Journal of
APPLIED PACKAGING RESEARCH

RESEARCH ARTICLE

Influence of Pinholes on Water Vapor and Oxygen Permeation of Packaging Foil and Films

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ABSTRACT

Controlling of moisture and oxygen permeation through the packaging material is essential in most packaging applications. With multilayer structures of different barrier materials, there is possibility to create desired barrier properties to the package. The challenge is to plan appropriate barrier level, e.g. WVTR and OTR, theoretically to the package before producing the package. Furthermore, in specific packages there is a need to control the permeation e.g. respiration in vegetable packages. Typically foils and polymer films have high barrier properties, therefore, permeation can be controlled by producing artificial pinholes in these kinds of packages.

Influence of artificial pinholes in aluminium foils and plastic films on water vapour and oxygen permeation was determined in this study. Results show clear correlation between the size and number of pinholes and gas permeation. Water vapour and oxygen permeation increase significantly when there are pinholes through the structure. It was observed that the greater the RH difference between inside and outside the sample in WVTR measurement, the greater the permeation was through the pinholes. Additionally, it was found that the effect of pinholes on the oxygen permeation on aluminium foil can almost be eliminated by using polyethylene coating on top of aluminium foil.

KEY WORDS
aluminium foil, polyethylene film, pinhole, permeability, gas barrier

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1. INTRODUCTION

Water vapor and oxygen permeation is very important property in packaging materials [1]. The demands for permeation limits depend on the product inside the package [2]. In general, there is a need to be able to control the amount of moisture and oxygen going through the packaging structure. Typically, this is done by choosing suitable materials, e.g. barrier polymers, which have the needed capability. [3], [4] With a multilayer structure consisting of different barrier film materials, there is possibility to create good and versatile barrier properties to the package [5], [6]. The challenge is to plan appropriate barrier level, particularly water vapor transmission rate (WVTR) and oxygen transmission rate (OTR), to package theoretically before producing the package.

In most cases, target is to prevent permeation through the packaging material. However, sometimes certain higher controlled permeation is needed and beneficial like in certain vegetable packages due to respiration [4], [7], [8], [9]. Therefore, higher permeation should be tailored to these kinds of packaging materials. Several studies [7], [8], [9], [10] have been made relating to respiration of packages to find the optimal barrier to the packaging materials. In the previous work [4] it was noticed that artificial pinholes can be generated to control permeation level. It was found correlation between pinhole size and amount of gas going through the film structure.

Some packaging materials, like aluminium foil and metallized layers, have pinholes naturally when they are very thin and therefore, permeation of gases is high [11]. It has been shown [2], [11], [12], [13], [14], [15] that polymer film layer can affect greatly on barrier performance of these materials, e.g. hydrophobic polyethylene film can be used together with aluminium foil to block permeation through the pinholes in aluminium foil. Similar results have been observed with grease proof -papers where good oxygen barrier properties have been achieved by blocking the pinholes with polymer coating [12].

It is well known that WVTR of packaging materials depends greatly on atmospheric conditions (temperature and humidity) [16]. WVTR is increased with rising temperature at constant relative humidity (RH) [16]. Lahtinen [16] studied the influence of the absolute water amount in air on WVTR. The amount of water in air (mixing ratio) can be determined by the temperature and the RH of air – mixing ratio increases with rising temperature at constant RH. Lahtinen [16] found that if the amount of water in air is kept constant in different atmospheric conditions (temperature and RH), WVTR through the pinhole-free polyethylene coated paper is constant.

Target in this study was to determine WVTR and OTR of packaging films and foils with various number and sizes of artificially made pinholes. Additionally, the influence of mixing ratio on aluminium foil containing artificial pinholes was investigated. Furthermore, those pinholes in aluminium foil were blocked with polyethylene extrusion coating and its effect on OTR was measured.

Table 1. Plastic films and their thicknesses and surface energies [17], [18], [19].

Polymer	**PET**	**PET**	**PET**	**LDPE**	**PP**	**OPP**	**BOPA**
Thickness [μm]	**12**	**23**	**50**	**70**	**57**	**22**	**20**
Surface energy	**44-47**	**44-47**	**44-47**	**32-34**	**30-31**	**30**	**36-44**

2. MATERIALS AND METHODS

For this study, several commercially available plastic films and 11 µm thick aluminium foil were purchased. Five commercial polymer films with various thicknesses were used, as follows: polyethylene terephthalate (PET), low-density polyethylene (LDPE), polypropylene (PP), oriented polypropylene (OPP) and biaxially oriented polyamide (BOPA). Film thicknesses and surface energies [17], [18], [19] are shown in Table 1.

The used aluminium foil was Saga (tradename) made by Metsä Tissue and the foil was tested for pinholes. Standard pinhole test [20] was used with the modification of enlarged test area. The test liquid containing red colourant in turpentine is spread on sample and possible pinholes are detected visually from the backside of the foil. Pinhole testing of SAGA aluminium foil can be seen in Figure 1. The typical 11 µm thick aluminium foil contains approximately 100-200 pinholes in one square meter [11].

Figure 1. Pinhole test for SAGA aluminium foil and no pinholes were detected.

Thus, the tested aluminium foil (Saga) seems to be better than usual foils as far as pinholes are concerned since no pinholes were visually detected.

Artificial pinholes were made to the aluminium foil and plastic films with different sizes of needles. The pinholes were made manually by piercing an acupuncture needle through the material. The needle diameters were as follows: 0.2 mm (Hwato) or 0.3 mm (Jiajian). Additionally, a normal pin needle with a diameter 0.6 mm was used. The sizes of artificial pinholes were determined by optical microscope (Zeiss Axioskop 40) with backlight. In Figure 2 is presented a microscopic image of an artificial pinhole made in aluminium foil.

The measurements of water vapor transmission rate (WVTR, [g/(m^2*d)]) were made by cup method (according to the standard ASTM E96-10) or by MOCON Aquatran 1G (according to the standards ISO 15106-3 and DIN 53122-2). WVTR for very low values (less than 1 [g/(m^2*d)]) were measured in different atmospheric conditions from two (Aquatran 1G) parallel samples. Cup method was used when permeation was greater than 1 [g/(m^2*d)] with five parallel samples.

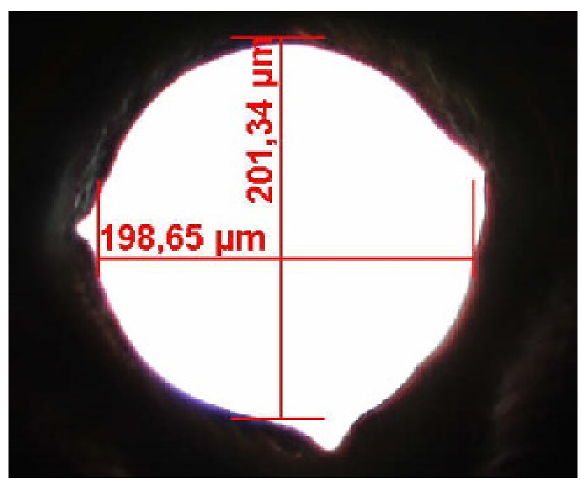

Figure 2. Optical microscope image of a pinhole in aluminium foil made with a Ø 0.2 mm needle.

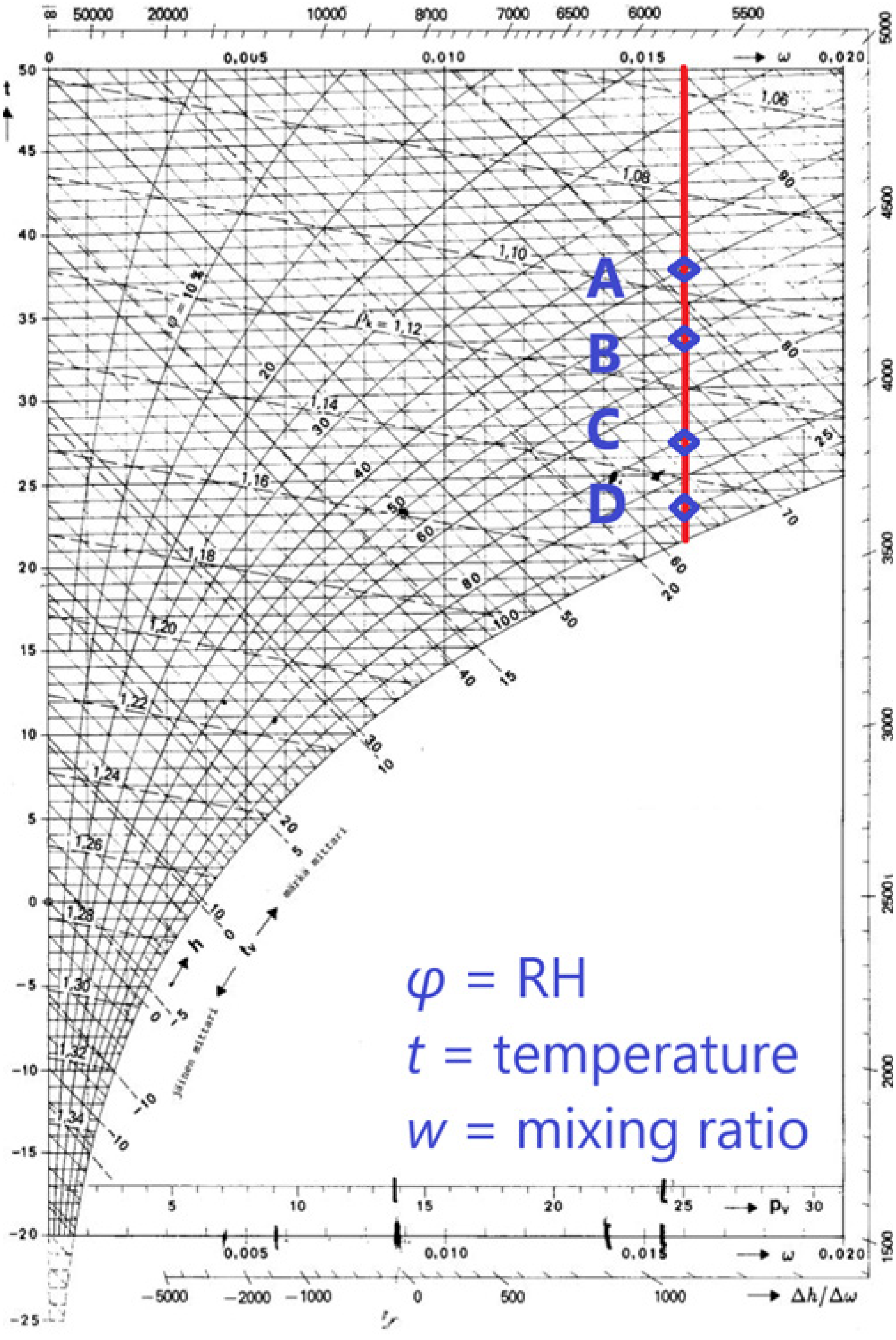

Figure 3. The diagram showing mixing ratio at different temperature and relative humidity levels. Selected conditions are marked from A to D. [21]

Table 2. Atmospheric conditions used in WVTR measurements and pointed out in Figure 3.

Test point	Temperature [°C]	Relative Humidity [%]	Mixing Ratio [%]
A	23	90	1.6
B	27	70	1.6
C	33	50	1.6
D	37	40	1.6

Atmospheric conditions (temperature and RH) for the WVTR measurement were selected according to observations of Lahtinen [16]. Lahtinen found that WVTR was constant when water content in air was constant at various atmospheric conditions. In this study the water content in air (mixing ratio = w) [kg of water / kg dry air] was decided to be constant 1.6% (in Figure 3, $w = 0.016$) by using different temperatures and the matching relative humidity was determined using diagram shown in Figure 3. In Figure 3 [21] the four selected measurement points (i.e. conditions) are pointed out. In these conditions the amount of water in air is the same – 0.016 (= mixing ratio = 1.6%) (Table 2).

The measurements of OTR were made by MOCON Ox-Tran 2/21 SS according to the standard ASTM D3985 from two parallel samples in 23°C and 0% RH. The oxygen content of the used test gas was 10% (nitrogen as a carrier gas) or 100% and the samples had the area of 5 cm^2 or 50 cm^2. For OTR values under 10 [$cm^3/(m^2*d)$] 100% oxygen gas and the area of 50 cm^2 were used. For OTR values over 1000 [$cm^3/(m^2*d)$] 10% oxygen gas and the area of 5 cm^2 were used.

Extrusion coating was used on top of aluminium foil to test blocking of pinholes. For this, the aluminium foil sheets with and without artificially made pinholes (Ø 0.3 mm needle) were taped on top of typical printing paper and then they were coated with LDPE (Borealis CA7230) at Tampere University extrusion coating line. The thickness of LDPE layer on top of aluminium foil was 27 µm and it was placed on the side where the needle was pushed through when producing the artificial pinholes. Figure 4 shows pinholes in aluminium foil with and without polyethylene coating.

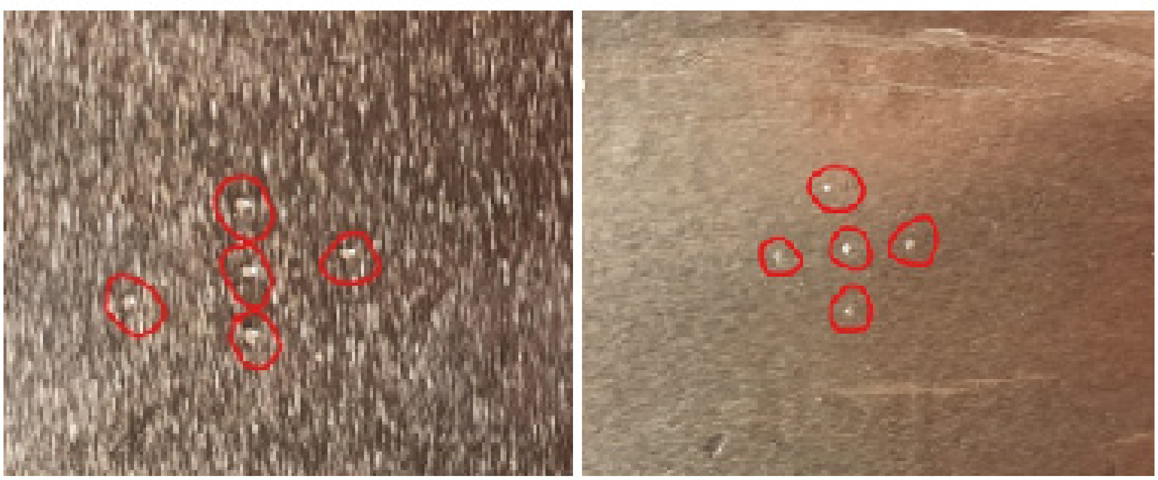

Figure 4. Five 0.3 µm holes in aluminium foil without (left) and with (right) LDPE extrusion coating.

3. RESULTS AND DISCUSSION

Study was started by making different size and number of artificial pinholes on aluminium foil and by measuring their WVTR in different atmospheric conditions. Two different testing methods were compared: cup method with salt or water. WVTR of polymer films was then measured in two different atmospheric conditions with different number and size of pinholes. Finally, OTR of aluminium foil was measured with different number of pinholes and with extrusion coated LDPE layer on top.

3.1. Making pinholes to aluminium foil

The target was to create artificial pinholes to the aluminium foil and study how they affect WVTR and OTR properties. The size of artificially made pinholes was measured by optical microscope with backlight. To evaluate the effect of artificial pinholes on moisture barrier of the aluminium foil, 6 parallel samples were made for each number and size of holes. The diameters of needles were: 0.2 mm, 0.3 mm and 0.6 mm. Table 3 presents the measured dimensions of the holes.

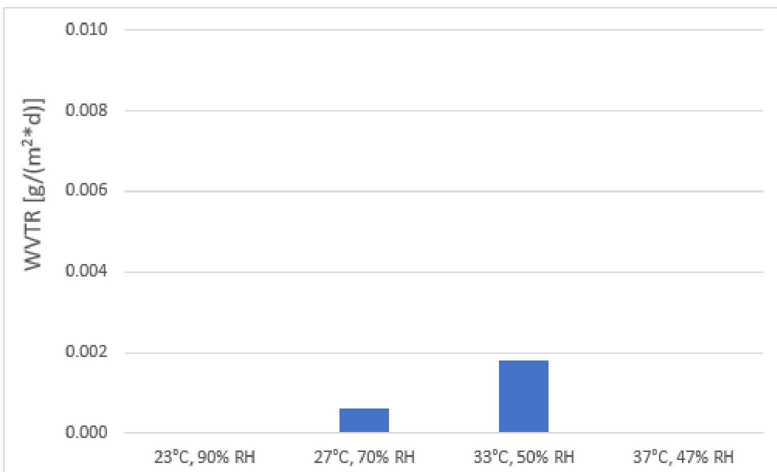

Figure 5. WVTR of aluminium foil (11 μm) in four different atmospheric conditions was measured with Aquatran 1G.

Table 3. The measured dimensions of the artificially made holes in aluminium foil

The size of holes	Ø 0.2 mm hole		Ø 0.3 mm hole		Ø 0.6 mm hole	
[μm]	Height	Weight	Height	Weight	Height	Weight
Average	193	194	305	308	638	641
Deviation	3.6	3.4	7.6	7.2	39	38

Table 4. Theoretical increase of pinhole area due to temperature rise of 10°C.

The size of holes	Ø 0.2 mm hole	Ø 0.3 hole	Ø 0.6 mm hole
Increase of pinhole area	1.45*10-6 mm2	3.27*10-6 mm2	13.1*10-6 mm2

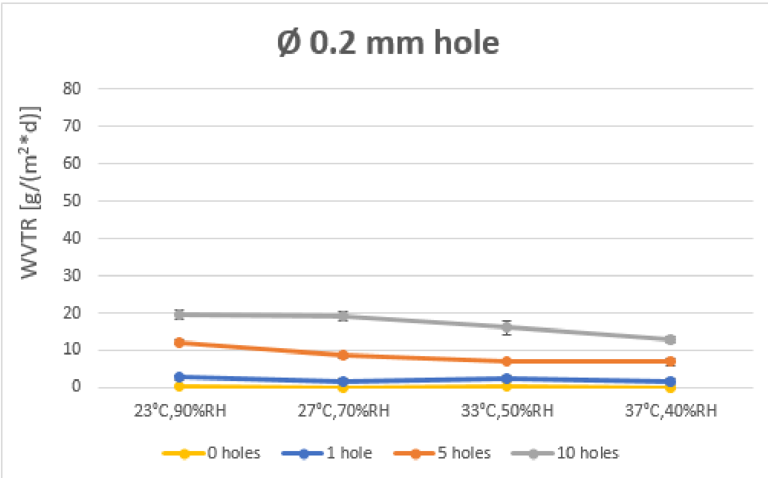

Figure 6. WVTR of pinhole-free aluminum foil in four different atmospheric conditions. Pinhole numbers were one, five or ten and size 0.2 mm.

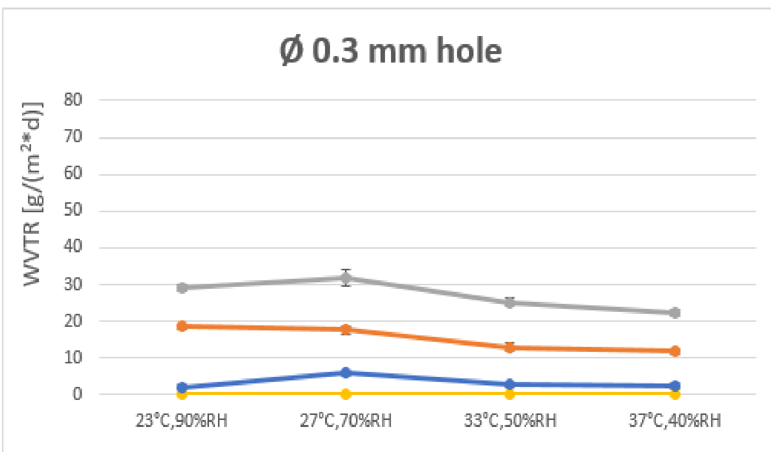

Figure 7. WVTR of pinhole-free aluminium foil in four different atmospheric conditions. Pinhole numbers were one, five or ten and size 0.3 mm.

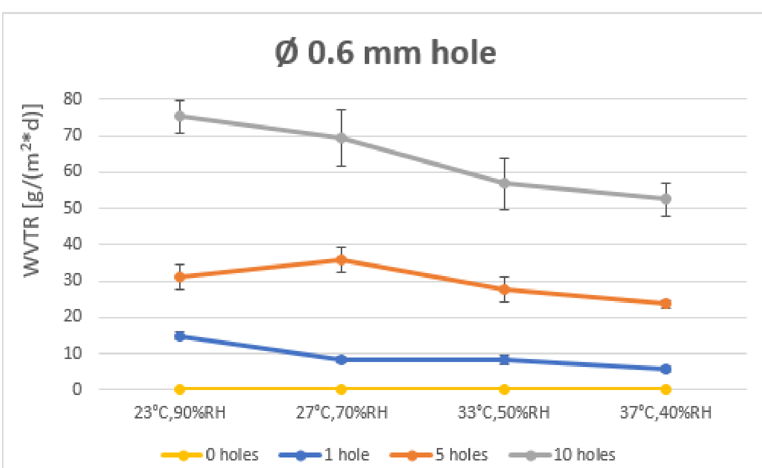

Figure 8. WVTR of pinhole-free aluminium foil in four different atmospheric conditions. Pinhole numbers were one, five or ten and size 0.6 mm.

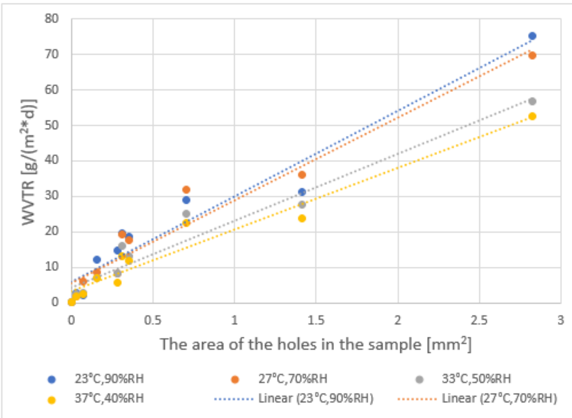

Figure 9. WVTR of aluminium foil in four different atmospheric conditions and with different area of holes.

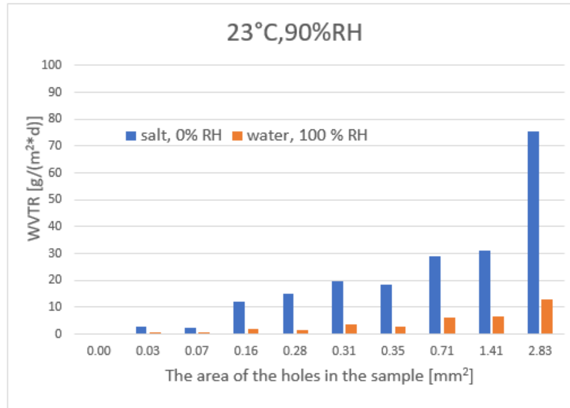

Figure 11. WVTR as a function of the hole area [mm2] and RH inside the cup.

WVTR was measured at four different temperatures 23°C (90% RH), 27°C (70% RH), 33°C 50% RH) and 37°C (40% RH), like shown in Figure 3 where mixing ratios are equal. Instead, the pinholes were made at constant temperature (~ 23°C) to the aluminium foil. The effect of heat expansion on pinhole sizes was calculated, as follows:

The expand coefficient to area (α) with aluminium is 0.00000231/K and areas expand twice as much as lengths do in Equation 1 [22]:

$$\Delta A = 2\alpha A_0 \Delta T \quad (1)$$

Where, A_0 is the area of the pinhole (πr^2) and ΔT is the temperature difference. In the calculation the rise of temperature is 10°C and the area is assumed be circle using the measured dimensions of height and width in Table 4.

Table 4 indicates that heat expansion of aluminium foil is negligible compared to the area of the actual pinholes.

3.2. WVTR of aluminium foil

WVTR of reference samples (pinhole-free aluminium foil) was measured in different atmospheric conditions. Figure 5 presents the WVTR values of pinhole-free 11 μm thick aluminium foil.

Results in Figure 5 show that WVTR of aluminium foil is extremely low and thus confirm that the foil is pinhole-free.

Figures 6, 7 and 8 show WVTR of aluminium foil with pinholes in different conditions. In the studied samples both the size and number of pinholes is varied.

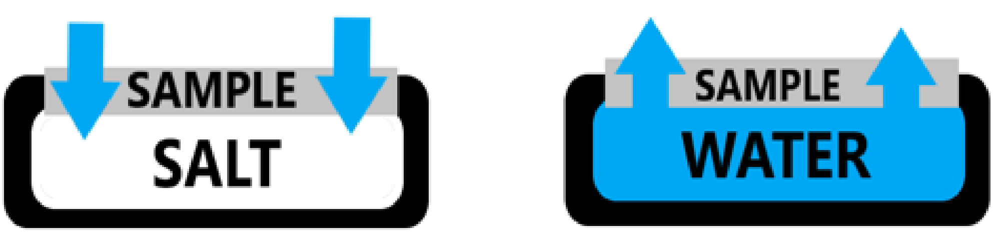

Figure 10

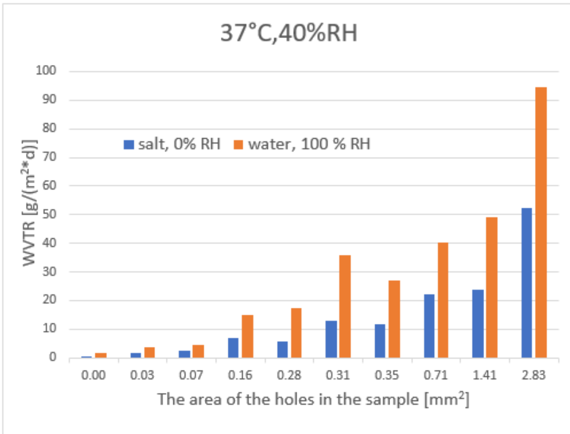

Figure 12. WVTR as a function of the hole area [mm2] and RH inside the cup.

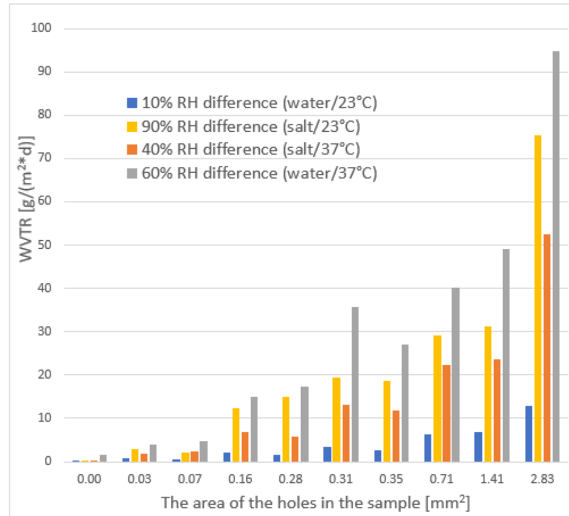

Figure 13. WVTR with RH difference between inside and outside of the cup and hole area. Two temperatures of 23°C and 37°C were used.

Figures 6, 7 and 8 indicate that when the size of pinhole or the number of pinholes increase, the greater are the WVTR values. The deviation of measured WVTR is greater when the diameter of the hole size was larger (especially Ø 0.6 mm) and the number of holes was higher (particularly 10 holes). Like shown in Figure 3, the four different atmospheric conditions (23°C, 90% RH // 27°C, 70% RH // 33°C, 50% RH // 37°C, 40% RH) contain the same amount of water in air (1.6%). In results shown in Figures 6, 7 and 8, where the temperature is increasing and relative humidity decreasing, WVTR of the aluminium foil is about at the same level or decreasing. The results presented in Figures 6, 7 and 8 are combined in Figure 9 where WVTR is plotted against the calculated pinhole area.

It is evident from the results that when the pinhole area is increased, the effect of relative humidity outside of the sample cup is pronounced: the higher the relative humidity, the higher the WVTR values. It seems that as the RH inside the cup was nearby 0% because of salt, the difference between relative humidity inside vs. outside of the cup is significantly affecting WVTR. Therefore, it was decided to examine this further on with another cup test procedure where distilled water replaces salt inside the cup (i.e. 100% RH).

Temperature	RH inside cup	RH outside cup	RH difference	WVTR
23°C	0% (salt)	90%	90%	75 g/m2*d
23°C	100% (water)	90%	10%	13 g/m2*d
37°C	0% (salt)	40%	40%	52 g/m2*d
37°C	100% (water)	40%	60%	95 g/m2*d

Table 5. Cup method (salt vs. water) WVTR results of aluminium foil with hole area of 2.83mm2 in two atmospheric conditions

3.3. Effect of testing conditions on WVTR

In order to study effect of testing conditions on WVTR results, two different test setups were used in the cup measurement (Figure 10), i.e. either salt or water inside the cup. Figure 10 presents in which direction the water vapor permeates through the sample in these WVTR measurement setups.

Figure 10. WVTR test cups showing the direction of water vapour in different test setups: salt inside the cup (left) and water inside the cup (right). If there is salt inside the cup, RH is 0% and if there is water inside the cup, RH is 100%.

In Figures 11 and 12 two extreme RH (the lowest and the highest) conditions were chosen for comparing WVTR results as a function of pinhole area. Additionally, WVTR was measured either by salt or water cup method. In Figure 11 WVTR is presented at 23°C, 90% RH, whereas in Figure 12 at 37°C, 40% RH.

When comparing WVTR results in Figures 11 and 12, it can be seen that at 37°C, 40% RH the average of WVTR values in general are higher than at 23°C, 90% RH. This observation is particularly pronounced at larger hole areas. Furthermore, there is a distinct difference in the measured values between salt and water cup. This can be more carefully analyzed from Table 5, where cup methods are compared in specific hole area (2.83mm^2).

Table 5 shows clearly that in similar atmospheric conditions WVTR results achieved with the salt and water cup methods differ remarkably from each other. Evidently these results can be explained by the increase in RH difference (inside vs. outside cup). Another thing to consider is the role of temperature in results and for analyzing that into Figure 13 are gathered all the results shown in Figures 11 and 12.

Figure 13 presents WVTR with RH difference between inside and outside of the cup and hole area. In addition, two test temperatures of 23°C and 37°C are shown in Figure 13.

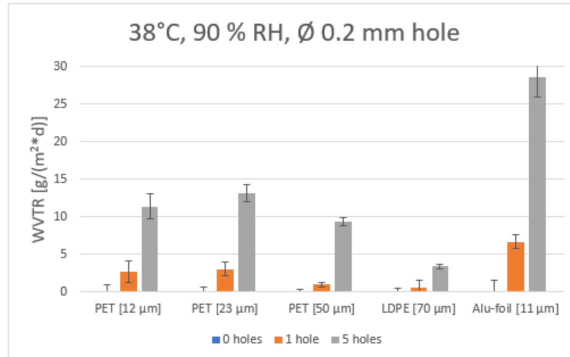

Figure 14. Normalized WVTR (38°C, 90% RH) values of polymer films and aluminium foil containing pinholes. Pinholes were made with a needle having diameter of 0.2 mm.

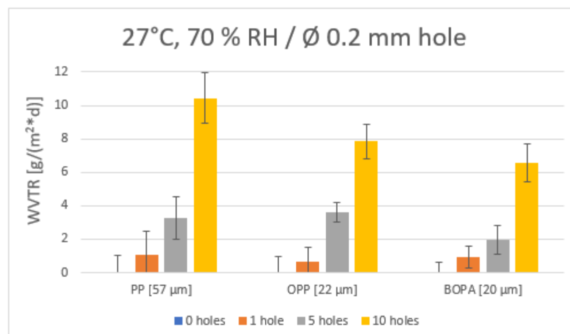

Figure 15. Normalized WVTR (27°C, 70% RH) of polymer films containing pinholes. Pinholes were made with a needle having diameter of 0.2 mm.

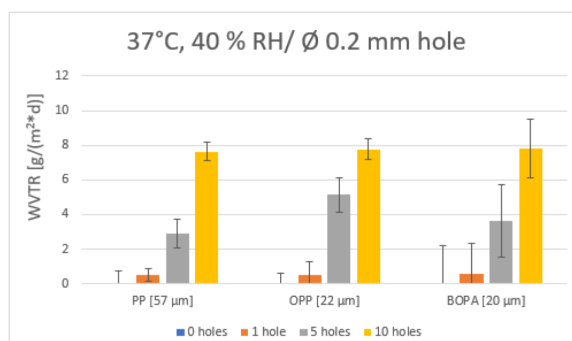

Figure 16. Normalized WVTR (37°C, 40% RH) of polymer films containing pinholes. Pinholes were made with a needle having diameter of 0.2 mm.

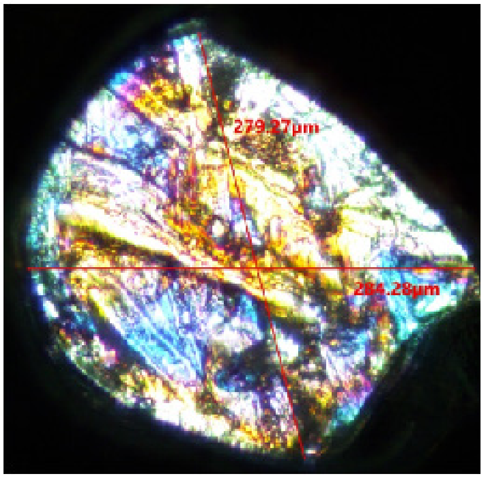

Figure 17. Optical microscope image of the pinhole done with an acupuncture needle (0.3 mm diameter) on aluminium foil. Extrusion coating surface (LDPE) can be seen in the pinhole.

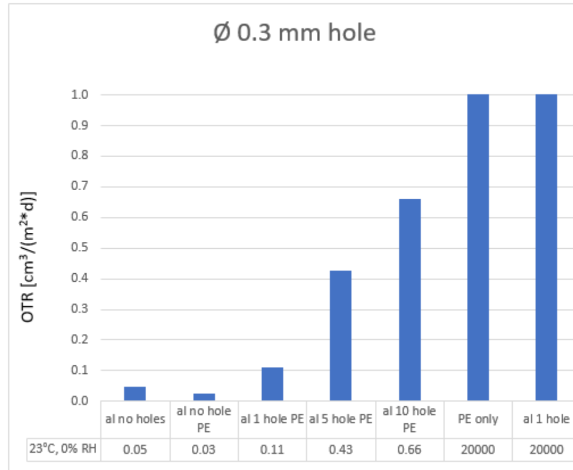

Figure 18. OTR of pinhole-free aluminium foil, LDPE-coated aluminium foil containing zero, one, five or ten pinholes, pinhole-free plain LDPE coating, and plain aluminium foil containing one pinhole.

Because it was shown earlier that RH difference has great impact on WVTR, it was decided to compare the average sums of blue and yellow bars to average sums of orange and grey bars. In this way the RH difference is equal in average sums and the comparison is between the temperatures of 23°C and 37°C. The average sum of WVTR values at 37°C is remarkably greater compared to the average sum of WVTR values at 23°C.

As explained in Table 5 for a large hole area, results in Figure 13 show similar tendency in WVTR results also for the smaller hole areas: the higher the RH difference, the greater the difference in WVTR values.

3.4 WVTR of polymer films

In this part, the target was to compare different types of polymer films and to study how WVTR is affected if they contain pinholes. The chosen polymeric films differ from each other mainly in their surface energy (e.g. hydrophobicity). Aluminium foil was the reference material. Furthermore, the influence of both thickness and orientation of film were studied.

Figures 14-16 present comparative WVTR values in different conditions for polymer films and aluminium foil having one, five or ten pinholes (with diameter of 0.2 mm).

For the sake of easier comparison of WVTR values, the graphs were normalized so that the WVTR value of the pinhole-free film was set as zero (0 holes bar in Figures 14-16). In this way the results should indicate only the influence of pinholes on WVTR. Thus, the effect of pinhole-free film material and its thickness is disregarded. Three different atmospheric conditions were used. Appendix A shows the actual measured WVTR values of Figures 14-16.

According to Figure 14, the most hydrophobic material (LDPE) has the lowest increase in WVTR. Instead, the influence of pinholes on WVTR with reasonably hydrophobic PET film is much less than in the case of aluminium foil (most hydrophilic). The number of pinholes (from one to five) increases logically WVTR values whereas the increased thickness of PET film seems to have only minor effect on WVTR through the pinholes.

Table 6. Calculated OTR through pinholes in aluminium foil filled with LDPE.

Number of pinholes in aluminum foil	Area of pinholes	Measured OTR	Substracted OTR	OTR of LDPE through one pinhole in aluminum foil
	[m^2]	[cm3/m2*d]	[cm3/m2*d]	[cm3/m2*d]
0	0	0.03		
1	90*10^{-9}	0.11	0.08	900000
5	450*10^{-9}	0.43	0.40	900000
10	900*10^{-9}	0.66	0.63	700000

Influence of mixing ratio on WVTR of different polymer films was investigated in Figures 15 and 16.

Mixing ratio has similar effect with polymer films containing pinholes as in the case of aluminium foil (Figure 9): the greater the RH difference (Figure 15, 70% RH vs. Figure 16, 40%) compared to the 0% RH (salt in the cup) the greater the WVTR values. When comparing the normalized WVTR results in Figure 15 and 16 it can be noticed that the effect of pinholes does not depend on thickness, orientation or the chosen polymer type.

Furthermore, Appendix A shows that WVTR of pinhole-free polypropylene films have almost equal WVTR values at the same mixing ratios (27°C, 70% RH and 37°C, 40%). This supports the conclusions Lahtinen [16] made with polyolefins.

3.5 OTR of aluminium foil

The aim of the last part of this study was to measure OTR of aluminium foil with different number of artificially made pinholes and with extrusion coated LDPE layer on top. Figure 17 shows an optical microscope image of a pinhole in aluminium foil. In the pinhole it can be seen the extrusion coating surface that has replicated the paper-like texture. This proves that the extrusion coating has penetrated through the pinhole to the backing paper.

OTR results in Figure 18 show that if there is even one pinhole in the aluminium foil, the oxygen barrier is completely lost. The extrusion coated LDPE layer was 27 µm thick and did not contain any pinholes. This is supported by earlier research where pinholes started to appear not until below about 15 µm thickness in LDPE coating [23]. While LDPE is not a good oxygen barrier itself, it greatly reduced oxygen permeation of aluminium foils containing pinholes.

Additionally, OTR of pinhole containing aluminium foil with pinhole-free LDPE coating seems to follow the number of pinholes. Therefore, the areas of pinholes (1, 5 and 10 pinholes with diameter of 0.3 mm) filled with LDPE were calculated and their theoretical influences on OTR determined. The pinhole-free aluminium foil with LDPE had OTR of 0.03 cm^3/(m^2*d) which is subtracted in Table 6 from the pinhole containing OTR results (Figure 18). The oxygen permeation through the LDPE coating in the areas of pinholes in aluminium foil were then calculated (Table 6).

Results indicate that pinholes destroy oxygen barrier of aluminium foil, but it can be greatly improved by extrusion coating with LDPE. Similar effect has been shown earlier by other researchers [12], [24]. OTR through pinholes filled with LDPE coating increases with the area of pinholes. It was possible to measure OTR of LDPE through the pinholes and the values ranged from 700000 to 900000 cm^3/(m^2*d). A typical LDPE layer has the OTR of 7000-8500 cm^3/m^2*d, when the film thickness is about 25 µm [25], [26]. Thus, the difference between the calculated results and literature values is about two decades. Ge et al. [27] have studied the effect to WVTR and OTR, when there is added extra layer/layers on top of aluminium foil or metallized PET film and the structure is flexed. They found out that for all laminates WVTR was less severely affected by flexing than the OTR [27]. The phenomenon, how adding a sealing (or any extra film) layer on top of different kind substrates or laminates affects to permeation properties, should be explored more carefully in future studies.

4. CONCLUSIONS

This study focused on barrier properties of aluminium foil and polymer films with artificially made pinholes. The permeability through the material consists of gas going through the matrix and possible pinholes. The results show that water vapor and oxygen permeation increase when there are pinholes in the packaging foil or film. Especially this was pronounced in oxygen permeation of aluminium foil where only one pinhole destroyed the oxygen barrier properties. While the pinholes in aluminium foil were covered by a polyethylene film, OTR values of the aluminium foil decreased to very low levels (less than 1 $cm^3/(m^2*d)$).

By using various number and sizes of pinholes, the areas of pinholes could be varied. It was found a clear correlation between the area of pinholes and the permeation values – when the area is increased the permeation increases. Hence, by artificial pinholes with specific size and number, it is possible to optimize and control water vapor and oxygen barrier properties of a packaging structure.

Furthermore, it was a clear conclusion that the atmospheric conditions inside vs. outside of the cup in WVTR measurement affect greatly permeation of water vapor through pinholes. According to the results, the most decisive factor influencing WVTR through pinholes was relative humidity. The greater the difference in relative humidity was between inside and outside of the pinhole containing sample, the greater the permeation of water vapor was to the dryer side of sample.

It would be worthwhile to investigate atmospheric conditions more carefully to find out accurate correlations in the case of pinholes. By utilizing various atmospheric conditions, it can be possible to model the permeation of water vapor through pinholes.

This study focused on barrier properties of monolayers. It would be interesting also to measure water vapor and oxygen permeation through multilayer polymer films containing pinholes. Additionally, by laminating films together, the influence of pinholes on permeation only in one single layer could be investigated.

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APPENDIX A

Figure 14 Results

[g/(m2*d)]		Ø 0.2 mm hole	
38°C, 90% RH	Number of holes		
Film with thickness	0 holes	1 hole	5 holes
PET [12 µm]	36.9	39.6	48.3
PET [23 µm]	21.2	24.2	34.4
PET [50 µm]	10.6	11.6	19.9
LDPE [70 µm]	5.1	5.7	8.5
Alu-foil [11 µm]	1.6	8.2	30.3

Figure 15 results

[g/(m2*d)]	Ø 0.2 mm hole			
27°C, 70% RH	Number of holes			
Film with thickness	0 holes	1 hole	5 holes	10 holes
PP [57 µm]	1.0	2.1	4.3	11.5
OPP [22 µm]	1.9	2.6	5.6	9.8
BOPA [20 µm]	43.7	44.6	45.7	50.3

Figure 16 results

[g/(m2*d)]	Ø 0.2 mm hole			
37°C, 40% RH	Number of holes			
Film with thickness	0 holes	1 hole	5 holes	10 holes
PP [57 µm]	0.9	1.4	3.8	8.5
OPP [22 µm]	2.4	2.9	7.5	10.2
BOPA [20 µm]	30.6	31.2	34.2	38.4