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Determination of Percentage of Polyethylene in Paper

Submitted to Western Michigan
College, Kalamazoo, Michigan
in partial fulfillment of the
requirements of the Course
"Research Problems in pulp and
paper".

James Alan Lewis

November 28, 1955

ABSTRACT

The purpose of this thesis was the development of a quantitative test for polyethylene in paper. A review of the literature discloses that no published method exists; however valuable information was obtained, which directed the planning of the experimental work. A method was developed for the determination of polyethylene in paper. This method involves the extraction of paper in a Soxhlet extractor with CCl_4 . The extract is evaporated to near dryness. The residue, containing polyethylene and waxes, is then extracted with diethyl ether to remove the waxes. The results of this method show that it is accurate on high molecular weight polyethylenes; however its accuracy declines with lower molecular weights.

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Literature Survey

Introduction

In recent years the use of polyethylene in the paper industry has been growing tremendously. The combination of a plastic material and paper improves the usefulness of both products.

Polyethylene by itself is a very versatile chemical. It can be molded, extruded, casted or calendered to form a great variety of objects. It can also be applied to other materials by these means.

Polyethylene is manufactured(1) by addition polymerization of ethylene. The polymerization is carried out under a pressure of 100 to 200 atmospheres and a temperature of over 300 degrees centigrade. Oxygen or a peroxide is used as a catalyst in this reaction.

The discovery (1) of a polymer of ethylene came about in England in 1933. The first polymerization of ethylene into polyethylene was carried out in a small autoclave. A continuous operation was perfected in an English laboratory in 1937. In 1938 a pilot plant was put into production in England. Full scale production began in 1939 and four years later in the United States.

Chemical Properties

The basic chemical structure of polyethylene is: $\begin{matrix} \text{H} & \text{H} \\ | & | \\ \text{-C-} & \text{-C-} \\ | & | \\ \text{H} & \text{H} \end{matrix} \text{.}_n$

Analysis of polyethylene (1) would show the amount of carbon is equal to 85.7% and hydrogen 14.3%.

A detailed study (1) of the chemical structure of polyethylene by infrared spectroscopy shows that some olefinic and some methyl groups are present in polyethylene. The total amount of unsaturation is a maximum of about one double bond per molecule. The concentration of methyl groups is much greater, being about one for each methylene unit to less than one for two hundred methylene units. Normal commercial polyethylene has approximately one methyl group for each thirty methylene units. The methyl groups lie at the end of side chains. The side chains are thought(1) to be at least four carbon atoms long.

In general, the chemical behavior(1) of polyethylene is that of a high molecular weight paraffin. Polyethylene is a thermoplastic, having a molecular weight between 3,000 to 40,000. Substantially a paraffinic structure, polyethylene is stable and inert. It is resistant to aqueous solutions of acids, bases and salts. However, it is not completely resistant to very strong acids, potassium, permanganate, hydrogen peroxide, elemental chlorine and other halogens.

Most of the chemicals which polyethylene will not resist are oxidizing agents. Oxidation of polyethylene will lead to the introduction of ketonic carboxyl groups(1) and increase the molecular weight because of cross linking between molecules.

Extensive oxidation leads to chain degradation and loss of volatile products.

Physical Properties

Polyethylene is a partially crystalline solid. Unbranched, it is almost completely crystalline. Most commercial polyethylene is 60% crystalline and 40% amorphous.

The degree of side chains has a direct relationship to the crystallinity of polyethylene as seen in Table I (1).

TABLE I

Branching CH ₂ groups per 100 methylene units	Density at 20 degrees C.	Degree of crystallinity
0 (polymethylene)	0.99	95%
1	0.96	80%
2	0.94	72%
3	0.92	60%
4	0.91	55%

The Crystal structure of polyethylene is similar to that of a solid, low molecular weight paraffin. The unit cell is orthorhombic, meaning that the molecules are in parallel planes.

The physical properties vary greatly with the molecular weight of the polymer. Table II (2) shows the general properties of polyethylene associated with various molecular weights.

TABLE II

Molecular weight of polyethylene	General property
500-1000	Grease like
4000	Wax like
7000	Soft resinous
10,000 and up	Plastic like

Polyethylene is thermoplastic, tough and flexible. It has extremely low water sensitivity and superior electrical properties. It is dimensionally stable at normal temperatures, however it is subject to distortion under light loads. Films of polyethylene exhibit a very low water vapor transmission rate.

Use of Polyethylene in the Paper Industry

Polyethylene is used in the paper industry as a pure film and also in wax formulations. Polyethylene is not used at present, internally in paper.

Hot solutions of polyethylene (2) can be spread upon a web of paper by means of coating equipment common to the paper industry. Hot solutions are generally prepared by dissolving the polymer in hot solvents such as xylene, hi-flash naphtha, toluene and other aromatic hydrocarbons. A minimum temperature of 90°C . is needed to prevent plugging of equipment. After the solution is applied, the solvent is removed by drying ovens. At a temperature of 90°C . a solution of 35% polyethylene can be obtained.

Polyethylene can be applied to paper from a hot melt. A screw type extruder supplies the first roll of a calender stack with the hot melt of polyethylene.

Polyethylene (3) is blended with paraffin and microcrystalline waxes. These modified waxes contain from 1% to 10% polyethylene. Modified waxes may also contain polyisobutylene

and resinous materials.

The polyethylene is mixed with crystalline and amorphous waxes in a hot melt. The final product is a self-supporting film, even if the wax content is very high.

Properties of Modified Waxes and Polyethylene Films.

It has been said(4) that "Polyethylene comes the closest of any plastic to producing the ideal coated paper". Polyethylene possesses excellent resistance to water and water vapor. It is non-toxic, tasteless, odorless and inert. Polyethylene is also resistant to greases.

Physical properties of polyethylene film, which make it a good paper are: resistance to creeping, non-blocking, low-temperature flexibility and heat sealability.

Economically, polyethylene is readily available and inexpensive.

Polyethylene modified wax(4) is improved over ordinary wax by having higher gloss, higher strength, greater hardness, greater creasing resistance and higher blocking temperature. Rub-off and chipping are reduced. The wax also has improved clarity and a stronger heat seal.

Determination of Polyethylene in Paper.

The molecular weights of polyethylene commercially available (2) range from 7000 to 24,000. The six most common molecular weights are: 7000, 10,000, 12,000, 18 to 19,000, 20 to 22,000 and 22 to 24,000.

Polyethylene with a molecular weight of 18,000 to 19,000 is used for 100% polyethylene coating on paper. Molecular weights of 5,000 to 12,000 are widely used to modify waxes.

One hundred percent polyethylene coatings will contain no plasticizers. Modified wax can contain paraffin, microcrystalline wax, polyethylene, resinous materials and possibly some polyisobutylene.

In the determination of polyethylene one must first find out if the material is present. By the use of polarized light (5) polyethylene can be distinguished from waxes.

By dry distillation (6) of polyethylene one may examine the gases evolved. The gas is a volatile monomer and will condense to a liquid, which is colorless, but which later changes color to yellow. The sample being distilled will remain colorless. Several other plastics give this same test, but only one of these, polyisobutylene is used in paper.

Another way which polyethylene will differ from normal paraffin is in its melting point characteristics. Pure paraffin melts quite sharply, while polyethylene melts over a wider temperature range. Melting points differ according to the molecular weights involved.

Polyisobutylene(8) is soluble in solvents such as chloroform, carbon tetrachloride, coal tar distillates and petroleum distillates. Many of these chemicals will dissolve polyethylene, but only at elevated temperatures. This could provide a method of separating polyethylene from polyisobutylene.

Polyethylene(1) is insoluble below 60 °C. in any solvent.

Waxes are soluble in hydrocarbons and chlorinated hydrocarbons below 60°C., but their solubility increases with increasing temperature. The fact that polyethylene is so insoluble below 60°C. would seem to distinguish it from other materials in paper coatings.

Because of polyethylene's inertness, a purely chemical determination would seem quite impractical. The most logical method of determining polyethylene would appear to be to take advantage of its solubility characteristics.

The best solvents for polyethylene (1) at elevated temperatures are benzene, toluene, naphtha, carbon tetrachloride and other halogenated hydrocarbons. The properties of these solvents are shown in Table III (1).

TABLE III

Solvent	Molecular Weight	Specific Gravity at 20°C.	Melting Point	Boiling Point
1) Benzene	78	0.879	5.5°C.	80.1°C.
2) Toluene	92	0.866	-95.0	110.6
3) Xylene (ortho)	106	0.879	-27.1	144.0
4) Naphtha (refined)	---	0.850	---	130-155
5) Carbon tetrachloride	153.8	1.595	-22.8	76-77

As Richards (9) has pointed out, the solubility of a high polymer is a very complex phase system. By heating polyethylene and a solvent a single phase may be reached and is stable. A precipitate will result if the temperature is lowered.

The temperature at which the precipitate is formed is called the "critical solution temperature". Each molecular weight of polyethylene has its own "critical solution temperature". The solid phase, deposited from a dilute solution (approximately 1% by weight) can be filtered from the mother liquid. However, the solid polyethylene will still contain a large amount of solvent.

Upon cooling a mixture rich in polyethylene a third phase appears, which is the solvent still containing some low molecular weight polyethylene. This low molecular weight polyethylene will finally sweat out to the surface.

The amount of solvent left in the polyethylene upon precipitation depends upon the amount of amorphous area in the polymer.

Polyethylene can be separated into various molecular weights by fractional precipitation. Table V (9) shows the relationship between temperature and molecular weights in the filtrate.

TABLE V
(precipitated from a 2.25% solution; original mol. wt. 13,900)

Temp. °C.	Wt. fraction of polyethylene in filtrate	Mol. wt. of polyethylene in filtrate	Conc. of filtrate (wt. % polyethylene)
20	0.007	1,200	0.016
51	0.034	3,800	0.076
54	0.047	4,800	0.167
58	0.094	7,700	0.220
62	0.254	13,000	0.571
65	1.000	13,900	2.250

The three main factors in dissolving polyethylene are 1) amount of chain branching, 2) molecular weight, 3) and temperature of solution.

The solubility of polyethylene will increase as the number of side chains increase. A large number of side chains produce a greater amount of amorphous areas and thus increase the solubility.

The higher molecular weights of polyethylene are less soluble than the low molecular weights. Certain high molecular weight polyethylenes do not disperse completely, even if boiled in a good solvent. This could be a sign(9) of cross linking. However, upon adding a small percentage of a polar material such as amyl alcohol, the polymer appears to disperse completely. Table VI (9) shows the relationship between molecular weight and solubility.

TABLE VI

Molecular weight of polyethylene	Solubility at 70°C. % by wt. (in xylene)
9,440	12.0%
11,000	10.0
13,000	2.0
16,000	0.30
31,000	0.05
40,000	0.01

Polyethylene will only dissolve in a solvent which is heated above 60°C.. Table VII (9) shows the temperature at

which a 5% solution of polyethylene can be made with various solvents.

TABLE VII

Solvent	Temperature (mol. wt. 9,400)	Temperature (mol. wt. 14,000)
N Heptane	69	77
Paraffin wax	86	97
Benzene	64	76
Toluene	61	--
Xylene	64	76
Carbon tetrachloride	57	--
Petroleum ether	73	77

A suggested method(10), currently being used, for finding the percent of polyethylene in modified waxes, consists of dissolving the wax in hot (above 60°C.) carbon tetrachloride. Upon cooling the solution the polyethylene will precipitate out, leaving the wax still in solution. The polyethylene is then dried and weighed. This method is unpublished.

No direct reference has been found in the literature as to a quantitative method of determining polyethylene.

Finish

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Plan of Experimental Work

Object

The object of my experimental work is to devise a method for quantitative determination of polyethylene in paper.

General Discussion of Procedure

A method of chemical quantitative analysis would be impractical due to the fact, that polyethylene is a chemically inert material. Therefore I will take advantage of polyethylene's unusual solubility characteristics. Polyethylene is insoluble in a cold solvent and soluble in a hot solvent (above 60°C.). This fact will enable me to separate polyethylene from other substances found in paper. Generally speaking all materials found compounded with polyethylene are soluble in hydrocarbons below 60°C. Upon cooling a solution of polyethylene and foreign materials, the polyethylene will precipitate quantitatively, while the other materials will stay in solution. This will be my general method of analysis.

Details of Procedure

The first step in the procedure will be to weigh out a sample containing about .5 gram of polyethylene. A sample of paper should be at least 5 grams.

The apparatus which will be the most satisfactory for

extraction will be a Soxhlet extractor.

The time required for complete extraction of polyethylene would appear to be at least 8 hours. If the polyethylene is not all extracted at the end of 8 hours, then the time will be increased.

The solvents that will be tried are 1)carbon tetrachloride, 2)toluene, 3)xylylene. The reason for choosing these three solvents is their differences in boiling points. Carbon tetrachloride has the lowest boiling point of the three solvents ($76^{\circ}\text{C}.$), while toluene is intermediate ($110.6^{\circ}\text{C}.$), and xylylene has the highest boiling point ($144^{\circ}\text{C}.$). By using the different solvents I will be able to tell the effect of temperature on extraction. The time it takes to extract could vary with the various solvents. After extraction the solution should be allowed to stand for 8 hours before separation.

Three methods of separating a precipitated polyethylene from a cooled solution exist. The first is by filtration through a tared sintered glass crucible or a tared filter paper. The second method would be by using a centrifuge and the final method is by decantation. All three of these methods will be tried and the quickest and most accurate will be selected.

The precipitated polyethylene should be washed twice with a warm solvent to remove all foreign materials.

The separated precipitate must be dried to remove any solvent left in the polyethylene.

Experimental Work

The time available for experimental work is five hours a week for seventeen weeks or a total of 85 hours. During this time I will perform experimental work and prepare data for a final report.

The first experiment will be the separation of a known quantity of polyethylene from a prepared formulation of polyethylene and paraffin. The formulation will contain 5% polyethylene. Two different samples of polyethylene will be used. One with a molecular weight of 20,000 and another with a low molecular weight of 2000. I shall try all three solvents on these two samples. If I can recover all the polyethylene present in these samples, I will then turn my attention to polyethylene on paper.

The samples of paper will be treated with the solvent which gave the best results in the above tests. I shall also have selected a method of separation. I will run a waxed paper without polyethylene in it as a blank.

The following is a outline of the experimental work I plan to do. All trials will be run in duplicate.

Part I. Total of 30 extractions. Time-48 hours.

- 6-10 g. samples - 100 % paraffin
- 6-10 g. samples - 95% paraffin
5% polyethylene, mol. wt. 2000
- 6-10 g. samples - 95% paraffin
5% polyethylene, mol. wt. 21,000
- 6-10 g. samples - 95% paraffin
5% polyethylene, mol. wt. 11-12,000
- 6-10 g. samples - 85% paraffin
15% polyethylene, mol. wt. 21,000

Instructions

1. Run all three solvents on all samples.
2. Try the three methods of separation.
3. Dry all precipitates at 105°C.

Part II. Total of ten extractions. Time-28 hours.

- 2-samples - base stock for waxed paper.
- 2-samples - paper coated with paraffin.
- 2-samples - paper coated with:
 - 1) paraffin
 - 2) microcrystalline
 - 3) 2-3% polyethyleneMol. wts.-12,000 & 20,000
- 2-samples - paper coated with:
 - 1) paraffin
 - 2) microcrystalline
 - 3) 5% polyethyleneMol. wts.-12,000 & 20,000
- 2-samples - paper coated with:
 - 1) paraffin
 - 2) microcrystalline
 - 3) 9% polyethyleneMol. wts.-12,000 & 20,000

Instructions.

1. Use the solvent found most satisfactory from part I.
2. Use the method of separation found most satisfactory from part I.

Finish

PROCEDURES AND RESULTS OF EXPERIMENTAL WORK

The purpose of this experimental work was the development of a quantitative test for polyethylene as found in paper. During the initial stages of experimentation the plan for experimental work was followed; however a new line of investigation presented itself midway in the work.

Basic Procedures of Experimentation

The initial areas of investigation consisted of selection of the following: (1) Equipment for extraction of polyethylene from paper, (2) A suitable solvent for the extraction, (3) A method of separation of polyethylene from other materials extracted.

The Soxhlet extractor was selected as the apparatus for extraction of polyethylene. As can be seen in Table I, the time for extraction of paraffin was thirty minutes in a Soxhlet. When polyethylene pellets were extracted in a Soxhlet, the time ran from one hour for a 2000 mol.wt. to three hours for 21,000 mol.wt. A battery of four Soxhlets were used for all further extractions.

The solvents used for extraction of polyethylene from paper must be a hydrocarbon or a chlorinated hydrocarbon. Three solvents were investigated as follows; (1) carbon tetrachloride, (2) toluene, (3) ortho xylene. All three solvents dissolved polyethylene and paraffin equally well. The solvent finally selected for use throughout further experiments was carbon tetrachloride(CCl_4). This solvent has

the advantage of being economical as well as non-inflammable. The quantity of solvent that is sufficient for extraction in the Soxhlet extractor is 125 ml.

Following the selection of a solvent, some experiments were run with the purpose of separation of precipitated polyethylene from soluble extracts. Pellets of polyethylene and paraffin were dissolved in CCl_4 . Two separate series of extractions were carried out, which consisted of (1) extraction of a 95% paraffin, 5% polyethylene 21,000 mol.wt. mixture and (2) extraction of a 95% paraffin, 5% polyethylene 2000 mol.wt. mixture.

The extract was placed in a constant temperature water bath at 40°C . This was done in order that the paraffin would stay in solution. After waiting for a period of twenty four hours the polyethylene precipitated to the surface of the solution. As might be expected the 21,000 mol.wt. polyethylene gave a better separation after standing, than the 2,000 mol.wt., which had a cloudy appearance up precipitation. Two methods of separating the polyethylene from the solvent were tried. Both filtration and separation by means of a centrifuge failed to give quantitative results.

Ether Extraction Method

A new approach to the problem of separation of polyethylene from waxes was introduced. A known weight of polyethylene and paraffin was dissolved in carbon tetrachloride.

The solution was evaporated to near dryness, leaving a residue on the bottom of the tared beaker. This residue was extracted with three successive 75 ml. volumes of diethyl ether. The residue left after ether extraction was dried and reweighed. The amount of material left after extraction was quantitatively the same as the amount of polyethylene originally introduced.

Known quantities of 2,000, 6,000, 12,000 and 21,000 mol. wt. polyethylenes and paraffin were run through the same process. The results of this method can be seen in Table II. The lower molecular weight polyethylene did not give as good results as those of high molecular weight.

Attention was now turned to actual paper samples, containing approximately known percentages of polyethylene. The method used on the papers is as follows: Four grams of paper was extracted with 125 ml CCl_4 in a Soxhlet extractor for six hours. The extract, while still hot, was transferred to a tared 250 ml. beaker. The extract was evaporated almost to dryness. The residue was extracted with 3-75 ml. portions of boiling diethyl ether in a hot water bath. Each time the supernatant ether extracts were drawn off by a suction pipet. The tared beaker was placed in a 105°C . drying oven for one hour and weighed. The weight by difference so found was taken to represent the polyethylene in the samples. The results of this work may be found in Table III.

Discussion of Results

In the case of the ether extraction method, run on known percentages of polyethylene, the lower mol.wt. polyethylenes were somewhat soluble in the ether. This is probably due to the fact that low mol. wt. polyethylene and paraffin are very similar chemically.

When the higher molecular weights were extracted the results were slightly high. The cause of high results could be due to some paraffin being surrounded by polyethylene. Thus the ether could not penetrate the polyethylene and dissolve the paraffin.

During the determination on commercial papers, high results were also obtained. This might be accounted for by materials other than polyethylene which were soluble in CCl_4 and not in ether. Also the paraffin could have been "covered" by polyethylene in this case as well.

Conclusion

From observations and data obtained, the following is a suggested method for determination of polyethylene in paper.

Scope

1. This method of analysis covers the procedure for determining quantitatively the amount of polyethylene in polyethylene coated paper and polyethylene, paraffin and microcrystalline blends coated on paper.

Apparatus

2. The apparatus shall consist of the following:
 - (a) Extractor-Soxhlet extraction apparatus.
 - (b) One 250 ml beaker.
 - (c) One suction filter.

Reagents

3. (a) Carbon Tetrachloride, c.p.
 - (b) Diethyl ether.

Sampling

4. The paper shall be sampled in accordance with TAPPI standard T 400-m-49.

Test Specimen

5. The test specimen shall be cut from the sample in such a way as to be thoroughly representative. It shall consist of not less than 4 g. of the paper in the as-received condition.

Procedure

6. (a) Place the test specimen in the Soxhlet extractor. The strips of paper should be below the surface of the carbon tetrachloride when the siphon cup is filled. A filter cup should be used in the extractor to collect any solid material or fiber coming off from the paper. Extract the specimen until all the coating is removed. The extraction generally requires at least four hours. The amount of carbon tetrachloride used should be at least 125 ml.

(b) Transfer the hot extract (above 65°C.) into a tared 250 ml beaker. Evaporate the extract on a hot plate to near dryness. Place the residue, which is in a beaker, in a hot water bath at 40°C. Extract the residue with 3-75 ml. of boiling diethyl ether. Each extraction shall take at least ten minutes. After each extraction let the solid residue settle and pipet off the supernatant ether extract. The residue left after the ether extractions shall be evaporated to dryness in a hot water bath and dried in an oven at 105°C. for one hour and weighed. The weight thus found shall be taken to represent the polyethylene in the original specimen.

Note- This procedure is based on the assumption that all carbon tetrachloride extracts are soluble in diethyl ether, except polyethylene. With lower molecular weight polyethylenes this assumption may not be true.

Report

7. The amount of polyethylene shall be reported as a percentage of the original weight of the paper coating.

Recommendations

It is recommended that any further investigation upon this method of determination should include the selection of a solvent, which is capable of a greater sensitivity between low molecular weight polyethylene and paraffin.

Finish

APPENDIX

Table I

Extraction of Paraffin with Various Solvents

Sample No.	Wt of Sample	Solvent	Time to Dissolve	Ml of Solvent	Precipitate Observed
1.	10.5739 g	CCl_4	30 min.	125 ml	None
2.	10.4802	CCl_4	"	"	"
3.	10.0085	Toluene	"	"	"
4.	10.3205	Toluene	"	"	"
5.	10.1223	Xylene	"	"	"
6.	10.1802	Xylene	"	"	"

Table II

Extractions with Diethylether on Polyethylene-Paraffin Blends

Wt of Paraffin	Wt of PE*	Mol.Wt.of PE	Wt of Non-extractables	%PE Present	%of Non-extractables
0.9778g	---	---	0.0005g	---	0.06
---	.8442g	2,000	0.8388	100.0	99.34
---	.6163	21,000	0.6151	100.0	99.81
0.4200	.2544	2,000	0.1896	37.8	28.2
0.3072	.2150	6,000	0.2050	41.3	39.2
0.3012	.1690	12,000	0.1760	35.9	37.3
0.5028	.5345	21,000	0.5494	51.5	53.0

*designates polyethylene

Table III

Determination of Polyethylene in Commercial Paper

Description of Coating on Paper	Mol.Wt.of PE	Approximate % PE in Paper	%Polyethylene in Paper by Determination
Base Stock	----	----	0.00
Paraffin & Microcrystalline	----	----	0.75
Paraffin & PE Microcrystalline	12,000 & 20,000	3%	3.27
Paraffin & PE Microcrystalline	12,000 & 20,000	9%	10.51