Rapid comparison of mineral oils vapor transmission rate through paper and board packaging materials

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ABSTRACT: This paper reports the development of a permeation test method to evaluate the transmission rate of a volatile organic compound, acting as a mineral oil simulant, through paper and plastic packaging materials. The method uses accelerated conditions, whereby the environment on the one side of a substrate is saturated with the vapor of a model organic compound, similar to mineral oil vapor, namely n-heptane. A suitable adsorbent is placed on the other side of the substrate to ensure a constant gradient of vapor pressure by adsorbing the organic vapor as it migrates through the substrate. This mechanism enables the measurement of the heptane vapor transmission rate through food packaging materials, which is an indirect measure of the mineral oil barrier properties. This research demonstrated that barrier-coated paperboards have the ability to behave in the same way as, and often even better than, commercial plastic films toward the migration of mineral oil.

Application: This new test method provides a quick and easy means to compare the ability of different packaging substrates to behave as barrier materials against the migration of mineral oil from primary, secondary, or tertiary packaging into foods via the vapor phase.

Recycling in the paper industry is encouraged as an economic way of ensuring the sustainability of natural resources and as a way of reducing the increasing levels of municipal solid waste. Food packaging commonly is made of recycled materials, which has a favorable environmental impact and economic benefits, such as the final cost of packaged articles. The recycling process also introduces a number of undesirable and often unknown compounds into the final packaging. A recent study found that mineral oils, originating mainly from the recycled fiber in paperboard (PB), can migrate via the vapor phase into food contained in recycled packaging [1]. This prompted further investigation, because such mineral hydrocarbons often are not food-grade approved, and toxicological assessments of the complex mixture of compounds remain uncertain.

Mineral hydrocarbons present in PB manufactured from recovered fiber have a carbon chain length in the range of n-C_{14} to n-C_{45}, but migration into foods is significant only for compounds with chain lengths up to n-C_{24}, and to a lesser extent up to n-C_{28} [2]. The reason for this is that migration of these hydrocarbons into foods occurs via the vapor phase [3], hence the ability to migrate remains proportional to their partial vapor pressure.

Mineral oils found in recovered fiber originate mainly from non-food grade solvents present in printing inks used in the newsprint industry (offset printing). Mineral oils in foods also can originate from several other sources, including inks from offset printing for decorative printing on cartons, waxes used to improve the water resistance of PB, components in adhesives, diluents for binders, and even additives used in the preparation of the foods. Most of these mineral oils are food-grade quality and typically are highly refined, indicating that all unsaturated and aromatic hydrocarbons have been removed [4,5]. Mineral oils used in offset printing inks generally are technical grades not intended for food contact applications, and therefore contain a combination of mineral oil saturated hydrocarbons (MOSH) and mineral oil aromatic hydrocarbons (MOAH).

Mineral hydrocarbons most commonly are analyzed by online coupled liquid chromatography and capillary gas chromatography (LC-GC) [3,6-8]. In the case of food analysis, Droz and Grob [3] used liquid chromatography (LC) preseparation with a silica column to separate naturally occurring oils in food from the mineral oil hydrocarbons. GC equipped with a flame ionization detector (FID) was then used to separate the mineral oil hydrocarbons according to carbon number. This presented the mineral oil hydrocarbons as a broad hump of unresolved material, topped by n-alkane peaks. Even though MOSH were not separated from MOAH, the broad hump indicated that more than 98% of the mineral oils had a branched or cyclic structure, and the total mineral oil content in food was quantified successfully [3]. In the case of gasoline and diesel samples, the LC stage was used to separate mineral hydrocarbon groups such as saturated, unsaturated, aromatic, and polar compounds. GC-FID allowed separation of the different groups according to carbon number [6].

Determination of mineral hydrocarbon levels in foods proved to be complex. The method evolved by the use of dual-column liquid chromatography (i.e., LC-LC-GC-FID) [9-

11], involving two silica gel columns; the first to separate fats and edible oils from hydrocarbons, and the second to separate saturated mineral hydrocarbons from unsaturated hydrocarbons naturally occurring in food oils or fats. Saturated n-alkanes also are present in natural products, but these usually are recognized by predominantly odd-numbered carbon atoms (which have larger peak sizes compared with the even-numbered paraffins), and thus can be distinguished from those of mineral origins. However, this method takes into consideration the presence of MOSH only, and MOAH is not included in the quantification of the mineral oil contents in foods. Similarly, Moret et al. [12] described a method using two-step liquid chromatography with intermediate solvent evaporation (SE), and automatic transfer to GC-FID (i.e., LC-SE-LC-GC-FID), by which mineral hydrocarbons are separated from food extracts such as fats and edible oils in the first silica gel column, and paraffins are separated from aromatics in the second aminosilane column. This method has been improved by using activated aluminum oxide for removal of long-chain n-alkanes originating from plants [4]. Furthermore, characterization of mineral hydrocarbons was achieved with high performance liquid chromatography (HPLC) followed by two-dimensional GC [1]. Results showed that foods packed in printed, recycled PB contained iso (branched) and cyclic MOSH and highly alkylated MOAH with predominantly one to three aromatic rings.

MOSH and MOAH consist of extremely complex mixtures, making it difficult to obtain suitable standards for calibration. For this reason, GC-FID still remains the method of choice for mineral hydrocarbon analysis; GC for its capability of separating hydrocarbons according to molecular mass, and FID because the response for a certain amount of paraffins is independent of the composition [13]. Quantification of the broad humps of unresolved compounds is still analyst-dependant. MOSH and MOAH can, therefore, be characterized and quantified separately only if these two groups are pre-separated.

As precautions against mineral oil migration, the German Federal Institute for Risk Assessment has proposed replacing recovered fiber with virgin fiber for all food contact products, use of inner liner bags from materials impermeable to mineral oil, or application of mineral oil barrier coatings to the inside of paper packaging that has direct contact with food. The latter option presumes several advantages, such as preventing migration of mineral oils from non–food grade decorative printing inks on the outside of the packaging, which, for instance, would not be prevented by the substitution of secondary fiber with virgin fiber. The use of additional packaging materials, such as inner liner bags, also would be an efficient route, but would require modification of the packaging processes, with the associated risk of increasing packaging costs substantially.

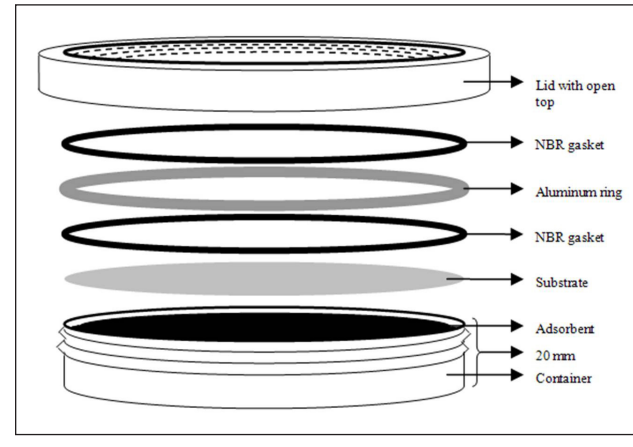
Mineral oil migration studies have only been carried out on food and board samples that have been stored under appropriate conditions for lengthy periods or taken from the shelf before the expiry of shelf-life. Consequently, these tests have been limited in terms of determining the total mineral oil migration at the expiry date. The total migration potential was estimated by assuming that 70% of all mineral oils up to n-C_{24} present in the PB will migrate into foods [2]. This assumption does not take into account novel strategies proposed to prevent mineral oil migration, such as the use of inner bags or barrier coatings between the mineral-oil-containing packaging and the food. In these cases, actual migration studies need to be carried out in order to measure the capability of mineral oil to migrate into foods through a barrier material. Such tests could take from a few months up to years, to determine the actual migration potential under real conditions of use.

This paper presents a rapid test method for evaluating and comparing the mineral oil barrier properties of PB intended for food packaging. The method differs from the previously mentioned complex analytical methods in that the transmission rates of mineral oil migrants through barrier substrates are measured with a highly accelerated permeation method, rather than measuring absolute concentrations of mineral oil in packaging materials and foods. The method can be used for screening different mineral oil barrier materials and coatings, and also for assisting in product development of coating formulations with mineral oil barrier properties. Because this method is quick, easy, and inexpensive, it also can be used by papermakers as a quality control tool to quickly monitor, in a cost-effective manner, the ability of paper and board packaging production lots to sustain tolerable transmission rates of mineral oil and other volatile organic compounds from primary, secondary, or even tertiary packaging into foods via the vapor phase.

EXPERIMENTAL

Materials

Shallow aluminum permeation cells (height 20 mm) with open-top, screw-on lids were manufactured (**Fig. 1**). Aluminum was used because it is impermeable and light enough to be weighed accurately on an analytical balance. An aluminum ring (inner diameter 79.70 mm, outer diameter 87.70 mm, and



1. Assembly of the permeation cell.

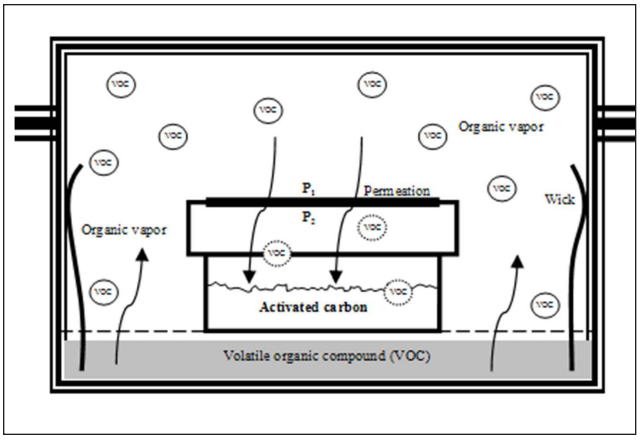
thickness 3 mm) and nitrile butadiene rubber (NBR) gaskets (inner diameter 79.70 mm, outer diameter 87.70 mm, and thickness 2 mm) were used to seal the cells. Other materials included activated carbon powder (100–400 mesh), heptane (CP grade, 99%), and mineral oil (viscosity of 12.5 cP at 23°C) used as a component in offset printing inks. Commercially available plastic films commonly used as food packaging materials were used as model packaging films. These included polypropylene (PP), polyethylene terephthalate (PET), and other polyethylene (PE) films, such as high density polyethylene (HDPE), low density polyethylene (LDPE), and PE-laminated paper. We also used PB of different grammages and paperboard coated with a filled acrylic coating intended for food contact applications.

Testing procedure

For the tests, we prepared desiccators to contain about 2 L of either an organic solvent (i.e., heptane) or mineral oil. We attached 14 flat cotton wicks (2 cm width and about 20 cm in length) to the inside of the desiccators with petroleum resistant adhesive, ensuring that the bottom ends hung in the liquid to facilitate rapid progression to the vapor pressure saturation point. The desiccators were kept closed for at least 24 h to ensure the environment was at equilibrium. Substrates of samples were punched with a circular cutting disk. All samples had the same surface area of 6.04×10^{-3} m² (diameter = 87.70 mm), to fit perfectly between the container and the lid. About 7 g activated carbon was weighed in the container. The substrate and lid, including a three-layered seal (Fig. 1), were used to close the permeation cell. After closing, the surface area of the substrate exposed to the environment was 4.99×10^{-3} m². The permeation cells were placed inside a desiccator containing the saturated environment of organic vapor, and maintained at a temperature of 23°C. After a designated period, the permeation cells were removed from the desiccator, the lid and substrate were removed, and the container was immediately weighed.

METHODOLOGY

Diffusion of volatile organic compounds (VOC) through substrates can occur via two mechanisms: sorption or permeation. In this test method, we used a permeation cell similar to that used in TAPPI T 464 om-06 "Water vapor transmission rate of paper and paperboard at high temperature and humidity." This test cell works on the concept of permeation (**Fig. 2**). The cell is filled with an adsorbent material, and a substrate is sealed onto the open mouth of the cell, with known area. The cell is then placed in a controlled environment of organic vapor pressure and temperature conditions. The organic vapor penetrant is present at high pressure, P_1, on the one side of the substrate and low pressure, P_2, on the other side of the substrate, on the inside of the cell. This causes a pressure differential ($P_1 >> P_2$), which is the driving force for organic vapor flux through the substrate. Diffusion of penetrant through the substrate, and subsequent adsorption onto the adsorbent, allows gravimetric monitoring of the amount of organic vapor that permeates through the substrate by weighing the adsorbent periodically. Keeping the cell in a constant environment of temperature and penetrant partial pressure, i.e. when P_1 and P_2 on both sides of the substrate are held constant, constant diffusion (and thus linear weight uptake as a function of time) of penetrant through the substrate occurs. This is known as the steady-state permeation [14]. The quantity of organic vapor that passes through a unit area of the substrate can be plotted as a function of time to obtain the permeation curve. The slope of this curve under steady-state conditions gives the transmission rate of the organic vapor through the substrate.



2. Permeation setup.

When heptane is used as the organic vapor, for example, the heptane vapor transmission rate (HVTR) can be defined as the mass of heptane-saturated vapor transmitted per unit of surface area of a specific substrate per hour, and can be calculated by the following equation:

$$\frac{wt_2 - wt_1}{A \cdot t} = HVTR\,(g/m^2/hr) \quad (1)$$

where wt_1 and wt_2 are the weights of activated carbon before and after exposure, respectively (measured in grams), A is the area of the exposed substrate (measured in square meters), and t is the time (measured in hours).

The penetrant could be any organic hydrocarbon that resembles mineral oils, but a lower molecular weight compound, such as heptane (C_7H_{16}) or octane (C_8H_{18}), generally would facilitate even higher diffusion rates than those already achieved by the concentrated environment, thus facilitating a highly accelerated method. The adsorbent material should be a substance that resembles dry foods, which are the types of food vulnerable to mineral oil migration from PB packaging materials, and with a high adsorptive capacity towards nonpolar vapors. Modified polyphenylene oxide is a typical dry food simulant, but this adsorbent material is rather expensive [15]. Activated carbon is a type of adsorbent with a very high

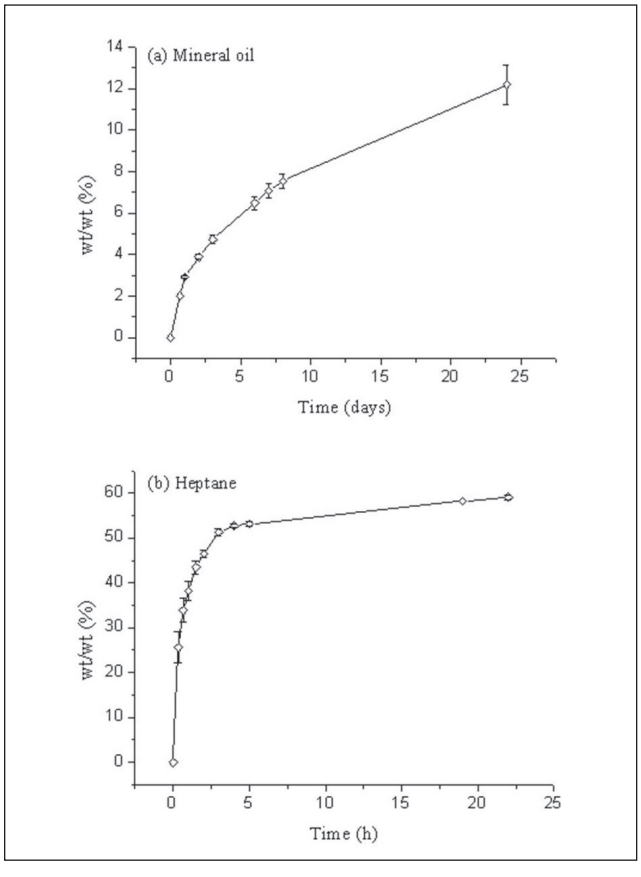
specific surface area [16]. It has a highly porous structure, which allows for the adsorption of organic vapors, and it often is used in gas purification filters to remove oil vapor and other volatile organic hydrocarbons [17-19]. Activated carbon would therefore constitute a suitable adsorbent of mineral oil vapor or its corresponding simulant vapor (i.e., heptane) in this new test method.

RESULTS AND DISCUSSION

Evaluation of heptane and activated carbon as suitable simulants

We selected heptane as a possible simulant of mineral oil, and activated carbon as a simulant of dry foods. An open test cell containing only activated carbon and no substrate was placed inside the saturated organic vapor environment. **Figure 3a** shows the adsorptive capacity of activated carbon to adsorb mineral oil vapor. The mineral oil vapor is adsorbed readily by the activated carbon, but full capacity is not reached even up to 24 days exposure time.

The adsorptive capacity of five different types of dry foods (i.e., sugar, cereal, polenta, bran, and rice) within 24 days exposure varied 0-3 wt%, which was much less than the 12 wt% obtained with activated carbon. Because nonpolar vapors adsorb readily onto activated carbon, this material has a higher adsorptive capacity than dry foods, and will therefore constitute a good simulant that is more severe than dry foods. Figure 3b shows the adsorptive capacity of activated carbon towards heptane vapor. The activated carbon was able to adsorb heptane vapor up to more than 50% of its original weight. Rapid adsorption occurred in the first few hours of exposure, after which the rate of adsorption started decreasing and finally the activated carbon increased up to 55 wt% within 22 h exposure. This indicates that heptane as a mineral oil simulant will be adsorbed much more rapidly than actual mineral oil vapor, due to the smaller size of the heptane molecules and a higher partial vapor pressure at room temperature. Heptane and activated carbon therefore were selected to simulate mineral oil and dry foods, respectively.



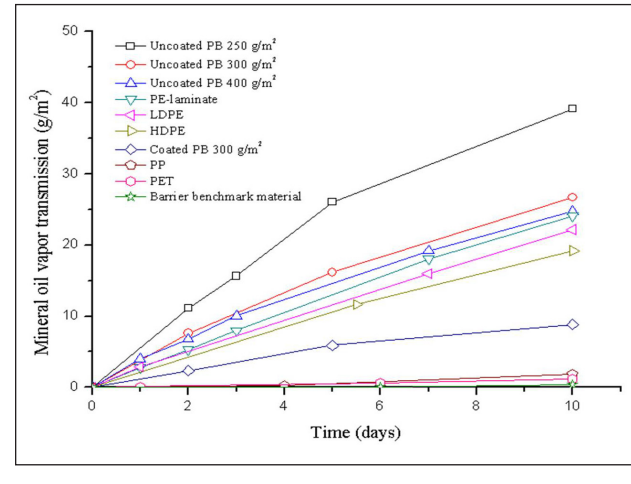
3. Adsorptive capacity of activated carbon for (a) mineral oil and (b) heptane vapor.

Evaluation of the sealing efficiency

The efficiency of the method is highly dependent on establishing a good seal between the substrate and the permeation cell to prevent leaking of organic vapor through the seal. We used a standard barrier benchmark material such as aluminum foil, impermeable to organic vapors or mineral oils as such, to evaluate the efficiency of the sealing system. A permeation cell with this benchmark material as the substrate (aluminum foil with a thickness of 100 μm) in the lid was placed inside the prepared desiccator. Different sealing materials were evaluated as the seal (i.e., Teflon, cork, polyvinylchloride [PVC], and NBR). The adsorbent showed no detectable weight gain (with an uncertainty of ±0.10 g/m²/h) when NBR was used as the sealing material. This served to confirm that no organic vapor passed through the impermeable benchmark substrate, nor through the connection points in the seal. The use of rubber between the metal connections allows for some compression when closing the lid, and prevents damage of the substrate surface.

Evaluation of model packaging materials

Figure 4 shows transmission of mineral oil vapor through various typical food packaging materials. These materials in-



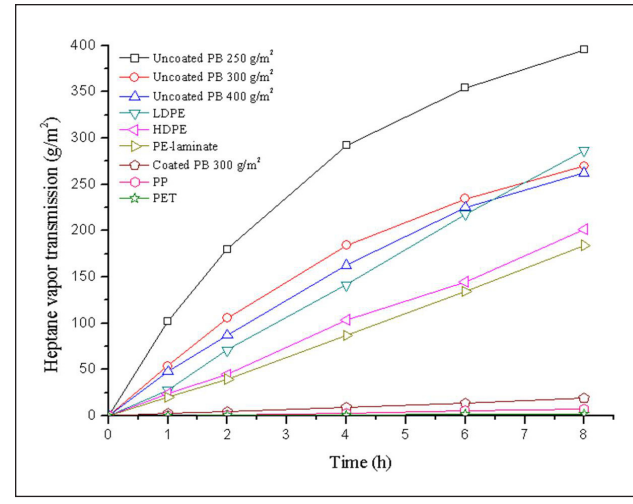
4. Mineral oil vapor transmission through various food packaging materials over a period of 10 days.

Substrate	MOVTR (g/m²/day)	HVTR Calculated from Steady State Conditions (g/m²/h)	HVTR Experimentally Determined within 1 h (g/m²/h)
Barrier benchmark material	0.00	0.10	0.00
PET	0.23	0.24	0.11
PP	0.25	0.55	0.25
LDPE	2.20	35.43	30.75
HDPE	1.84	25.08	26.12
PE-laminate	2.52	23.13	22.68
Uncoated PB 400 g/m²	3.37	40.14*	47.76
Uncoated PB 300 g/m²	4.14	45.65*	59.10
Uncoated PB 250 g/m²	5.45	71.52*	105.09
Coated PB 300 g/m²	0.90	2.36	3.04
*Steady state conditions for uncoated paperboard (PB) samples were achieved within 4 h, and calculated within 8 h for all other samples.			

I. Mineral oil vapor transmission rate (MOVTR) and heptane vapor transmission rate (HVTR) values of different food packaging substrates.

cluded plastic film, plastic laminated paper, uncoated PB, and PB with a coating configuration likely to give good barrier properties to mineral oil vapor. For uncoated PB samples, a deviation from linearity in the mineral oil vapor transmission rate (MOVTR) was reached within 10 days. This is most likely the result of highly permeable materials allowing high quantities of mineral oil vapor to permeate through the substrate and, subsequently, the vapor pressure on the inside of the permeation cell (P_2) increasing. Even though the activated carbon has not reached its full adsorptive capacity, the fast transmission rate and subsequent change in pressure through highly permeable materials leads to a decrease in the transmission rate after about 5 days. We therefore recommended that the actual transmission rate be measured from the steady state conditions (all calculated transmission rates are reported in **Table I**). Furthermore, PB is a porous material, and therefore not considered a barrier material to volatile compounds [20]. Uncoated PB samples, produced from the same constituents, and differing only in grammage had higher transmission rates when the board grammage decreased. The MOVTR increased from 3.37 g/m²/day to 5.45 g/m²/day as grammage was reduced from 400 g/m² to 250 g/m². The boards with higher porosity will exhibit faster migration of organic vapor compared to less porous boards with higher grammage.

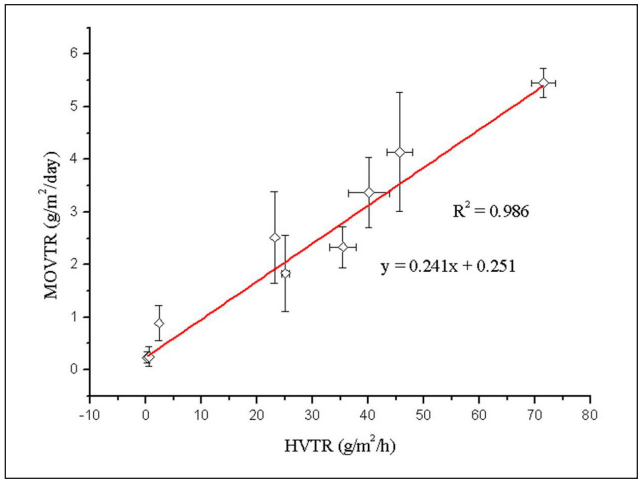
The MOVTR through uncoated PB also was much higher than the plastic materials, considered to be poor mineral oil barriers, such as polyethylenes (e.g., high density polyethylene [HDPE]) and PE-laminated paper. PB will allow easy migration of organic vapors into foods if no barrier is used, but coated board may act as a barrier to organic vapors, as was found with the coated PB 300 g/m² sample. The MOVTR of PB 300 g/m² was reduced from 4.14 g/m²/day for the uncoated sample to 0.9 g/m²/day for the coated sample. Results also confirmed that polyethylene terephthalate (PET) and polypropylene (PP) are very good barriers to mineral oil vapor, as was found in a previous study [2].



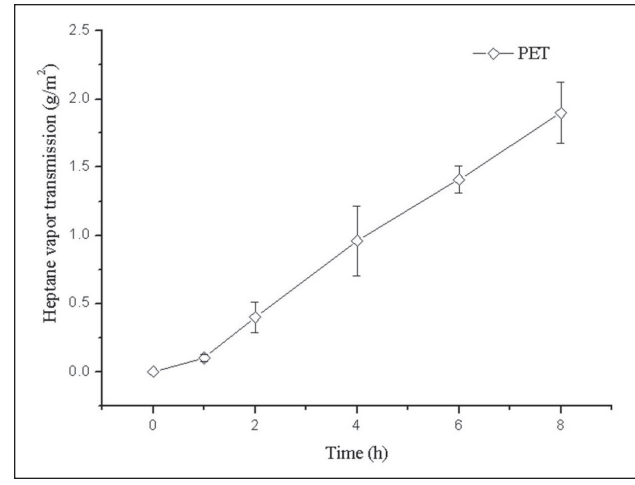
5. Heptane vapor transmission through various food packaging materials over a period of 8 h.

Heptane was used as the penetrant in the accelerated test method, and HVTRs were measured over a period of 8 h. **Figure 5** shows that PET and PP exhibit excellent heptane vapor barrier properties (similar results were found for MOVTR). A similar trend as observed for the uncoated PB with the lowest grammages was observed; namely, the HVTRs started to decrease after 4-6 h. For packaging materials with better organic vapor barrier properties, the HVTR was found to be constant over 8 h, showing a steady state of organic vapor permeation.

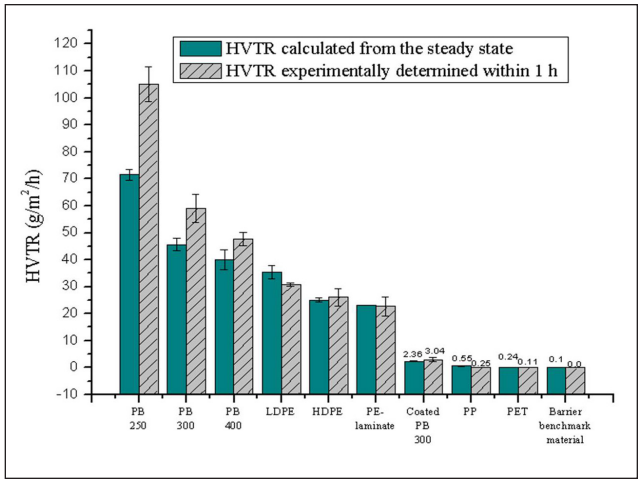
To establish whether HVTR values are representative of true MOVTR, we plotted MOVTR values against HVTR values (**Fig. 6**). A linear correlation between MOVTR and HVTR values, with a correlation factor of 0.986, indicates that heptane constitutes a good simulant of mineral oil, and can be



6. Correlation between mineral oil and heptane vapor transmission rates.



8. Heptane vapor transmission through polyethylene terephthalate (PET) as a function of time.



7. HVTR calculated from steady state conditions and experimentally determined within 1 h.

used as a means of comparing the transmission performance of mineral oil migrants through packaging substrates with barrier protective coatings.

Results show that even the shortest measuring time of 1 h was sufficient to distinguish between the HVTRs through the different materials (Fig. 5). HVTR values calculated over a period of up to 8 h, depending on the steady state conditions, proved to be well correlated to the HVTR values determined over 1 h (**Fig. 7**). A relatively high deviation was observed for very good barrier materials such as PP and PET, as the calculated HVTR after 8 h (0.55 g/m²/h and 0.24 g/m²/h, respectively) was higher than the HVTR experimentally determined after 1 h (0.25 g/m²/h and 0.11 g/m²/h, respectively). This might be a result of the time lag taking place at the onset of permeation, which often is found in polymer films with low permeability when the concentration of the penetrant varies throughout the film as a function of time [14]. In these cases, steady state diffusion conditions only occur once a constant penetrant concentration is reached throughout the film thickness. The time lag effect in PET illustrates that the steady state was not reached within the first hour, and was only reached after about 2 h, from which a constant transmission rate was observed (**Fig. 8**). The time lag was only evident for very good barrier materials and, in such cases, it might be necessary to increase the measuring time to ensure the calculated HVTR values are representative of the actual permeability of the barrier material.

Evaluation of the repeatability

Repeatability studies for HVTRs were carried out on at least three samples. The repeatability was estimated as the relative standard deviation (RSD) of penetrant in activated carbon, calculated by:

$$\frac{Standard\ deviation}{Average} \times 100 = RSD\ (\%) \qquad (2)$$

RSD for HVTR measurements within 1 h varied 2.5% to 12%; except for extremely good barriers with very little vapor mass uptake and for which the time lag effect was evident, the RSD increased up to 21%. However, apart from experimental errors associated with the method, the sensitivity of the balance contributes to 17% RSD for the lowest mass uptake measured. RSD for measurements performed within 8 h were between 0.5% and 6.5%, and good barriers had an RSD up to 10.5%. We therefore recommended increasing the time of measurement to more than 1 h for materials exhibiting low HVTRs. Differences in experimental data between samples could be attributed to disturbances in the heptane vapor–saturated environment as a result of opening the sample chamber during weighing intervals.

CONCLUSIONS

A standard method for measuring HVTR was proposed and evaluated to assess organic vapor migration through paper

and board intended to come into contact with food. The method was evaluated by measuring the permeation through various packaging substrates, highlighting a good correlation between MOVTR and HVTR. We found that measuring times as short as 1 h are sufficient to evaluate the HVTR of a material, should the steady state conditions be reached. Paperboard coated with a suitable barrier coating showed significant improvement in HVTRs, and transmission rates comparable to commercial plastic films were obtained. The advantage of this method is that it is quick, easy, and inexpensive, as opposed to previously reported methods for measuring absolute concentrations of mineral oil in packaging materials and foods. **TJ**

ACKNOWLEDGEMENTS

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ABOUT THE AUTHORS

We chose to conduct this research because the presence of mineral oil in food is a current topic of discussion and one of the priorities for Mpact Limited as a food packaging supplier.

This research differs from previous mineral oil migration studies in that it presents a much simpler method to rapidly predetermine whether coated paperboards have the ability to act as barriers to mineral oil migration. The most difficult part of the study was to keep the methodology as simple as possible, because the issue of mineral oil vapor migration is quite complex.

From this study, we discovered that plastic film was not always superior to barrier-coated paperboard as a means of protecting foods against vapor migration. Mills may use this method in the future as a means of characterizing their product according to its mineral oil vapor barrier properties.

The next step will be to correlate the heptane vapor transmission rate from this work to actual mineral oil vapor migration, as quantified by high performance liquid chromatography-gas chromatography-flame ionization detector (HPLC-GC-FID) from accelerated migration experiments using specific food simulants. In addition, we will follow the progress on toxicological assessments and subsequent regulations regarding the presence of mineral oils in foodstuffs.



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