International Science and Technology Journal
المجلة الدولية للعلوم والتقنية

Volume 37 العدد
Part 1 المجلد

المجلة الدولية للعلوم والتقنية
International Science and Technology Journal
ISTJ

http://www.doi.org/10.62341/mbhc3990

Received	**2025/09/02**	**تم استلام الورقة العلمية في**
Accepted	**2025/09/28**	**تم قبول الورقة العلمية في**
Published	**2025/09/30**	**تم نشر الورقة العلمية في**

Chemical Analysis and Immersion Testing of a Hydrocarbon Film (Benzene) After Deposition on Glass Slides Using Plasma-Enhanced Chemical Vapor Deposition (PECVD)

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Abstract:

Thin films deposited on glass surfaces can be produced by various methods, including physical methods (thermal evaporation in vacuum, sputtering, etc.) and chemical methods (chemical deposition, chemical vapor deposition, thermal chemical analysis, etc.). In this paper, thin films were deposited on glass slides using the plasma-enhanced chemical vapor deposition (PECVD) technique at pressures (10,30 and 50 mTorr) for varying durations of (5,10 and 15 minutes). In addition, the results indicates that increasing the deposition time significantly influenced the film thickness while the adhesion does not directly depend on time.

Keywords: Chemical Analysis, Immersion Testing, Hydrocarbon Film, Glass Slides.

http://www.doi.org/10.62341/mbhc3990

التحليل الكيميائي وإختبار الغمر لفيلم من مادة هيدروكربونية(بنزين) بعد ترسيبها على شرائح زجاجية بإستخدام التبخير الكيميائي المطعم بالبلازما (PECVD)

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1. الملخص:

الافلام الرقيقة التي ترسب على سطح زجاجي يمكن ترسيبها بطرق مختلفة ومنها طرق فيزيائية (التبخير الحراري في الفراغ، الترذيذ.)، وطرق كيميائية (الترسيب الكيميائي، الترسيب الكيميائي البخاري، التحليل الكيميائي الحراري.) وفي هذه الورقة تم ترسيب أفلام رقيقة على شرائح زجاجية باستخدام تقنية الترسيب البخاري الكيميائي المدعم بالبلازما المتولدة بواسطة موجات الميكروويف PECVD عند ضغوط (10،30 و50 ملي تور) خلال أزمنة مختلفة (5، 10 و30 دقيقة). وأظهرت النتائج أن زيادة زمن الترسيب أثرت بشكل كبير على سمك الغشاء في حين أن الالتصاق لا يعتمد لا يعتمد على زمن الترسيب.

الكلمات المفتاحية: التحليل الكيميائي، اختبار الغمر، فيلم هيدروكربوني، شرائح زجاجية.

1. Introduction:

Thin film technology is one of the most important technologies contributing to current developments in scientific and technical fields such as semiconductor studies, digital computers, transistor manufacturing, detectors, and solar cells. With the advancement of science and technology, methods for preparing films, their thickness, and homogeneity have evolved. These thin films are prepared using chemical and physical methods as follows [1,2].
Firstly: Physical Methods, Including thermal evaporation in vacuum and Sputtering.
Secondly: Chemical Methods, including electrochemical deposition, Chemical vapor deposition and thermal chemical

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decomposition. Chemical Vapor Deposition (CVD), the chemical vapor deposition is defined as the deposition of a solid material on a hot surface from a chemical reaction in the vapor phase. It belongs to the category of vapor transport processes that are atomic in nature, meaning that the deposited species are atoms or molecules or a mixture of both [3,4]. Plasma-Enhanced Chemical Vapor Deposition (PECVD), the thin films produced by PECVD has properties suitable for a range of applications. In the field of surface engineering for structural metals such as steel, zinc, or aluminum, protective coatings against corrosion, along with aesthetic properties, are of utmost importance. Recent research has demonstrated that metals can be effectively protected against corrosion by depositing thin films using plasma [5].

There are several types of PECVD processes based on plasma generation methods, with the most common being microwave-generated plasma at a frequency of 2.45 GHz, and radio frequency (RF) at a typical frequency of 13.56 MHz [6].

Microwave Plasma-Enhanced Chemical Vapor Deposition (MPECVD) has numerous advantages compared to other traditional polymerization processes [7].

2. Practical Aspect:

2.1 MPECVD Experiment:

Sample Preparation:

Samples were prepared from glass slides with dimensions (25 mm x 85 mm x 1 mm) for analysis. The samples were cleaned and polished with sandpaper (200, 400, 600) and then cleaned with acetone. A vacuum chamber was prepared to deposit a series of thin films on the glass slides.

2.2 Description of the Deposition System:

The local deposition system designed and built in the plasma research laboratories consists of a cylindrical chamber made of stainless steel, 25 cm long and 14.5 cm in diameter, centered on the horizontal axis as shown in figure (1). This chamber is equipped with four ports: the first connected to a commercial magnetron (microwave oven) operating at a frequency of 2.45 GHz with a power output of 540 watts. The second port is connected to a vacuum system consisting of a turbine pump and a mechanical pump to achieve a base pressure of approximately 0.1 mTorr, which is adequate for successful deposition results. Benzene vapor is introduced through the third port, regulated by a needle valve. The

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fourth port is for observing the plasma emission spectrum during the deposition process through a quartz glass window attached to the port.

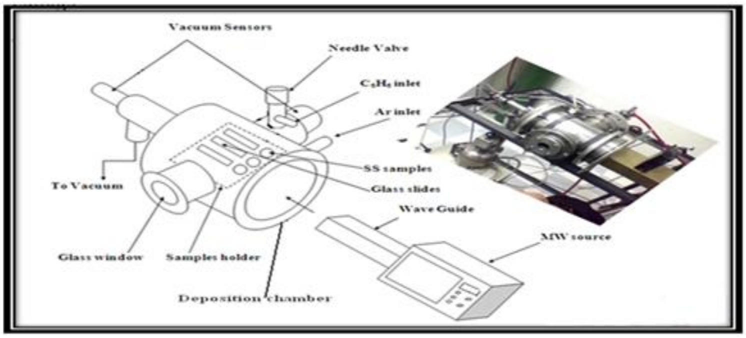

Figure 1: A schematic diagram of the plasma-enhanced chemical vapor deposition system [8].

2.3 Working Steps:

After evacuating the chamber with a mechanical pump connected to a turbine pump and placing the samples inside the chamber, the microwave device is activated, setting the operational power at 60% of the total microwave capacity (540 watts), and the material to be deposited (benzene vapor) is introduced. The microwave radiation ionizes the gas molecules into positive ions and negative electrons, leading to repeated collisions between electrons and ions with other atoms [8,9]. This process generates a large number of electrons and positive ions, resulting in plasma that heats and vaporizes the deposition material, which is then deposited as a polymer on the slide surface.

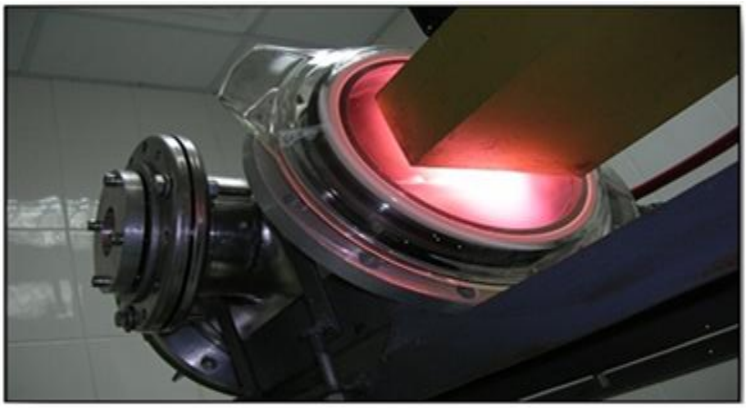

Figure (2): Image of plasma inside the chamber during the deposition process.

Benzene (C_6H_6) was chosen among several organic hydrocarbon compounds for the deposition process due to its low boiling point (80°C), requiring no heating unlike toluene (C_7H_8) or xylene (C_8H_{10}), and it does not require a long time to achieve the base pressure.

Table (1): Properties of some carbohydrate materials in terms of evaporation temperature and vapor pressure.

Compound	Vapor Pressure (kPa)	Boiling Point (°C)
Toluene (C_7H_8)	2.8 at 20°C	111
Xylene (C_8H_{10})	1.2 at 20°C	138.58
Benzene (C_6H_6)	13.33 at 25°C	80.1
Butane (C_4H_{10})	10°C at 170	-1 to 1°C

2.4 Deposition Variables:

Benzene was deposited on glass slide samples under varying deposition times and working pressures.

Table (2): Deposition process variables.

Variables	Variable Values		
Deposition Time (minutes)	15	10	5
Operating Pressure (mtorr)	50	30	10

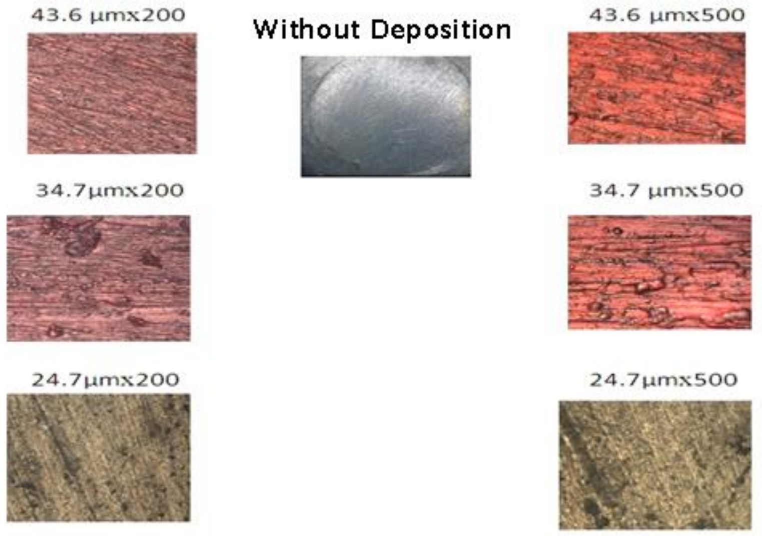

Figure (3): Some images of the film material under a light microscope at magnifications of 200 to 500 times.

2.5 Chemical Analysis of the Film After Deposition:

After depositing the film (C_6H_6) and transforming it from a liquid state to a polymer on the glass slide due to plasma influence as in table 3, the film thickness was measured using optical interference experiments. Spectral analysis was conducted using Fourier Transform Infrared Spectroscopy (FTIR) to identify the chemical structure of the film, revealing functional groups: C-H, C=C, and O-H.

2.6 Effects of Environmental Factors on the Deposited Thin Films:

2.6.1 Immersion Testing:

Table (3) shows the deposition variables before immersion in seawater solution for the deposited film on samples.

Table (3): Deposition variables before the immersion process.

Samples	Operating Pressure (mTorr)	Deposition Time (min)	Sample Thickness (μm)	Surface Characteristics
B1	10	5	14.5	Rough, sanded with 200 grit
B2	30	10	24.7	Smooth, 100% pure gasoline
B3	30	10	37.7	Smooth, 30% Ar, 70% gasoline
B4	50	15	43.6	Smooth

Figure (4): Images of the samples at 500X magnification, before the immersion process, according to the symbols as shown in the table 3.

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The samples were placed in a sealed flask with seawater solution for a month. After the first ten days, samples B2, B3, and B4 lost their film, while sample B1 retained its film. Sample B1 was re-immersed for another month without affecting the film layer, although its color changed due to interaction with seawater.

2.6.2 Immersion Testing for Adhesion Study:

In a previous study on benzene deposition on stainless steel slides, adhesion was studied using an optical tensiometer by calculating the contact angle [10,11]. After placing drops of distilled water on the film. It was observed that adhesion strength increased with deposition time while maintaining a working pressure of 50 mTorr, using Young’s equation.

3. Results and Discussion:

3.1 Chemical Analysis Results of the Film:

Figure (5) illustrates the spectra of the glass slide without deposition while figure (6) illustrates the spectrum of benzene used for deposition were compared.

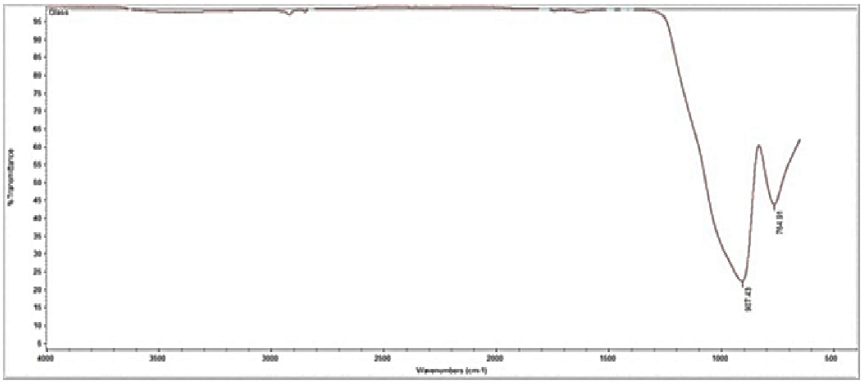

Figure (5): Spectrum of the glass slide without deposition

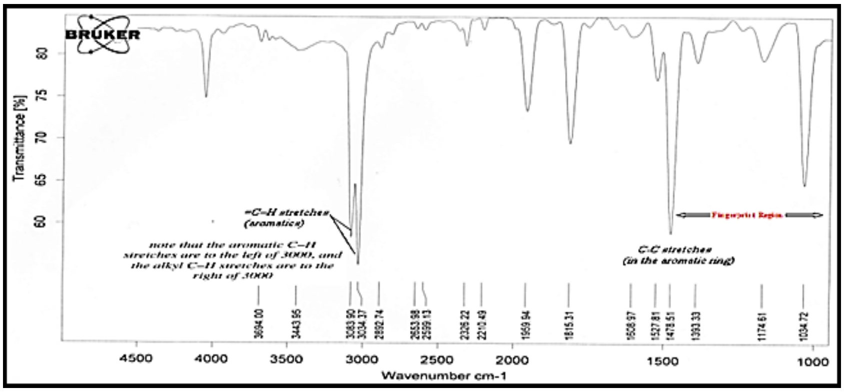

Figure (6): Spectrum of the benzene used in the deposition process.

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From figure (7), At a working pressure of 50 mTorr and a deposition time of 5 minutes, the presence of C-H, O-H, and C=C compounds was noted. C=C showed a higher absorption rate than O-H and C-H, indicating that benzene had decomposed into these compounds and transformed into a polymer.

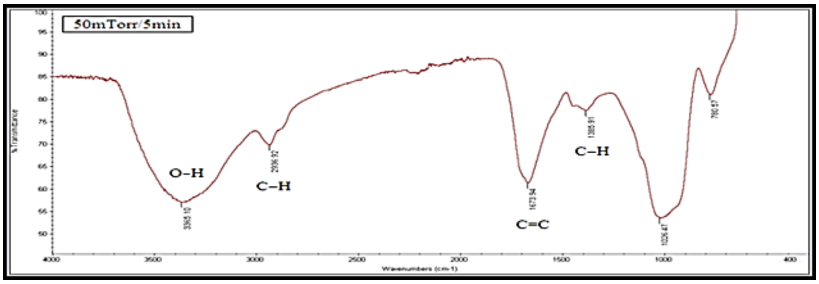

Figure (7): Spectrum of the benzene used in the deposition process at 50 milli Torr and a deposition time of 5 minutes.

3.2 Impact of Deposition Variables on the Film:

3.2.1 Impact of Working Pressure at 30 min Deposition Time:

Increasing the working pressure showed significant changes in the film's compounds. At pressures of 10 and 30 mTorr, the triple bond functional group C≡C appeared at wavenumbers 2177 cm⁻¹ and 2184 cm⁻¹ as in figures (8) and (9), while it disappeared at 50 mTorr, where the C-H functional group appeared at 2945 cm⁻¹ as in figure (10), absent at 10 and 30 mTorr due to insufficient benzene vapor in the chamber.

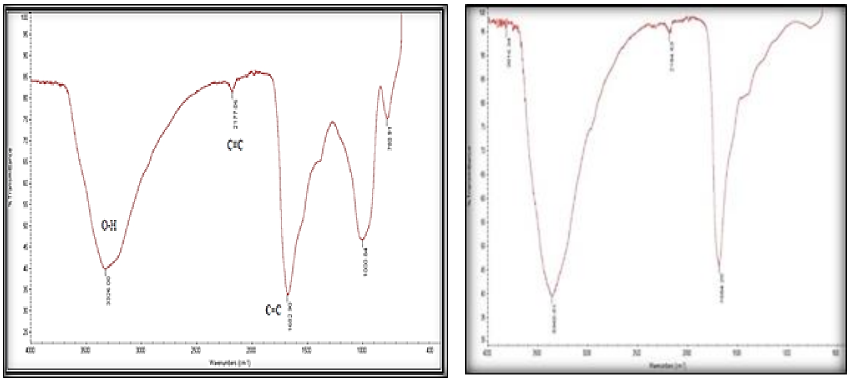

Figure (8): Spectrum of the deposited film at 30 milliTorr.

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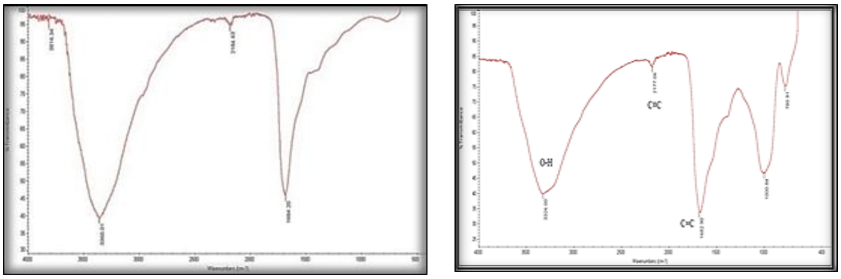

Figure (9): Spectrum of the deposited film at 10 milliTorr.

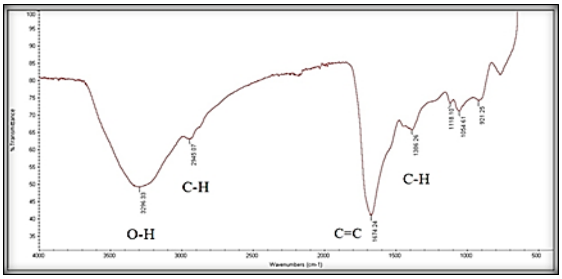

Figure (10): Spectrum of the deposited film at 50 milliTorr.

3.2.2 Impact of Deposition Time, Working Pressure, and Argon on Deposition Rate:

Table (4) illustrates the effects of deposition time, working pressure, and argon gas with benzene on the deposition rate of the film.

Table (4) shows the thickness of the deposited film and the deposition rate

	Deposition Rate s / Å	**Thickness µs m**	**Deposition Time Min**	**Operating Pressure mTorr**	**Sample**
	483.3	14.5	5	10	B1
pure 100% benzene	411.6	24.7	10	30	B2
70%benzene, Ar 30%	628.3	37.7	10	30	B3
	484.4	43.6	15	50	B4

In sample B4 at 15 min and 50 mTorr, and sample B1 at 5 min and 10 mTorr, the deposition rates were very similar despite differences in film thickness. However, with argon (Ar) as shown in sample B3, the deposition rate increased by 52% compared to

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sample B2 under the same conditions, confirming that argon enhances the deposition rate.

Summary of the Chemical Analysis Results of the Film Material:

- Compound H-C: Present at a working pressure of 50 mTorr, while it disappears at a pressure of 30-10 mTorr due to insufficient pumping of benzene into the deposition chamber.
- Compound C ≡ C: Disappears at a pressure of 50 mTorr and is present at a working pressure of 10-30 mTorr.
- Compounds C=C and O-H: Present at all pressures used.
- Film Thickness: Increases with longer deposition time.

-Increased Deposition Rate: A 30% increase was observed when argon gas was pumped with 70% benzene in the deposition chamber under the same pressure and deposition time conditions, as in sample B3.

3.3 Results of the Immersion Test:

Sample B1: Which underwent sanding with 200 grit paper, did not lose the deposited layer (film). This indicates that the sanding process is an important factor in improving the adhesion strength between the film and the sample surface.

Color Change in Sample B1: When comparing the image of sample B1 before figure (11a) and after immersion Figure (11b) it was observed that the sample changed color due to the reaction between the film material and seawater.

Adhesion: In this test, adhesion does not directly depend on time, as it does between the film material (benzene) and the stainless steel sample surface, but rather on the sanding process of the slide surface.

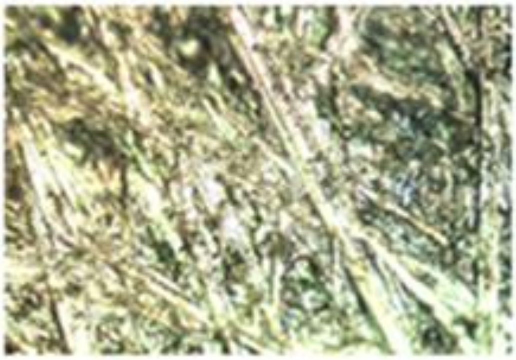

Figure (11a): Sample B1 before the immersion process.

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Figure (11b): Sample B1 after the immersion process.

Discussion:

Benzene C6H6: Is easy to handle in the deposition process because its boiling point is around 80.1°C, and its vapor pressure is 25°C at 13.33 kPa, meaning it does not require high heating. In contrast, toluene and xylene have higher boiling points and require more heating. The base pressure for plasma generation is about 0.01 Torr, while the evacuation pressure when using benzene is less than 0.1 Torr.Analysis of the Film Material (Benzene): Using FTIR, it was concluded that the operating pressure has a significant effect on the compounds of the film material, C-H, C=C, and O-H. These compounds were present at a working pressure of 50 mTorr, while the C-H (hydrogenated-carbon) compound did not appear at operating pressures of 10-30 mTorr due to insufficient pumping of benzene 30-Minute Deposition Time: At a working pressure of 10 mTorr, the compound C≡C was observed at the absorption wavelength region (2177 cm-1).

Increased Deposition Rate: When argon gas was pumped as a co-agent with the film material, the deposition rate of sample B3 increased compared to sample B2, with an increase of about 34% under the same conditions of time and pressure. Film Thickness: Increases with longer deposition time, as seen in samples B1 and B4, with close deposition rates. Adhesion After Sanding: The adhesion between the film and the slide surface in sample B1 with a thickness of 14.5 µm was better than in samples with a smooth surface.

Conclusion:

Benzene is a manageable material for deposition. Incorporating argon at a ratio of 30% with 70% benzene in the deposition chamber

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increases the deposition rate. Sanding the sample before deposition is crucial for achieving strong adhesion between the film and the sample surface.

The FTIR analysis confirmed that benzene (C_6H_6), after transitioning from liquid to solid (polymer), had indeed decomposed into C-H, C=C, and O-H compounds.

The method used for measuring film thickness was practical and yielded good results. Immersion testing indicated that sanding is a key factor for deposition, showing that adhesion between the film and the sample surface does not solely depend on deposition time, as previously observed with benzene on stainless steel.

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