CORNET

NOTEBOOK NO. 9

ISSUED TO GEORGE SADLER

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Abbreviation Table

Abbreviation	Definition
HPP	High Pressure Processing
PET	Polyethylene terephthalate
MOCON	Ox-Tran ten fifty oxygen permeation tester

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Project No. ~~Acme Cerone~~ 1

TITLE Impact of HPP on O_2 Permeation of PET Book No. _____

PURPOSE - Little published data exists identifying the impact of HPP processing on individual polymer films. While a larger volume of data exists for multilaminate structures, these often fail to provide insights into the behavior of each constituent polymer.

The purpose of this experiment it to determine wheather HPP influences the O_2 permeability of PET,

METHODS

A) Squares of PET film (½ mil, ~50% crystalline) were cut from roll stock. Samples were > 4 in² to accomodate mounting into the mocon unit.

B) Four squares were folded into paper lab napkins and sealed dry into Kaypak laminate pouches, 1 square per pouch. Four additional samples were folded (no napkin) placed in Kaypak pouches to which sufficient water had been added to surround and allow mobility to the folded polymer samples. Each pouch contained 1 polymer square.

C) Polymers were processed to the following conditions

Initial Temp prior to comp. _____
Final Temp e compression. _____

Final Pressure _____
Hold Time at max (final) pressure _____

Time held before HPP but after packaging _____
Time post-process before pochets were opened _____

Recorded by:	Date	Verified by:	Date
Jamye Adler	1/07/05		

D. Two samples of the dry, napkin-wrapped PET samples were removed from the KayPak overwrap, trimmed to size and mounted into the Mocon unit. Similarly two samples of the water-exposed PET polymer were removed from their KayPak overwrap, trimmed for mounting into the Mocon, and sealed into the unit.

Film time in mocon before purging 15 hrs
Film time in Mocon for carrier purge (Bypass Sensor) _____
Film time in Mocon for carrier purge / insert sensor _____

E. Test gases were 3% Hydrogen in N_2 carrier and Pure O_2

Observations & Results

1. Napkin fibers imprinted on PET films. Fiber-embossed films were matte in appearance and no longer transparent.

2. Films were mounted into the Mocon unit in the following order

A) (Napkin) (Napkin) LEAK (Water) (Water) NO FLOW (47cc per day STD)

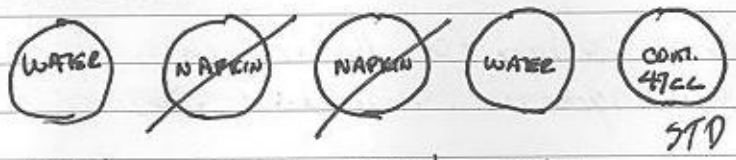
172 cc / day · m² · sec

188 cc / day · m² · sec

47cc/day · N² · atm

Recorded by: | Date | Verified by: | Date

B) Run 2 - New polymers · 4days later- same process batch as "A"



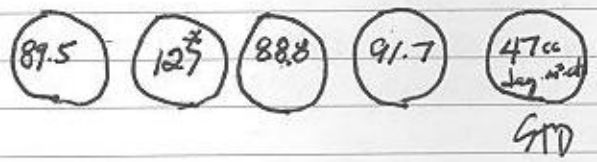
125 cc Pinhole Pinhole 94.6 cc

day · m² · atm day · m² · atm

Sensor was removed when samples 2 & 3 (HPP in napkins) reading exceeded 2500 cc/day m² · atm A "scarlet moo" dye test indicated that pinholes had formed at fold lines.

Neither the water or napkin samples lacked creasing; the two water samples were not pierced by the process.

C) Run 3- Control (NO HPP) PET



* The high O_2 reading at station #2 may be due to leakage. The baseline reading to cell #2 was ~ 379μV compared to 39μV for other stations. While the 127cc/day · M² · atm reading posted above is corrected for baseline; the coincidence of high background and high adjusted values on this one cell is suspicious.

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Run #4

Virgin 1/2 mil PET samples were bathed for 16 hrs in water to simulate samples prepackaged for a HPP run scheduled for the following morning.

At 16 hours samples were removed, mounted on the MOCON, purged with N_2/H_2 carrier gas as per SOP. Then run for oxygen at ~30min. There results were:

2683 MV	7837 MV	8581 MV	9081 MV	2340 MV
53.8 cc / day·M²·atm	157 cc / d·m²·atm	172 cc / d·m²·atm	182 cc / d·m²·atm	CONT. 47 cc / d·m²·atm

PET sample treated as described under #4 above were held 4 days in the MOCON w/o flow before testing the final readings were.

125	124	129	125	CONT. 47

$$\frac{cc\ O_2}{Day \cdot m^2 \cdot atm\ O_2}$$

Conclusions -

PET is known to decrease in O_2 permeation when moisture is high. The results are inconclusive. It appears the trend is followed with station #1 (538 cc/day·M² atm.) However subsequent readings suggest an increase in migration which slowly falls with time.

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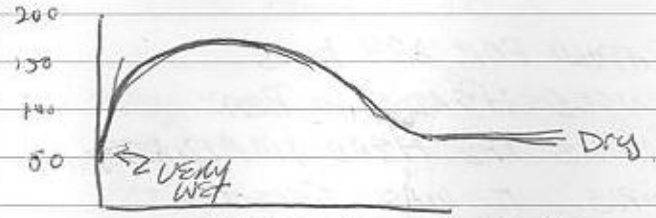
IF A GRAPH WERE TO BE MADE IT WOULD HAVE AN IMPROBABLE DYNAMIC BELOW



DECREASING MOISTURE.

NEXT STEPS

(1) ~~REAT~~ REPEAT VIRGIN MATERIAL. THIS WILL SHOW WHETHER THE MACHINE IS DRIFTING.

(2) REPEAT High MOISTURE. HOWEVER only USE 1 head and TEST FRESHLY REMOVED SAMPLES only

RUN #5 IN ACCORDANCE WITH STEP 1 ABOVE. VIRGIN PET NON-PROCESSED SAMPLES WERE EXAMINED FOR O_2 MIGRATION. THE RESULTS WERE

1065 μV	1108 μV	1088 μV	1093 μV	390 μV	SENSOR DISCARDED
126	133	131	131	47	

$$\frac{cc\ O_2}{day \cdot m^2 \cdot atm}$$

RUN #3 & RUN #5 WERE BOTH CONDUCTED ON VIRGIN MATERIAL USING THE SAME PROTOCOLS. O_2 PERMEATIONS SHOULD THEREFORE BE SIMILAR. HOWEVER RUN 5 DATA ARE ~50 cc/day·m²·atm Greater than RUN #3

CAUSES?

1) NEWLY RECHARGED SENSOR
2) > H_2O in Bubbler
3) FLOW > High in #5 for Carrier

Recorded by:	Date	Verified by:	Date

RESULTS OF UNTREATED PET. IN H_2O

SEVERAL REPLICATIONS OF PET BATHED FOR ≥24 HOURS HAVE BEEN EXAMINED. ONLY A SINGLE HEAD HAS BEEN USED TO MINIMIZE ERROR. THIS IS THE HEAD IMMEDIATELY TO THE RIGHT OF THE CONTROL SAMPLE. IT WAS SELECTED SINCE IT HAD THE GREATEST CONTROL OF N_2 FLOW! THE OTHER HEADS WERE TAKEN OFF ACTIVE STATUS. THE RESULTS GAVE:

SAMPLE 1	$\frac{767\ mV}{311\ mV}$	116 cc/day. M^2. atm	~ 24hrs in H_2O
SAMPLE 2.	$\frac{20,000\ uV}{500\ mV}$	1880 "	~ 48hrin H_2O
SAMPLE 3	$\frac{800\ uV}{436\ mV}$	86	~ 24hrs "
SAMPLE 4	$\frac{812\ mV}{453\ mV}$	84	~ 24hrs "
SAMPLE 5	779/420	87	~ 48hrs "
	9500 ~~9720~~/588		96 Hr
Sample material	32,000/588		
	800/514 = 73		100hr + Grease

Erratic readings are common. Some Mocon unit refinements were made. These consisted of.

1) The screw fastener on one of the film stations was stripped. The knob was replaced with a standard hardware screw which required a screw driver to close in lieu of the screw knob. The top half of the screw was filed down as shown below

The clamp design required the upper screw threads to be removed in order for tight clamping to occur

2) New high precision needle valves were installed in the 5 film stations. Their operation improved uniformity of flow to the film stations. However, the valves did not appear identical to replaced units. Perhaps as evidence of difference the flow rates were limited to the first 10% of the flow meter while purge flow to the upper polymer surface was excessive.

3) A needle valve was inserted as a restrictor to the gas in the upper film chamber. This allowed careful control of upper/lower ratio of flow control.

4) Flow tubes were cleaned with chromic acid, washed with water and finished with an acetone rinse. Cleaned tubes were dried approximately 2 hours at 100°c, then examined for fluid and remounted into the Mocon Vait

The electric connections, currently intertwined, should be soldered. Hmm where did I put that solder?

Recorded by:	Date	Verified by:	Date 4/21/05

Project No. 1

Book No. 1 TITLE TOPPAN Films

O_2 Permeation through Toppan Films

Page 7 listed several innovations to the Mocon 10-50 machine. The impact of these changes was assessed on a commercial structure called TOPPAN

Results:

25 μV	8 μV	2 μV	25 μV	2,350 μV
0.5 cc	0.16 cc	0.04 cc	0.5 cc	47 cc
day · m² · atm	d · m² · a	d · m² · a	d · m² · a	Control

READINGS are lamentably only approximate. There was significant lag in Achieving equilibration. I am Changing the Colouy detector in hopes sensitivity will increase.

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TITLE Recommissioning of Mocon unit

Book No. _____

BACKGROUND: Mocon efforts were abandoned previously due to erratic readings. Part of the problem arose from over hydration of the colorimetric cell. When pure water was used in the bubbler the detector became swamped with condensate. When the moisture trap was removed from the flow stream the sensor dried out. either the sensor needs to be heated or the hydrated flow gas

Procedure:

(1) The N_2/H_2 bubbler was cooled to 10°C

The mocon unit was set to 10 minutes and placed on automatic mode. Each time the sensor advanced from Station 1 (containing 47cc Std PET 1/2 mil film) A spike in reading is produced. This spike has become sharper and thinner with each subsequent scan. IT appears once the rotor advances to a given ~~new~~ station Oxygen reading is minimal.

Conclusion

When advancing from station 1 to station 5 the inset sensor valve must be turned out for 1 minute so O_2 spike can bypass the sensor:

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			Oct 20 '05

OXYGEN PERMEABILITY and SEAL STRENGTH

Oxtran:

The Oxtran 10/50 (Mocon Instrument, Modern Controls Inc, Minneapolis, Minnesota) measures the rate at which oxygen gas passes through a barrier material clamped into its test chambers.
The following test materials to be evaluated:
Alcan (60 Biaxial Nylon / Adhesive / 5.0 ml EVOH Coextruded Sealant), Alcan MRE (Metallized container), Smurfit-Stone
Pyramid clear tranparent (Nylon/polyolefin), Pyramid Flexible Packaging
Pyramid metallized package(Nylon/polyolefin), Pyramid Flexible Packaging.
12.5x12.5 cm (5x5 in) diameter round shape specimens were cut from the pouches.
The test process consist of following:
Testing material is clamped in testing chamber. The test specimen requires a period of time to equilibrate to the testing conditions and reach equilibrium. The conditioning period varies and is a function of many factors such as barrier composition, thickness and test temperature. When equilibrium is reached nitrogen and 2% H_2 (carrier gas) is routed through the lower half of the cell. Oxygen is fed through upper test cell. Oxygen permeates through the specimen and is picked up by the carrier gas. The amount of oxygen in carrier gas is measured by the coulox sensor to determine the transmission rate.

Instron:

For the seal strength test, 24.5 mm wide and 95 mm length specimens were cut from the pouches. An **Instron** series **4302** tensile testing machine (Instron Corporation, Canton, Mass) equipped with data acquisition system was used to measure stress-strain behaviour. The seal strength from each specimen was obtained from the maximum peak of the stress-strain curve.