110 Ioannina, Greece

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Abstract

Paper and board are widely used as food packaging materials, mainly for disposable products. As public interest in conservation of natural resources has accelerated in the past several years, the use of recycled paper and board has increased. Recycled fiber materials can be used in certain limits as food contact materials. The safety of recycled fiber-based materials for food contact applications is largely dictated by the ability of post-consumer contaminants to be absorbed into recycled materials and later released by the packaging material and trapped on the food. The present work was undertaken with the aim of investigating the physicochemical behavior of selected model contaminants on paper and board, in contact with foodstuffs thus producing a fundamental set of data about their mobility from recycled paper and board into foods. More specifically, the kinetics of migration of selected model contaminants (surrogates) from contaminated recycled paper packaging samples into dry foodstuffs with different fat content was studied using a method based on solvent extraction and GC-FID quantification. Results showed the ability of selected contaminants of various types and various volatilities to potentially transfer to dry foods. The proportion of substances migrated to food was strongly dependent on the nature of the paper samples, fat content of the food, chemical nature and volatility of the migrant. The highest level of migration of organic pollutants was observed for the substrate with the highest fat content. Furthermore, it is shown that contact time and temperature have a significant effect on migration of model contaminants into foods.

Keywords: Recycled paper; Food packaging; Migration; Solid food matrices; Model contaminants; Food contamination

1. Introduction

Paperboard packages represent a large and constantly growing part of the food packaging industry, due to their advantages compared with other traditional packaging materials. It represents the largest sector of the food and drink packaging market, being used in the 48% of all packaging (Song, Park, & Komolprasert, 2000). Continuous improvements, concerning both material and package design, have led to better products and increase usage of paperboard packages. In 1999, the Confederation of European Paper Industries (CEPI) member countries produced 85.2 million tons of paper and board. In volume terms, the EU's paper production can be qualified thus. (1) Graphic paper grades: 50%, (2) packaging paper: 40%, (3) hygiene and specialty papers: 10%. This means that half of the European paper and board production is directly or indirectly concerned by food-contact standards (Escabasse & Ottenio, 2002).

To protect the environment, new ways of recycling waste, including packaging materials (paper and plastics), are appearing on the market. The environmental pressure and the general tendency of recycling pose recycled plastics and paperboard in the situation of being used as food contact materials (Triantafyllou, Karamani, Akrida-Demertzi, & Demertzis, 2002). Paper and board partly or fully produced from recycled fibers is already being used in contact with certain foodstuffs in many countries in Europe. Recycled paper is mainly used in direct contact with

* Corresponding author. Tel.: +30 265 10 98 340; fax: +30 26510 98795.
E-mail address: pdemertz@cc.uoi.gr (P.G. Demertzis).

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dry foodstuffs like flour, grain, sugar, salt, rice and pasta (Binderup et al., 2002). Food-contact materials, including recycled fiber-based paper, have to comply with a basic set of criteria concerning safety. This means that recycled paper for food contact should not give rise to migration of components, which can endanger human health.

Recovered paper and board (P & B) may vary in origin and could include paper containing printing inks, adhesives, trace elements, waxes, fluorescent whitening agents and dyes, sizing agents, organochlorine substances, plasticizers, aromatic hydrocarbons, volatile organic compounds, curing and grease-proofing agents, amines, biocides and surfactants (Binderup et al., 2002; Castle, Offen, Baxter, & Gilbert, 1997; Grob, Biedermann, Artho, & Egli, 1991; Sipiläinen-Malm, Latva-Kala, Tikkanen, Suihko, & Skyttä, 1997; Tice & Offen, 1994; Vinggaard, Korner, Lund, Bolz, & Petersen, 2000; Ziegleder, 2001). Recently, Damant and Castle (1999) wrote an extensive review on potential contaminants in recycled paper and board food contact materials. This work makes evident that there are many potential contaminants in recovered paper. Therefore, chemicals from these sources may migrate into foodstuffs and potentially cause a risk to human health. There is thus a strong need to better understand migration phenomena and develop appropriate methodologies to test migration from paper and board food contact materials. In the last years, more and more publications about migration of contaminants from paperboard packaging materials into food and food simulants have been published to evaluate the suitability of recycled paperboard for direct or indirect food contact applications (Anderson & Castle, 2003; Aurela, Kulmala, & Söderhjelm, 1999; Boccacci-Mariani, Chiacchierini, & Gesumundo, 1999; Salafranca & Franz, 2000; Sarria-Vidal, Montana-Miguélez, & Simal-Gándara, 1997; Song, Begley, Paquette, & Komolprasert, 2003; Summerfield & Cooper, 2001; Triantafyllou, Akrida-Demertzi, & Demertzis, 2002).

As part of a EU research project FAIR-CT98-4318, with the acronym 'recyclability', an analytical method to study the migration of selected model compounds from paper and board onto a suitable food simulant has been developed. The method involved fortification of paper strips with a mixture of selected model contaminants, measurement of their uptake by the paper samples and determination of the extent of migration of these compounds into a food simulant using GC analysis. Modified polyphenylene oxide (MPPO) called tenax has been studied as solid simulant for specific migration of contaminants released from recycled paperboard samples (Franz, 2002; Triantafyllou et al., 2002). In the present work, a considerable amount of data concerning the mobility of different organic pollutants from recycled paper packaging onto dry foodstuffs has been gathered using the developed method. More specifically, the kinetics of migration of selected model contaminants (surrogates) from contaminated recycled paper packaging into several solid matrices was studied. The main goal was to evaluate the ability of contaminants to migrate to real dry foodstuffs, in an effort to closely examine the interactions between paperboard and packaged goods. The possibility of using MPPO as a food simulant for different types of dry foodstuffs was also evaluated. Obtained results were discussed in terms of the possibility, from a safety point of view, to use recycled fibers for food contact applications.

2. Materials and methods

2.1. Reagents, solutions and food samples

A set of 10 model substances or surrogates was selected which can be considered as representative with regard to chemical structure, polarity, molecular weight, volatility and functionality in the paper making process. The selected chemicals are the following: (1) *o*-xylene, (2) acetophenone, (3) *n*-dodecane, (4) naphthalene, (5) diphenyl ether, (6) 2,3,4-trichloroanisole, (7) benzophenone, (8) diisopropylnaphthalenes, isomeric mixture (DIPN), (9) dibutyl phthalate, (10) methyl stearate. All reagents were of analytical grade and were obtained from Sigma, Aldrich and Fluka. Standard solutions of these reagents in ethanol (Merck) at the appropriate concentrations (0.2–20 ppm) were analyzed for external calibration.

As dry foods for migration studies semolina (fat content 1.9%), instant baby cream (fat content 13.5%) and infant whole milk powder (fat content 27.7%) purchased from a local supermarket were used, representing foods with low, intermediate and high fat content, respectively.

2.2. Paper samples

For migration studies two paper samples were used, having different pulp percentage of recycled matter, grammage and thickness. Their properties are listed in Table 1.

2.3. Fortification of paper samples with the surrogates

Strips of each of the paper samples with approximate dimensions 6×1 cm were placed in 22 ml septum glass vials. The vials were filled with 20 ml of contamination solution (solvent: absolute ethanol HPLC grade) having surrogate concentration of approximately 250 mg l^{-1}. The vials were sealed and left for approximately 2 h in a horizontal position (paper strips were totally immersed into the contamination liquid) at room temperature. The contaminated paper strips were then removed and placed on a steel lattice in a ventilated hood at room temperature. After approximately 15 min drying, the samples were ready for being used for the migration test.

2.4. Determination of contaminant concentration in paper samples

The determination of the initial concentration of the sorbed surrogates was performed as follows: contaminated

Table 1
Properties of the paper samples used in the present study

Sample	Type	Recycled (%)	Grammage (g m^{-2})	Density (kg m^{-3})	Thickness (μm)
P1	Fluting	30	107	511	209
P2	Kitchen towel	100	46.7	248	188

paper samples were cut in small pieces (1 × 1 cm) and were placed in 5 ml vials with 4 ml of ethanol. Extraction was performed by gentle shaking of the vials for 1 h at room temperature. The ethanol extracts were directly analyzed by GC.

2.5. GC analysis

The GC unit was a Fisons 9000 series gas chromatograph equipped with an auto injector and a FID detector. The separation column was a 30 m × 0.32 mm internal diameter fused silica capillary DB-1 (non-polar, 100% cross-linked dimethyl polysiloxane) with a film thickness of 0.25 μm. The following GC parameters were kept constant: detector temperature, 290 °C; injector temperature, 240 °C; injection mode: split with split ratio ca. 15 ml/min; injection volume, 1 μl. Column temperature program: 60 °C (3 min), from 60 °C at a rate of 10 °C/min to 270 °C (3 min). Carrier gas: He, at a flow rate of 1.45 ml/min.

2.6. Kinetics of migration in contact with food powder

Kinetic migration studies were carried out with strips of both paper samples fortified with the mixture of surrogates as described previously. Each contaminated strip was placed on the interior wall of a 22 ml septum glass vial lying in horizontal position. Strips were evenly covered with 0.75 g of each of the three food powders and the vials were sealed. The exact experimental temperature/time exposure conditions were as follows:

- 70 °C for 20, 40, 90, 120, 240 and 360 min
- 100 °C for 10, 20, 30, 40, 60 and 120 min.

Triplicate determinations were carried out. The determination of the amount of the desorbed surrogates in the dry food substrate under all temperature/time conditions was performed as follows: after removal of the respective paper strip the migrated surrogates were extracted from food powder using 3 ml of ethanol as extraction solvent. The extraction was carried out at room temperature for approximately 5 min under gentle agitation of the sample. The extraction was repeated twice (3 extractions in all). The combined extracts were concentrated under nitrogen stream and were GC analyzed.

The relative precision RSD of the method was found to be ±2–12% for all substances. The detection limits for all contaminants were in the range 0.2–0.5 ppm. Finally, the calculated recovery values were all in the range 87–104%.

3. Results and discussion

3.1. Surrogate determination in the contaminated paper samples

Migration experiments were performed using a contamination solution with relatively high soaking concentration (250 ppm) in order to fortify the 6 cm^2 paper strips at a workable concentration for the migration experiments. Absolute ethanol was found to cause complete extraction of sorbed surrogates from both paper and food powder samples, under the applied experimental conditions. Obtained results show that, under the same sorption conditions (concentration of contamination solution and temperature), large differences in the sorbed amounts of contaminants for both paper types have been observed. P2 paper samples exhibit a greater sorption capacity than P1 samples, due to the high absorbent nature of this paper type (kitchen towel). Details on this matter have been given elsewhere (Triantafyllou et al., 2002).

3.2. Migration studies

Migration from paper and board has not been as extensively studied as migration from plastic materials. However, it has been demonstrated that migration from paper and board occurs even to dry food. Migration to dry foods was reported at least for phthalates, diisopropylnaphthalenes (DIPNs) and benzophenone (Aurela et al., 1999; Boccacci-Mariani et al., 1999; Johns, Jickells, Read, & Castle, 2000). Especially benzophenone migrates readily to foods even during frozen storage (−20 °C). As migration from paper and board has been studied much less than migration from plastics, the modeling of migration from fiber materials is only just starting.

To evaluate if the recycled paper and board could be used as food packaging and/or food-contact material, migration studies of selected surrogates from contaminated paper and board into foods have been performed at two different temperatures in a wide range of time. The test conditions represent short-term exposures at high temperatures (rapid testing). It is important to know the transference capacity of these pollutants, which is the process called migration. A large number of experimental data have been obtained and used to evaluate the effects of high temperatures to the actual mass transfer during contact with foods. Migration was expressed as percentage of the initial surrogate concentration in the paper.

The results of the time dependent migration into semolina for both paper samples at 70 and 100 °C are given in Figs. 1 and 2. Of the 10 substances employed for migration

testing into semolina some migration cases did not performed well enough for particular substances to monitor migration. This was the case of *o*-xylene (highest volatility of the group) and dodecane which were not detected during migration studies from both paper samples to semolina. This can be attributed to the very low initial concentration levels of these surrogates into the contaminated paper strips and to losses by volatilization from the heated food into the air space of the vial.

The general trend of uptake into semolina show higher values of percentage of migrated compounds over time. Equilibrium was achieved at 100 °C in ca. 1 h. At 70 °C, 4 h were found to be enough to reach equilibrium. A plateau is then reached corresponding to a saturation of the food phase (semolina). It can also be seen that there is a difference in efficiency of transfer of the substances monitored. A more efficient rate of transfer was observed for the less volatile substances. It should be noticed that volatile surrogates such as acetophenone and naphthalene might have escaped from the paper/food-contacting phase system due to thermal desorption, so negligible migration was observed. In contrast, volatilization was lower for DBP, methyl stearate and DIPNs even at 100 °C due to the high boiling points of these compounds. DIPNs and methyl stearate demonstrated most efficient transfer, calculating to ca. 40–80% at equilibrium.

Concerning the migration behavior of the two different paper samples into semolina, it is shown that the migration from P2 samples was generally higher than that from P1 samples. Migration from P2 samples increased when

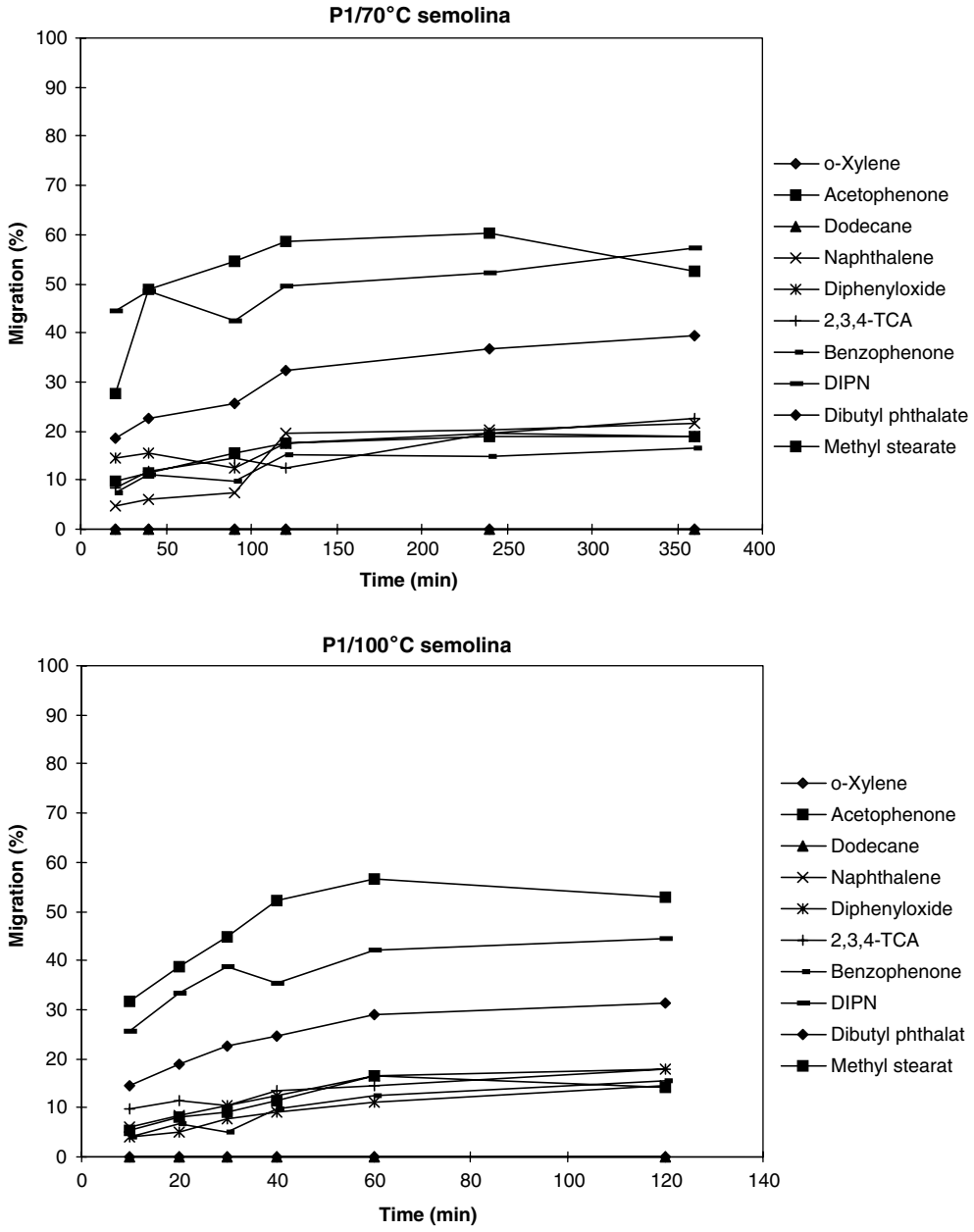

Fig. 1. Percentage of migrated surrogates from P1 paper sample into semolina at 70 and 100 °C, respectively.

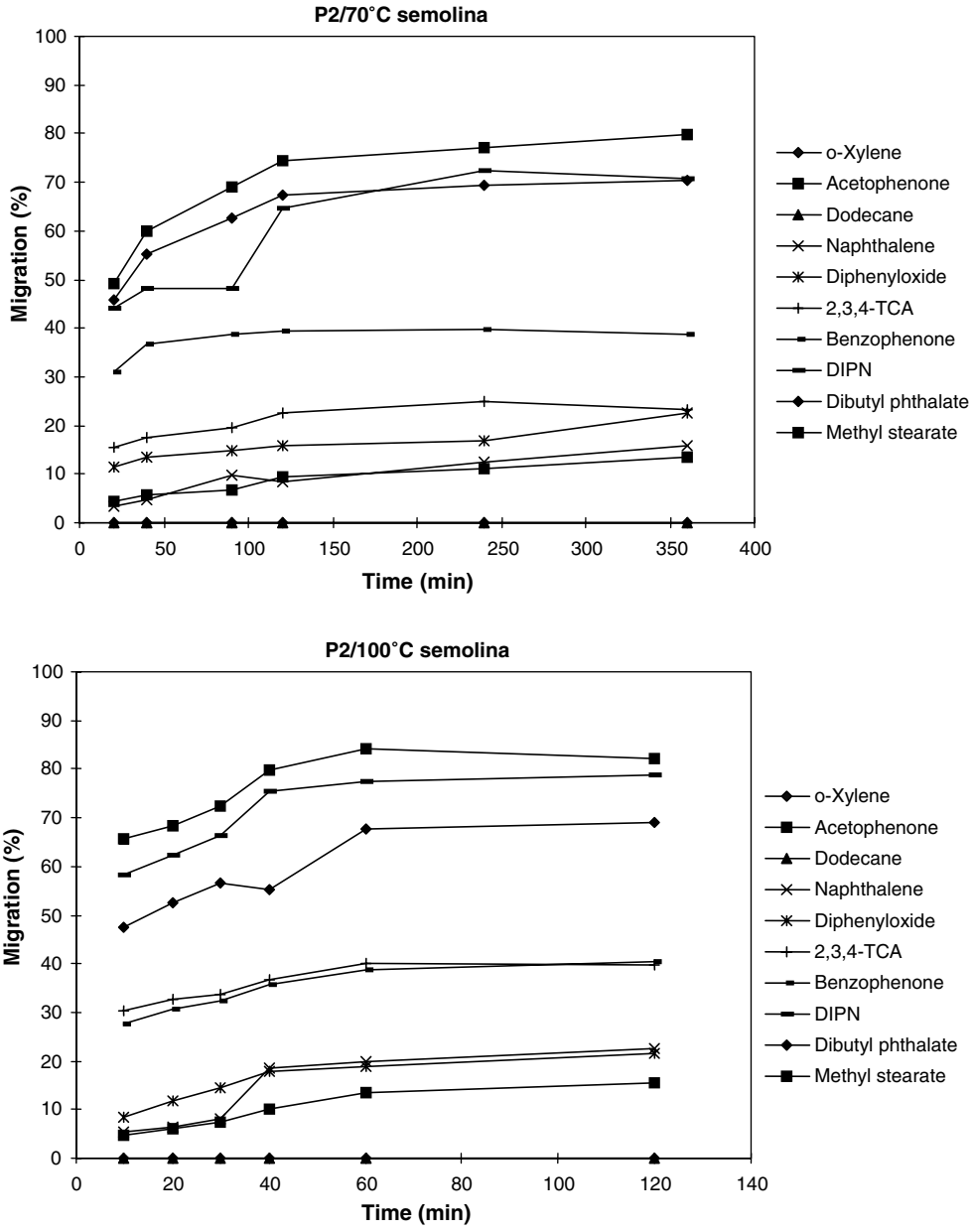

Fig. 2. Percentage of migrated surrogates from P2 paper sample into semolina at 70 and 100 °C, respectively.

temperature was increased. On the contrary, migration from P1 samples generally decreased with increasing temperature. At the higher temperature studied, release and migration of surrogates from paper sample P2 (the more absorbent sample) into semolina seems to be the predominant process and loss by volatilization is comparatively low. On the other hand, P1 paper sample has high grammage and retains the adsorbed surrogates more strongly, thus reducing the migration tendency of them. Moreover, the loss by volatilization, especially at elevated temperatures, seems to be larger than any additional migration due to temperature increase and consequently increasing the temperature from 70 to 100 °C generally resulted in a decrease of surrogate concentrations in the dry food.

Obtained migration values from both paper samples for semolina are comparable with those obtained with tenax (Triantafyllou et al., 2002).

The results of the time dependent migration of surrogates from both paper samples at both temperatures into instant baby cream (fat content: 13.5%) are presented in Figs. 3 and 4. The corresponding results for infant whole milk powder (fat content: 27.7%) are shown in Figs. 5 and 6.

It is observed that short exposure times were enough to achieve equilibrium of migrants between paper and fatty dry foods, since papers are rather open and porous structures. Concerning the migration behavior of both paper samples, the concentrations of migrants into fatty dry

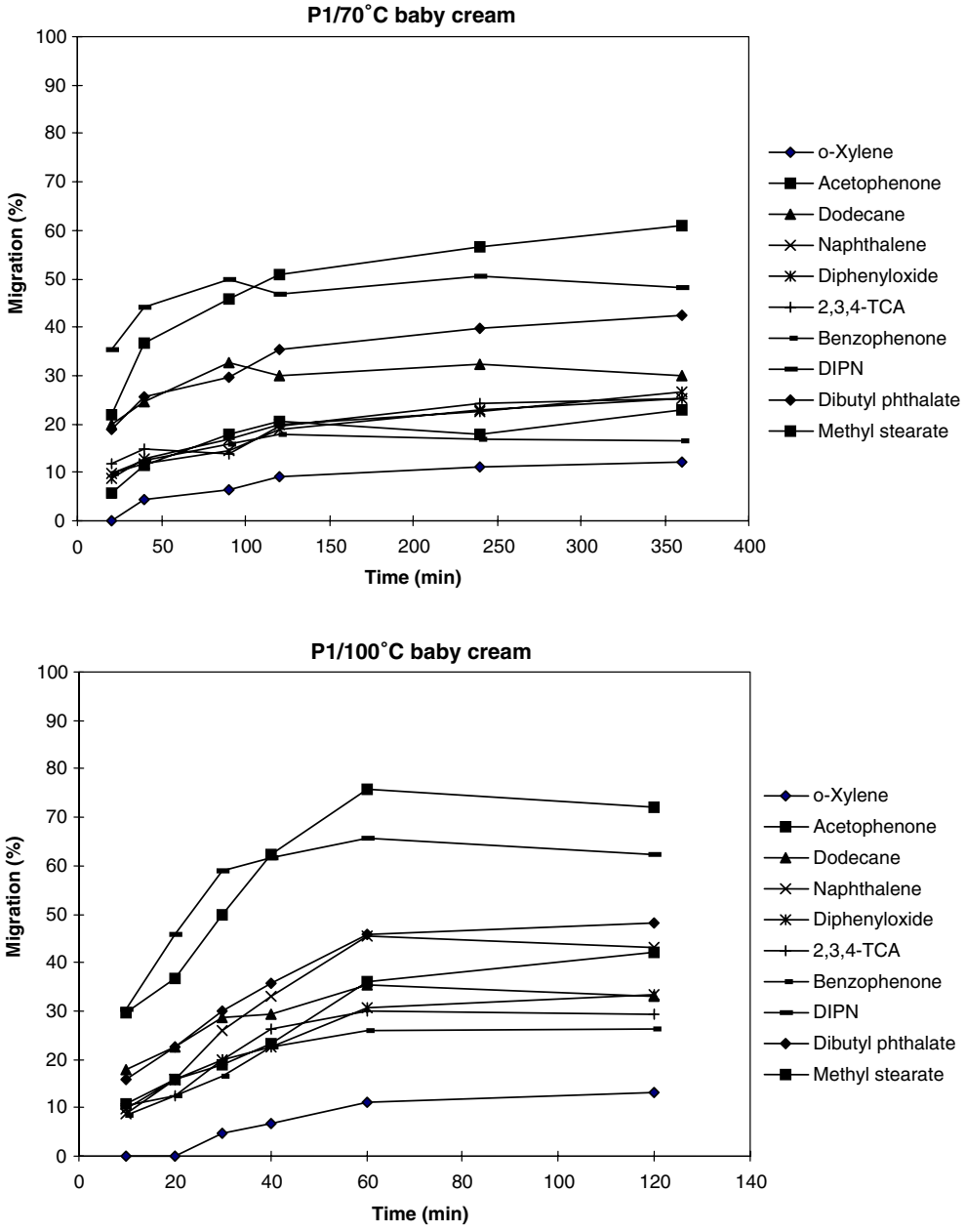

Fig. 3. Percentage of migrated surrogates from P1 paper sample into baby cream at 70 and 100 °C, respectively.

foods were generally increased as time and temperature increased. The high fat content of the foods contributed to a more pronounced migration tendency. Dry foods have no free fat or oil phase on the surface but at high temperature the fat matter eventually melts and can penetrate into the paper as well. Once on the bulk of the paper such liquids can extract the eventual contaminants bringing them to the surface for diffusion. From the surface these substances can then migrate into the food.

Comparing the results of the present study with those obtained in our previous study using tenax as food simulant (Triantafyllou et al., 2002) the following conclusions are extrapolated: as the selected organic surrogates have generally a higher solubility in fat containing media the percentage migration values into the food with the highest fat content (infant milk powder, fat content 27.7%), are higher than those into tenax. Benzophenone, for example, which is widely used as a photo-initiator for inks and varnishes that are cured with ultraviolet (UV) light, is a highly fat-soluble compound and it is not surprising that whole milk powders in direct contact with paper samples pick up benzophenone at higher level compared to tenax (Anderson & Castle, 2003). In case of the substrate with intermediate fat content (instant baby cream, fat content 13.5%) obtained migration values were comparable with those obtained with tenax at 70 °C but generally higher at 100 °C. This can be attributed to losses of compounds from tenax through a thermal desorption process which

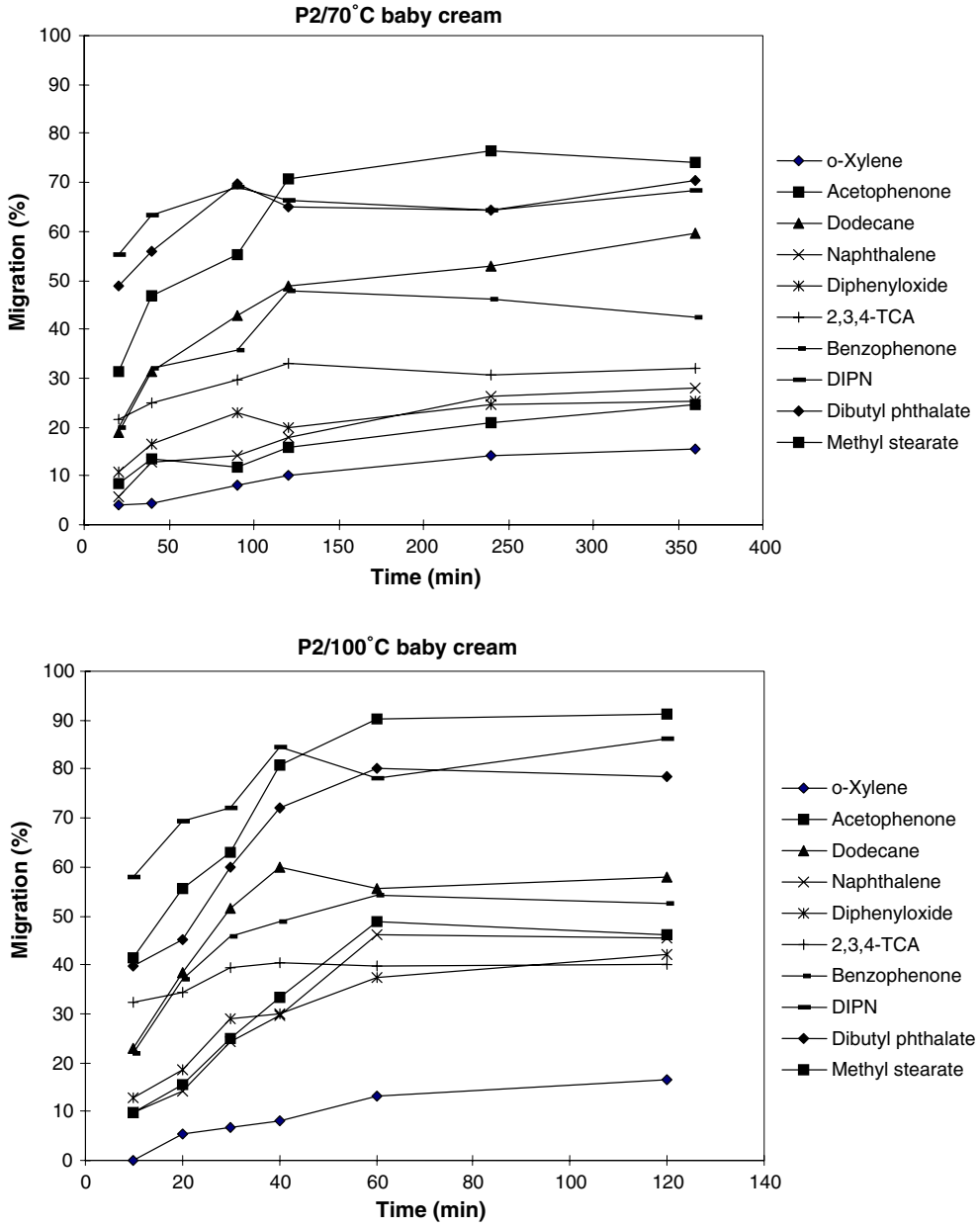

Fig. 4. Percentage of migrated surrogates from P2 paper sample into baby cream at 70 and 100 °C, respectively.

takes place at high temperatures (Nerin, Rubio, Cacho, & Salafranca, 1998; Salafranca, Cacho, & Nerin, 2000).

Migration values for DIPN, DBP and methyl stearate into both food powders with intermediate and high fat content were also found to be very high, eventually higher than expected according to their molecular weights. Lipids encourage the absorption of these apolar, non-volatile compounds. Sturaro, Parvoli, Rella, Bardati, and Doretti (1994) reported the migration of DIPNs from recycled paper and board into rice. They proposed that lipids present in the rice encouraged the absorption of the DIPN isomers. Taking into account their chemical structure, probably the activation energy to break the steric hindrance has been surpassed at high temperatures, and consequently, the migration tendency is higher than expected for these compounds. The loss by volatilization from the heated food was also negligible for these contaminants due to the high boiling points of them. On the other hand, the most volatile compounds (e.g., acetophenone, *o*-xylene, naphthalene) exhibit lower migration percentage values. In this case, it can be assumed that small amounts of surrogates were lost in the vapor phase. It is important to mention that not all the compounds released from the paper are trapped by the simulants or by the food, which in terms of real migration conditions is an advantage.

Concerning the migration behavior of the two different paper samples, it was found that the migration from P2 samples into both fat-containing food powders was higher than that from P1 samples. This is in agreement to the results obtained with semolina and can be partially

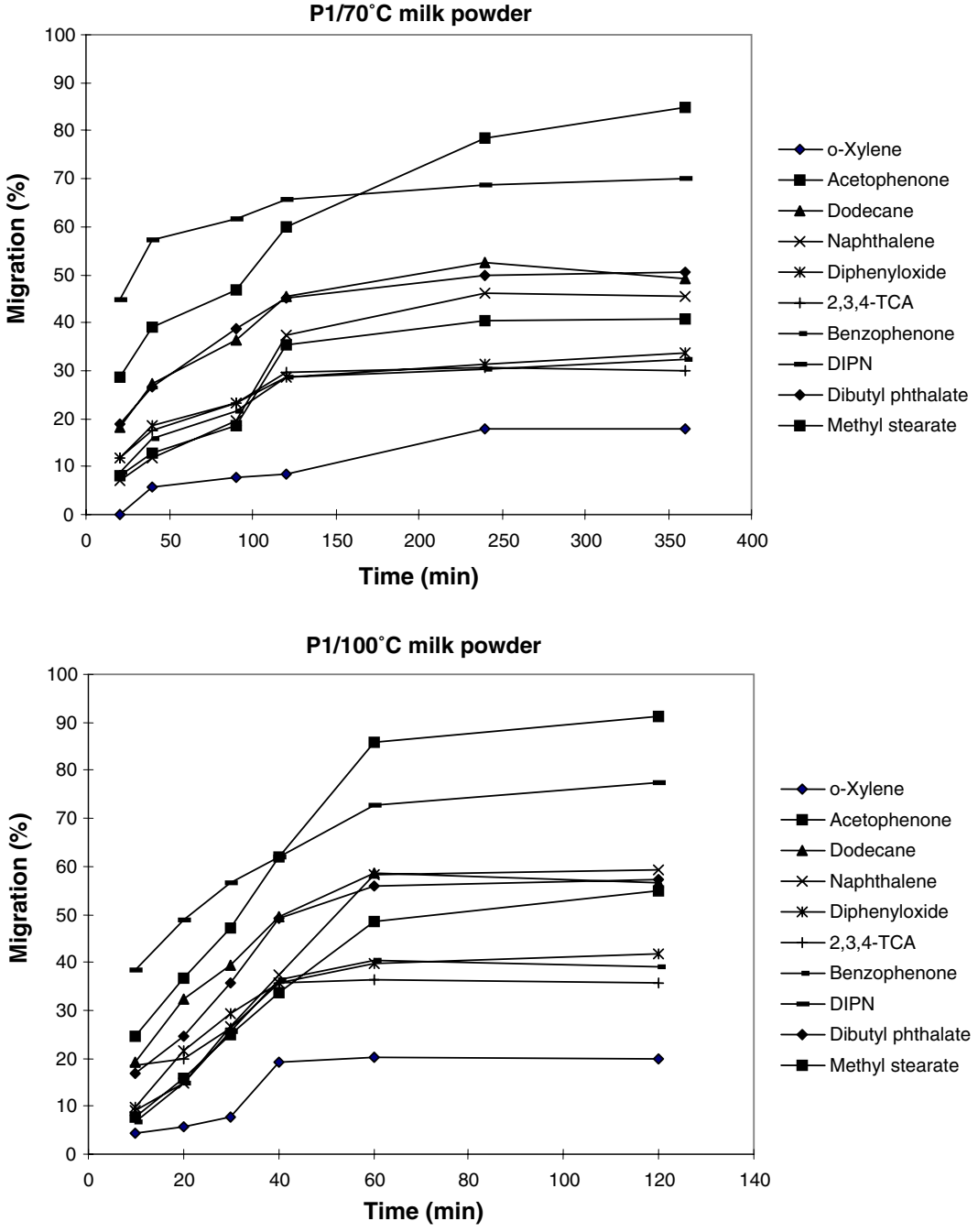

Fig. 5. Percentage of migrated surrogates from P1 paper sample into milk powder at 70 and 100 °C, respectively.

attributed to the higher absorbent nature of P2 paper samples (higher initial surrogate concentrations) in comparison with P1 samples. Moreover, P2 sample has lower thickness, density and grammage than P1 and this could facilitate the release of adsorbed compounds into food and/or food simulants. In other words, the migration of the surrogates to the food phase is higher in the case of thinner samples. Aside from grammage and thickness, the paper composition, in combination with the chemical structure of the surrogates play a role in the extent of migration.

To summarize, the results of the migration tests carried out revealed that migration would occur when there is direct contact between recycled paper and a dry food powder. Migration is in general rapid and extensive and to keep migration in acceptable limits a low storage temperature should be applied in combination with a suitable barrier layer for indirect contact.

4. Conclusions

In this work, migration of selected model contaminants from paperboard into dry foods was studied. Two paper samples of different pulp percentage of recycled matter, grammage and thickness were compared and the suitabil-

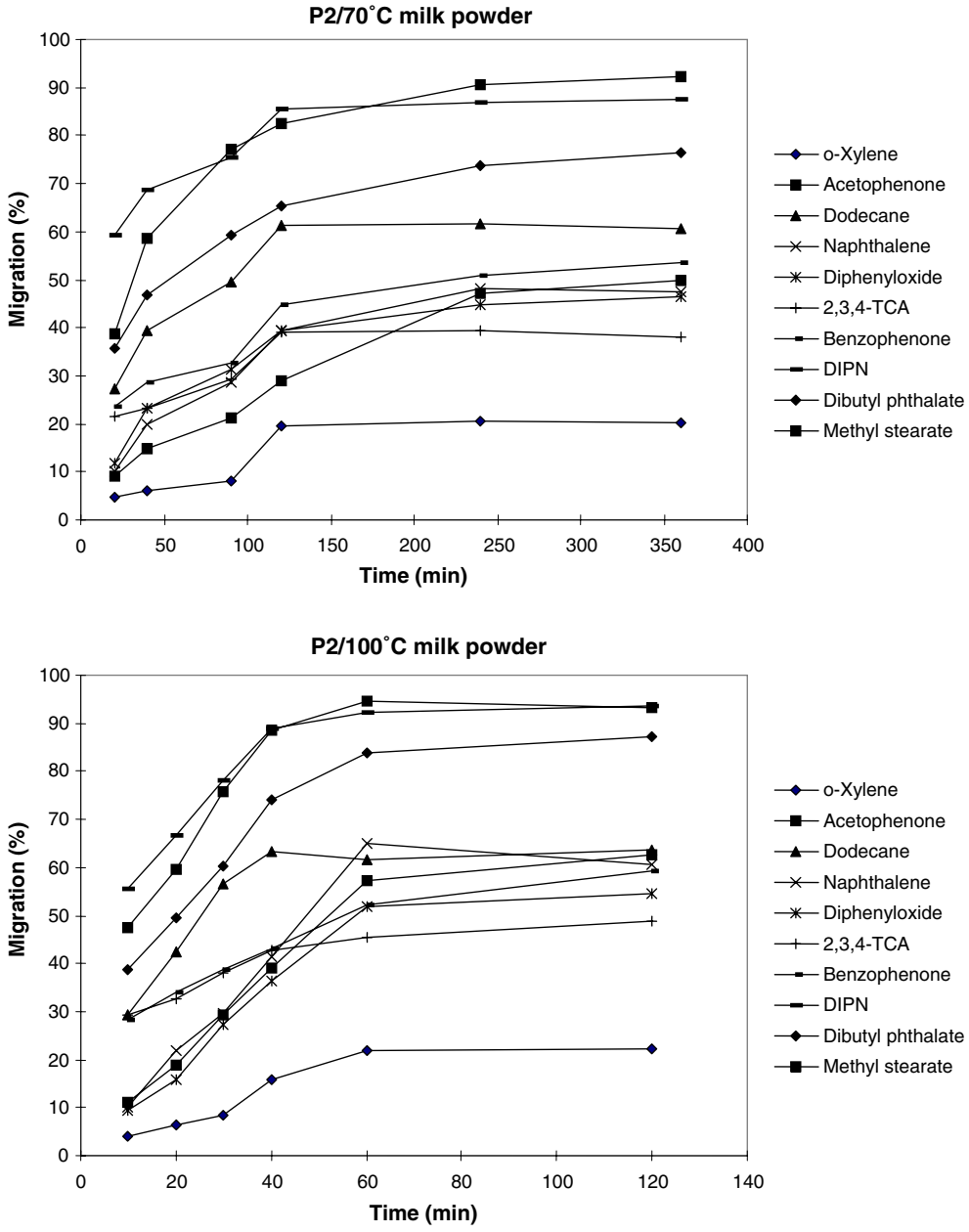

Fig. 6. Percentage of migrated surrogates from P2 paper sample into milk powder at 70 and 100 °C, respectively.

ity, from a safety point of view, of recycled fibers for food-contact applications was evaluated. One major issue was to focus on the influence of temperature on the time dependent migration course. Useful conclusions were extrapolated about the migration behavior of several surrogates, which are representative in terms of chemical structures and physical properties. The migration kinetics was found to be dependent on the temperature, the nature (grammage, thickness and pulp composition) of the paper sample and the nature (molecular size, chemical structure and volatility) of the surrogate. Migration tests have demonstrated how rapid migration occurs at elevated temperatures. There was efficient and rapid transfer, with equilibration (maximum migration level) reached after a short period of time.

The results were compared against test results that have been carried out similarly with tenax as potential food simulant. Tenax has been found to be a suitable food simulant for dry foods with low and intermediate fat content, such as semolina and instant baby cream. On the other hand, dry food with high fat content, such as infant whole milk powder, exhibit higher migration tendency. It should be noted that the migration results of the present study represent a "worst case scenario" as the tests were performed in sealed containers at elevated temperatures. However, such studies can be applied as rapid screening methods for anticipating the potential of certain contaminants from paper and board food contact materials to migrate into foods under regular conditions of packaging, distribution and storage.

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