Online ISSN: 2249-4626
Print ISSN: 0975-5896

Global Journal

of Science Frontier Research: B

Chemistry

Asphalt Chemistry

Photocatalytic Activity of Phenol

Highlights

The Mechanism of Oxidation

Periodate Ion in Acidic Medium

Discovering Thoughts, Inventing Future

VOLUME 15 ISSUE 2 VERSION 1.0

Global Journal of Science Frontier Research: B
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Chemistry
Volume 15 Issue 2 (Ver. 1.0)

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Contents of the Issue

Global Journal of Science Frontier Research: B
Chemistry
Volume 15 Issue 2 Version 1.0 Year 2015
Type : Double Blind Peer Reviewed International Research Journal
Publisher: Global Journals Inc. (USA)
Online ISSN: 2249-4626 & Print ISSN: 0975-5896

Asphalt Chemistry

By Sharon A. Lewis, Terry Phillips, Kayla Love, Martell McKinney, Quintessa Jackson, Denzel Pugh, Evann Comeaux, Lindsay Davis, Megan Bowlin, Rajah Singh, Phoebe Carter, Nichole Newman[€], LeMarcus Lott, Ryan Johnson, Brittany Vann & Mehari Future

Langston University, United States

Abstract- The students were taught to generate oil (binder) from recycled asphalt pavement (aggregate) using a Buchi Rotavapor R-15 for submission for elemental analysis (C, N, H, O) and engineering rheology testing and microbial growth. We obtained 18 samples from Oklahoma Department of Transportation divisions and two construction companies to compare the physical and chemical properties of these samples.

GJSFR-B Classification : FOR Code: 039999

Strictly as per the compliance and regulations of :

Asphalt Chemistry

Sharon A. Lewis [α], Terry Phillips [σ], Kayla Love [ρ], Martell McKinney [ω], Quintessa Jackson [¥], Denzel Pugh [§], Evann Comeaux [χ], Lindsay Davis [ν], Megan Bowlin [Θ], Rajah Singh [ζ], Phoebe Carter [£], Nichole Newman [€], LeMarcus Lott [₣], Ryan Johnson [₴], Brittany Vann [₲] & Mehari Future [₢]

Abstract- **The students were taught to generate oil (binder) from recycled asphalt pavement (aggregate) using a BuchiRotavapor R-15 for submission for elemental analysis (C, N, H,O) and engineering rheology testing and microbial growth. We obtained 18 samples from Oklahoma Department of Transportation divisions and two construction companies to compare the physical and chemical properties of these samples.**

Subject: comparison of various samples of recycled asphalt pavement.

I. Introduction

We collected eighteen samples from 5 Oklahoma Department of Transportation divisions and 2 construction companies. The objective of this research was to generate the oil (binder) from recycled asphalt pavements (aggregate) samples using the BuchiRotavapor R15. We were able to determine the age of the sample by observing the physical properties of the sample. We sent the samples to Galbraith Laboratory for carbon, nitrogen, hydrogen, and oxygen elemental analysis. They learned to independently use the Rotavapor and the Humboldt to process samples from the beginning to final product of generating binder. I learned the protocol from mixing samples to recovering binder.

II. Problem Statement

Recycled Asphalt Pavement (RAP) contains valuable binder and therefore, RAP should be used with new millings on surface roads and not just to patch up holes. Today the oil being produced from refineries is not as rich and of as good of quality as the oil produced twenty years ago or the roads being laid down these days would not have potholes and cracks so soon.

Roads in the United States disintegrate over a shorter period due to increasing traffic. So a focus in today's society is repairing and replacing these roads which cost a significant amount of money. The government is taking available funds and stretching them to meet our restoration needs. Recycled asphalt pavement can save money for the government, create additional business opportunities from research, save energy, and conserve diminishing resources of aggregate. Over the past decades RAP usage has increased. It is the most recycled material in the US. It has been proven that mixtures with RAP can perform as well as mixtures made with virgin binder. Increasing the usage of RAP in new mixtures can reduce the amount of new material being added.

III. Background

Once all of the binder is removed from RAP, engineers are then able to perform rheology testing. Rotational Viscosity test, Performance Grading test to include the following: Dynamic Shear Rheometer, Rotational Thin Film Oven, Pressure Aging Vessel, and Bending Beam Rheometer. You can analyze binder properties from Mechanical Empirical Pavement Design Procedures (MEPDP).

Once all the binder is removed from the RAP, the original aggregate remains for sieve analysis of extracted aggregate, specific gravity of fine and coarse aggregates, Los Angeles Abrasion test, Micro Deval Abrasion test and sand equivalent.

IV. Protocol

It is the American Association of State Highway and Transportation Officials (AASHTO) designation: T 164-08, test method B. Crush RAP to generate a weight of 300 grams and put in a 1000 ml beaker. Add Trichloroethylene (TCE) to reach the 900 ml beaker level, actually 750 ml TCE. Saturate RAP with TCE by stirring with a spatula. Allow RAP to soak in the TCE for an hour while stirring randomly. To filter the fines such as small aggregate, use the Humboldt Reflux Extractor H-149S. We used 2 sieves with appropriately wetted filter paper and set inside of the Humboldt. Connect two radiator hoses to the lid on Humboldt. One end of the lid had a hose connected to a water source. The other end of the lid had a hose connected to allow flow of water into the trough. Heat the hot plate to 120°C. Pour RAP/TCE into the top funnel. Turn on water. Continue filtration until all the binder collects into the bottom of the Humboldt. All the aggregate and fines collected into the top two sieve.

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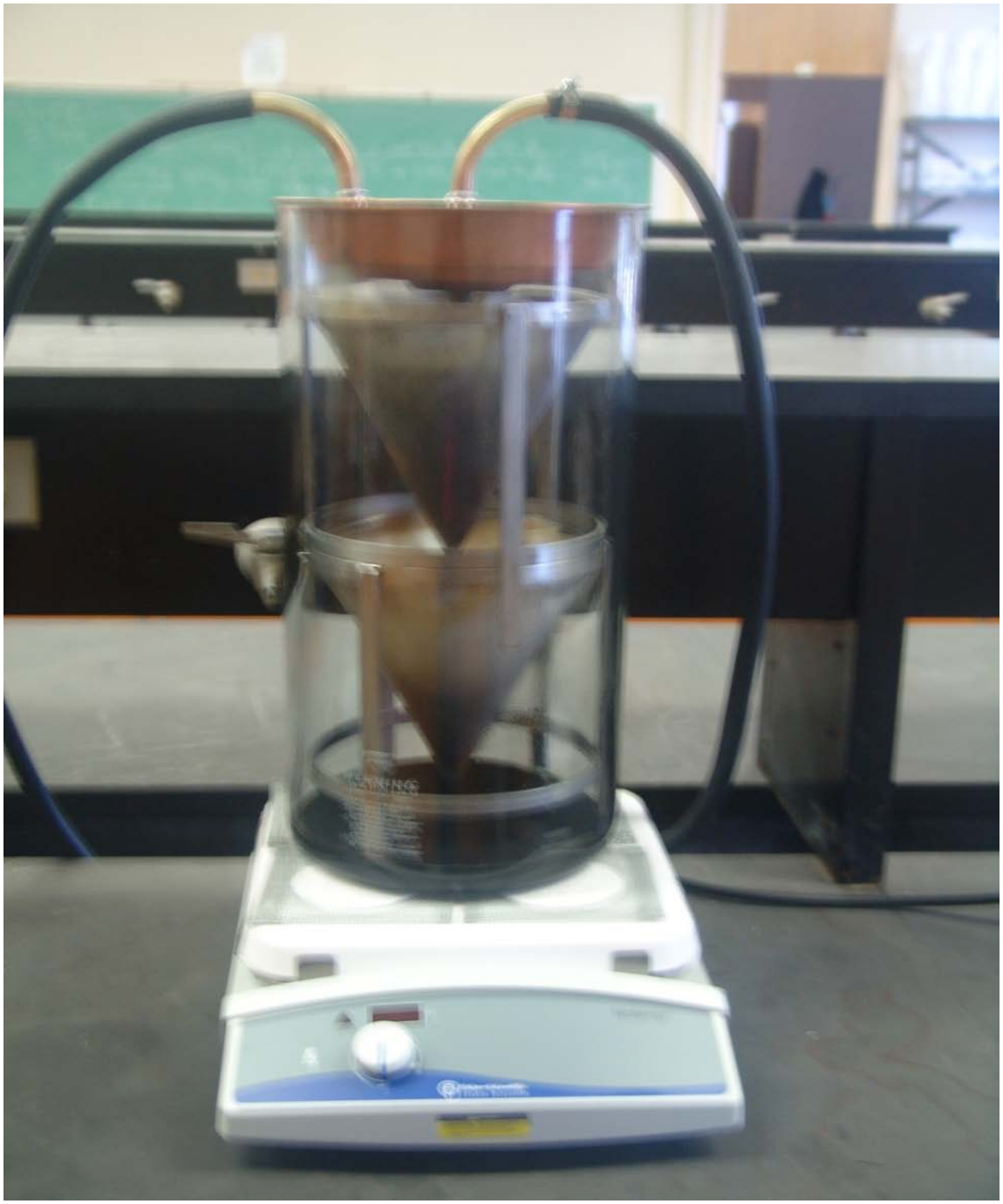
Figure#1-Humboldt



After the Humboldt activities are complete, we are no longer handling RAP/TCE. At this point, we are handling binder/TCE. Decant the binder/TCE mixture into a 2 L evaporating flask. Connect the evaporating flask to the Rotavapor. Put fresh ice into ice trap. Replace distilled water in the water bath. Turn on the pressure unit where the pressure decreases from 970 mbar to 63-67 mbar. Lower the flask into water bath and start rotating the flask at 135 rpm or higher. Every hour, depressurize the system to remove the thawed ice from the cold trap. The TCE is condensed from the binder/TCE mixture into the solvent collecting flask. When solvent stopped collecting in the collecting flask, the system is depressurized, and the flask is removed. The binder is recovered by placing the flask in an oven for ten minutes and setting it upside down over a tin can.

Figure #2-Rotavapor R15

V. Results

There are two result tables. Table #1 reflects the sources of the samples and the conditions. Where you see gaps between starting the sample and processing the sample, the Rotavapor was incapacitated. The menu on the Rotavapor recommends 33mbar pressure for TCE. However, the binder/TCE would boil and reflux into the collecting flask. After troubling shooting the protocol, we noticed that 63-37mbarr worked. Also we had a problem with the water in the water bath boiling the sample even when the temperature of the water was room temperature. Increasing the pressure solved this problem also. The instructions for the Rotavapor states that optimal conditions included having a 40 point difference between the water setting and the pressure. We came very close to that requirement after much troubleshooting.

Table#1 : Sources and Conditions in the order of work performed

	Source	Date	Pressure	Bath Temp	Results
Sample #1	Sq#1	9/26/14	67mbarr	20°C	8 grams 11/20/14
Sample #2	Hwy75 Okmulgee County	10/7/14	67mbarr	20°C	19 grams 11/19/14
Sample #3	Muskogee County	9/26/14	67mbarr	20°C	10 grams 11/19/14
Sample #4	Haskell County	9/24/14	67mbarr	20°C	11 grams 11/18/14
Sample #5	HL Norman Medium RAP	9/12/14	63mbarr	20°C	10 grams 11/14/14
Sample #6	Seq. County	10/1/14	63mbarr	20°C	8 grams

					11/14/14
Sample #7	HL East Plant Medium RAP	9/10/14	63mbarr	20°C	8 grams 11/13/14
Sample #8	Haskell Lemon 5% shingles, no RAP	9/3/14	66mbarr	23°C	5 grams 9/3/14
Sample #9	Haskell Lemon 5% shingles, no RAP	9/3/14	66mbarr	23°C	82 grams 9/3/14
Sample #10 Crust	Haskell Lemon 5% shingles, no RAP	9/3/14	66mbarr	23°C	37 grams 9/3/14
Sample #11	Division 7 (Chickashe, OK)	2/17/14	68mbarr	19°C	10 grams 4/1/14
Sample #12	No sample				
Sample #13 Binder	Haskell Lemon 25% Rap with 5% shingles	8/28/14	100mbarr	23°C	12 grams 8/28/14
Sample #14 Crust	Haskell Lemon 25% Rap with 5% shingles	8/28/14	100mbarr	23°C	47 grams 8/28/14
Sample #15	Haskell Lemon East Plant Medium RAP with Aggregate	3/31/14	68mbarr	27°C	4 grams
Sample #16	Division 5 (Hydro, OK)	8/21/14	68mbarr	19°C	16 grams 8/27/14
Sample #17	Haskell Lemon West Plant	2/18/14	67mbarr	27°C	6 grams
Sample #18	Division 4 (Quapaw)	2/17/14	60mbarr	40°C	8 grams

Table #2 reflects the elemental analysis. The procedure used for elemental analysis was GLI ME-14 for carbon, hydrogen, and nitrogen. The procedure used for oxygen was GLI E8-4. Some of the samples were special. Samples #8 & #9 are together. This was an interesting looking sample. It looked grainy, thick, and viscous before running through the Humboldt. Four hundred grams of shingles collected on the first filter paper of the Humboldt. Once removing the shingles, crust not binder was removed from the filter paper. Sample # 7 was very fluid. Sample #13 had a lot of sand but very little aggregate. All of the liquid part of the sample was put in the Humboldt. The student patiently poured the RAP/TCE trying not to pour any of the sand into the first seive of the Humboldt. All of the sample flowed through the first filter immediately. It took forever for the sample to flow through the second filter paper.

Table #2 : Elemental Analysis

	Source	%Carbon	%Hydrogen	%Nitrogen	%Oxygen
Sample#1 Binder	Sq#1	81.93	10.05	.51	3.52
Sample#2 Binder	Hwy75 Okmulgee County	83.36	10.48	.52	2.75
Sample #3 Binder	Muskogee County	9.44	.51	<.5	18.53
Sample#4 Binder	Haskell County	83.90	10.52	.50	2.51
Sample #5 Binder	HL Norman Medium RAP	79.94	10.04	.65	4.29
Sample #6 Binder	Seq. County	1	2	3	4
Sample #7 Binder	HL East Plant Medium RAP	10.74	,.5	<.5	24.87
Sample #8 Binder	Haskell Lemon 5% shingles, no RAP (5grams)	75.98	9.42	<.5	2.32
Sample #9 Binder	Haskell Lemon 5% shingles, no RAP (82 grams)	81.56	10.11	2.06	
Sample #10 Crust	Haskell Lemon 5% shingles, no RAP The weight of the crust remaining on filter paper was 37 grams of from Samples #8 & #9)				
Sample #11	Division 7	82.75	10.20	.52	2.31

Binder	(Chickashe, OK)				
Sample #12	Miss count				
Sample #13 Binder	Haskell Lemon 25% Rap with 5% shingles	84.33	10.50	<.50	2.48
Sample #14 Crust	Haskell Lemon 25% Rap with 5% shingles **The weight of the crust left on the filter paper was 47 grams.**				
Sample #15 Binder	Haskell Lemon East Plant Medium Rap with Aggregate				
Sample #16 Binder	Division 5 (Hydro, OK)	82.67	10.60	.53	2.23
Sample #17 Binder	Haskell Lemon West Plant				
Sample #18 Binder	Division 4 (Quapaw)	74.41	8.95	<0.5	2.23

VI. Conclusion

This research is beneficial to the work of engineers because they would be able to perform rheology testing: Rotational Viscosity test, Performance Grading test, Dynamic Shear Rheometer, using Rotational Thin Film Oven, Pressure Aging Vessel, and Bending Beam Rheometer. In conclusion, we used the Rotavapor to extract binder from different sources to perform carbon, hydrogen, nitrogen, and oxygen elemental analysis data.

VII. Anticipated Benefits

The anticipated benefit is to present this analysis to the eight ODOT divisions in Oklahoma so that they will realize that RAP is more valuable to them than usage as a black rock. We recommend that the ODOT divisions start using RAP at various percentages with virgin aggregate on surface roads. They must first start separating RAP sources from various projects.

VIII. Acknowledgements

The authors would like to thank Langston University, Department of Chemistry; Haskell Lemon Construction Company, specifically Jay Lemon; and several divisions of the Oklahoma Department of Transportation.

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Global Journal of Science Frontier Research: B
Chemistry
Volume 15 Issue 2 Version 1.0 Year 2015
Type : Double Blind Peer Reviewed International Research Journal
Publisher: Global Journals Inc. (USA)
Online ISSN: 2249-4626 & Print ISSN: 0975-5896

Evaluation of Pure and Ag-Doped Tio_2 Films in the Photocatalytic Activity of Phenol

By Adriana B. Araújo, Sônia M. A. Jorge, Pedro M. Padilha, Dorotéia F. Bozano, Gustavo R. Castro & Ariovaldo O. Florentino

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Abstract- Pure TiO_2 films, modified by the addition of Degussa P25 powder and Ag-doped were synthesized using sol-gel route and immobilized in borosilicate support at different withdrawing rates and dipping time. They were characterized by X-ray diffractometry, scanning electronic microscopy and high-resolution field-emission gun scanning electronic microscopy. Their photocatalytic activities under UV irradiation were examined using phenol. The higher activities were achieved through the Ag-doped films deposited with 0.4 mm s^{-1} and two dipping. Approximately 90% of the organic contaminant was photodegradated after 24 h of irradiation.

Keywords: TiO_2 films, photocatalysis, doping, phenol, photodegradation.

GJSFR-B Classification : FOR Code: 259999

Strictly as per the compliance and regulations of :

Evaluation of Pure and Ag-Doped Tio_2 Films in the Photocatalytic Activity of Phenol

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Abstract- **Pure TiO_2 films, modified by the addition of Degussa P25 powder and Ag-doped were synthesized using sol-gel route and immobilized in borosilicate support at different withdrawing rates and dipping time. They were characterized by X-ray diffractometry, scanning electronic microscopy and high-resolution field-emission gun scanning electronic microscopy. Their photocatalytic activities under UV irradiation were examined using phenol. The higher activities were achieved through the Ag-doped films deposited with 0.4 mm s^{-1} and two dipping. Approximately 90% of the organic contaminant was photodegradated after 24 h of irradiation.**

Keywords: TiO_2 films, photocatalysis, doping, phenol, photodegradation.

I. Introduction

Heterogeneous photocatalysis has received a great deal of attention as an advanced oxidation process (AOP) for degrading persistent organic compounds, reducing chemical oxygen demand (COD) and eliminating microorganisms in water bodies to allow for its reuse [1-5]. The advantage of this process is that it leads to the total mineralization of the organic contaminants, whether they are simple or complex molecules. The photocatalysis mechanism is based on the activation of a semiconductor, by sunlight or artificial light (Ultraviolet visible radiation). The interaction of light with the semiconductor excites electrons in the valence band toward the conduction band. This excitation produces holes (h^+) in the valence band that will act as oxidizing sites, while the photogenerated electrons (e^-) combine with the dissolved oxygen, preventing the recombination of the electron-hole pair. In addition, the oxygen reacts with reduction sites, the electrons of the conduction band, initially forming superoxide ion-radical ($O_2^{\cdot-}$) and later, peroxides. The latter can react with the photogenerated electron (or superoxide ion-radical) forming $^{\cdot}OH$ radicals. By this mechanism, both molecular and dissociated water (OH^- groups bound at the TiO_2 surface) react with the photogenerated h^+ holes to form highly oxidizing hydroxyl radicals [6]. The organic pollutant can thus be degraded directly at the surface of the semiconductor through the photogenerated hole, or through the hydroxyl radical, resulting in complete mineralization or in the formation of persistent intermediaries for subsequent oxidation.

Different semiconductors have been tested for photodegradation of several organic pollutants. Of all, titania has proved to be the best material because of its many desirable properties. It possesses photostability, is biologically inert, inexpensive, and displays chemical stability within a wide range of pH [7]. Although this method is quite efficient, its disadvantage is that the catalyst is suspended in the solution in the form of powder, which of course makes the separation process difficult and expensive, rendering the method unfeasible on a large scale. In order to resolve this problem, more recent studies propose the use of TiO_2 in the form of films obtained via sol-gel immobilized in inert substrate [8,9].

Although the use of films is an advance for enabling of large-scale photocatalysis, the physically adhered film can be leached during the photocatalytic process. To overcome this difficulty it is necessary to prepare films with suitable physical and chemical properties for photocatalysis and firmly adhered to the substrate.

Another important factor is the greater efficiency of the photocatalytic process. It makes the recombination of the electron-hole pair difficult, extending the occurrence time for reactions that lead to the formation of oxidizing species. This can be achieved through modifications on the semiconductor's surface and/or structure or by the addition of sensitizers that improve the system's quantic yield [10-12].

The addition of noble metals on the surface of the semiconductor can improve its photocatalytic activity [13-17]. This deposition can produce traps to withdraw photoinduced electrons or holes leading to a decrease of the electron-hole recombination in photocatalytic process and an increase in the ability of visible light absorption by TiO_2 [13]. Some noble metals such as Pt, Pd, Rh and Au are very expensive to be used in industrial scale, leading to the search for less valuable metals but which have similar performance as sensitizers. According to the literature, among the metal dopants such as Fe, Cr, Zn, Ag, etc, Ag is the most

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promising as a modifier of TiO_2 for organic pollutants photodegradation [17].

Thus, this study describes the synthesis and characterization of thin titania films prepared by the sol-gel route and modified with silver ions. These films were immobilized in borosilicate support and their photocatalytic activities under UV irradiation were analyzed using phenol as a model organic contaminant.

II. Experimental

a) Chemicals

All chemicals used in this study were of analytical grade without further purification. Titania, supplied by Degussa (P25), was predried at 150°C under vacuum for 10 h and then used immediately.

b) Preparation of the TiO_2 films

Titanium dioxide suspensions were prepared by the sol-gel method, according to the following procedure: titanium(IV) isopropoxide ($Ti(i\text{-}OPr)_4$, Aldrich Chemical, 97%) was mixed quickly with glacial acetic acid under constant stirring (molar ratio $H^+/Ti = 4$). Next, this solution was diluted with isopropyl alcohol in 1:1 Ti/alcohol ratio and the resulting solution (solution A) kept under agitation for ~1 h. Alcohol and a nitric acid solution (solution B) were then added to the mixture, still under continual stirring, keeping the molar ratios at alcohol/Ti = 7.6 and $H^+/Ti = 0.5$ [18]. Solution A and Solution B were mixed and the resulting solution (Solution C) was kept under stirring for ~2 h. The immobilized films prepared immediately after the obtainment of this sol were called pure 0 h TiO_2. When these films were kept under refrigeration for 24 h before to be immobilized they were denominated pure 24 h. It was also prepared a sol following the same procedure above described, being the Solution A the same. However, to the solution B was added TiO_2 P25 in a proportion of 1.0 g of titania per liter of ethanol and this solution was sonicated for ~20 min before to be mixed with Solution A. These films immobilized immediately after sol formation were named mixed 0 h TiO_2 and those immobilized after to be kept under refrigeration for 24 h were called mixed 24 h TiO_2. Titania films modified by silver were also prepared. For this, beyond TiO_2 P25 in a proportion above mentioned, it was added $AgNO_3$ to the Solution B to get Ag/TiO_2 films at different Ag loadings from 0.15 to 5 weight%.

The TiO_2 solution prepared (Solution C) were immobilized, by the dip-coating deposition method, in a tubular borosilicate support, whose area available for the formation of the thin film was ~ 132 cm^2. The withdrawing rate was varied as well as the dipping time. The films were dried at room temperature and then calcined at 250°C for 15 min and at 450°C for 4 h.

c) Characterization

The thin films were characterized by several techniques. X-ray diffractometry (XRD) was performed with a Rigaku diffractometer model D/Max-2100/PC automatic, equipped with a nickel monochromator using Cu K_α (λ = 1.5405 Å) radiation at 40 kV and 20 mA. The method employed was the small angle, using a scanning velocity of 2^0 min^{-1} and scans between 15^0 and 80^0. Scanning electronic microscopy (SEM) and high-resolution field-emission gun scanning electronic microscopy (FEG-SEM) were done on a Zeiss, model DSM 94A and on a Leo, model Supra 35 microscopes, respectively.

d) Photocatalytic experiments

The photocatalytic activity, using a solution containing 15.0 mg L^{-1} of phenol, was investigated. The photodegradation experiments were conducted in a cylindrical reactor in which the sample was kept at a temperature of 25°C, under stirring, saturated with oxygen, and irradiated with light at predominant wavelength of 365 nm, produced by four 15 W light bulbs. The incident irradiation, measured with a model PMA2100 version 1.16 Solar Light Company Inc. photometer/radiometer, was 5.11 mW cm^{-2}. Aliquots of 7 mL of the solution were removed at various times during the photocatalytic process and the photodegraded phenol content was evaluated by UV-VIS spectro photometry by using a spectrophotometer Thermo Spectronic model Genesys 6.

III. Results and Discussion

a) The pure 0 h TiO_2 and pure 24 h TiO_2 films

The photocatalytic activity of the pure 0 h TiO_2 and pure 24 h TiO_2 films immobilized in a support of borosilicate utilizing 0.4 mm s^{-1} withdrawing rate and two dipping was evaluated towards phenol. Under these circumstances, approximately 55% and 65% of phenol degradation after 24 h of illumination was achieved using the pure 0 h TiO_2 and pure 24 h TiO_2 films, respectively. These results may be considered satisfactory since the covered surface area of the substrate was equal to 0.0132 m^2. Without the catalyst, only 5% of the standard phenol solution was found to have become photo degraded after 24 h of illumination.

Micrographs were obtained by SEM and FEG-SEM for pure 24 h TiO_2 film with different withdrawing rates and dipping time. It was observed a decrease of particles` size, roughness and porosity of film as well as an increase of micro cracks on the surface film when the withdrawing rate increased. The photo catalytic activity of this film towards phenol also decreases with the increase of withdrawing rate in agreement with the results of film`s characterization. The increase of this parameter promotes the increase of micro cracks leading to the decrease of the amount of available catalyst for degradation of the organic contaminant [19-

21]. A decrease in the photo catalytic activity was also observed when the dipping time increased. This is probable due to higher recovering of actives sites by successive layers of titania hindering the contact of UV radiation. Another fact to be considered under this condition is the increase of rutile phase. Several studies have shown that pure anatase TiO_2 has a higher activity than pure rutile under oxidizing conditions and a discussion about this was taken by Balasubramanian *et al*. [19]. The higher photo catalytic activity was confirmed for the pure 24 h TiO_2 film deposited with 0.4 mm s^{-1} and two dipping, and its thickness, using SEM micrograph, was estimated at 140 nm.

b) The mixed 0 h TiO_2 and mixed 24 h TiO_2 films

The photo catalytic activity of the mixed 0 h TiO_2 and mixed 24 h TiO_2 films was also evaluated for the degradation of phenol and the results are presented in Figure 1. In the same Figure are shown the activities for the pure 0 h TiO_2 and pure 24 h TiO_2 films. As may be observed it was similar for pure 0 h TiO_2 and mixed 0 h TiO_2 films. On the other hand, the higher activity was found for the mixed 24 h TiO_2 film. This behavior may be attributed to the higher viscosity of the sol after aging process for 24 h. According Balasubramanian *et al*. [21], the addition of TiO_2 P25 to the sol also increase the viscosity since it leads to the particle growth during the drying step and serve as nucleation sites. Thus the particle growth occurs on the surface of the P25 particles.

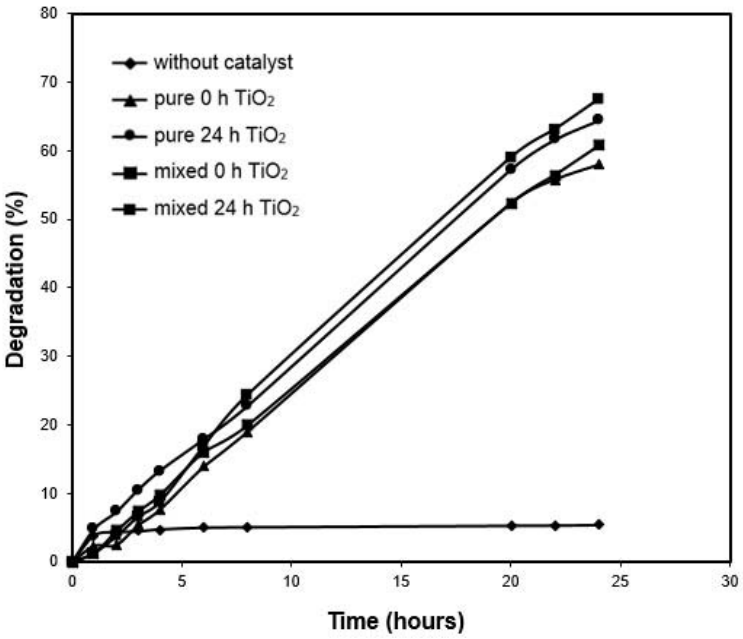

Figure 1 : Photo degradation of phenol as a function of irradiation time in the absence and presence of catalysts: pure 0 h TiO_2, pure 24 h TiO_2, mixed 0 h TiO_2 and mixed 24 h TiO_2 films under UV illumination, oxygen atmosphere and stirring (pH 5).

The thickness of the mixed 24 h TiO_2 film, estimated using SEM micrograph was 188 nm, being higher than that obtained for the pure 24 h titania film (140 nm). This result corroborates the increase in viscosity of mixed film and is consistent with published studies which show that the film thickness increases with increasing viscosity within a certain range [21,22]. Furthermore, Figure 1 shows that the phenol degradation percentage in the first 8 h of irradiation is around 25%, which increases to 68% after 24 h of irradiation. This difference is probable due to the presence of intermediates hindering the process of photo degradation at the beginning of irradiation.

Photo degradation of phenol using the mixed 24 h TiO_2 film was also evaluated at different pH values as shown in Figure 2. As may be seen, the photo catalytic activity increased with decreasing pH. This result may be attributed to the characteristic of the TiO_2 whose surface becomes positively charged with increasing acidity promoting electrostatic attraction with the phenolate anion. As a result, the adsorption of the substrate increases. Pecchi *et al*. have also observed that the percentage of pentachlorophenol adsorbed on TiO_2 P25 increases with decreasing pH [23].

c) The Ag doped TiO_2 films

The same sol utilized to prepare the mixed 24 h TiO_2 film was used to get Ag doped TiO_2 films. So, $AgNO_3$ was added to Solution B for obtaining films with the following silver content: 0.15, 0.5, 2 and 5 weight %, named as 0.15%Ag/TiO_2, 0.5%Ag/TiO_2, 2%Ag/TiO_2 and

5%Ag/TiO_2, respectively. By using all these films, the photo degradation of phenol was about 90% after 24 h of irradiation as may be observed in Figure 3. Accordingly, these results were better than those obtained with the mixed 24 h TiO_2 film.

As the photo catalytic activity was similar for all doped films, studies of characterization were carried out for the 0.15%Ag/TiO_2 film. FEG-SEM micrographs showed little particles, porous structure and increased thickness when compared to those

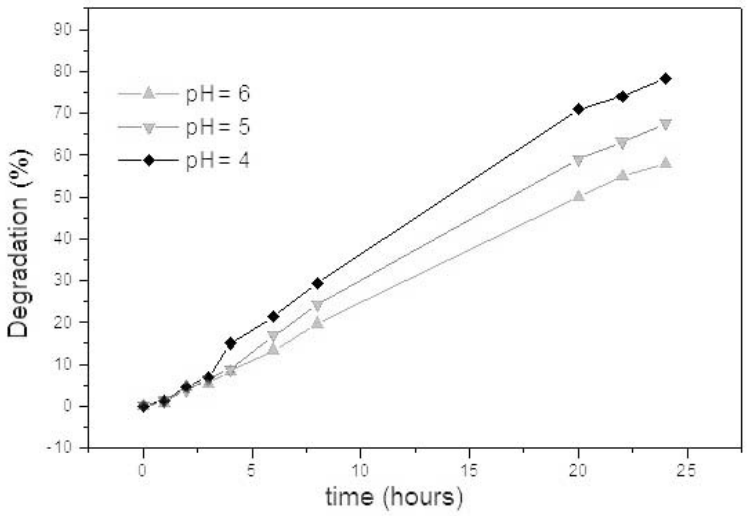

Figure 2 : Photo degradation of phenol as a function of irradiation time for mixed 24 h TiO_2 films at different pH values. (a) pH 4, (b) pH 5 and (c) pH 6.

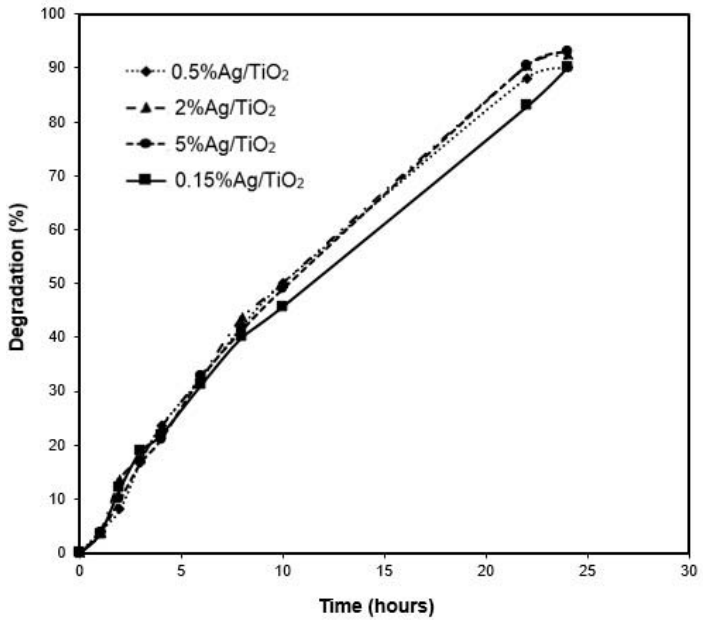

Figure 3 : Photo degradation of phenol as a function of irradiation time for Ag doped TiO_2 films at different Ag loadings

obtained for the mixed 24 h TiO_2 film (188 nm). The thickness of the 0.15%Ag/TiO_2 film was estimated at 210 nm.

The influence of Ag doping on the microstructure of the film was analyzed through X-ray diffraction and the results are presented in Figure 4. The patterns for the mixed 24 h TiO_2 and the 0.15%Ag/TiO_2 films showed the predominance of the anatase phase with a pronounced (1 0 1) peak at $2\theta = 25.3^{o}$. Other peaks of smaller intensity, at 37.7^{o}, 38.5^{o}, 48.1^{o}, 55.1^{o}, 59.3^{o}, 62.4^{o} were attributed to the same phase [9,20,24].

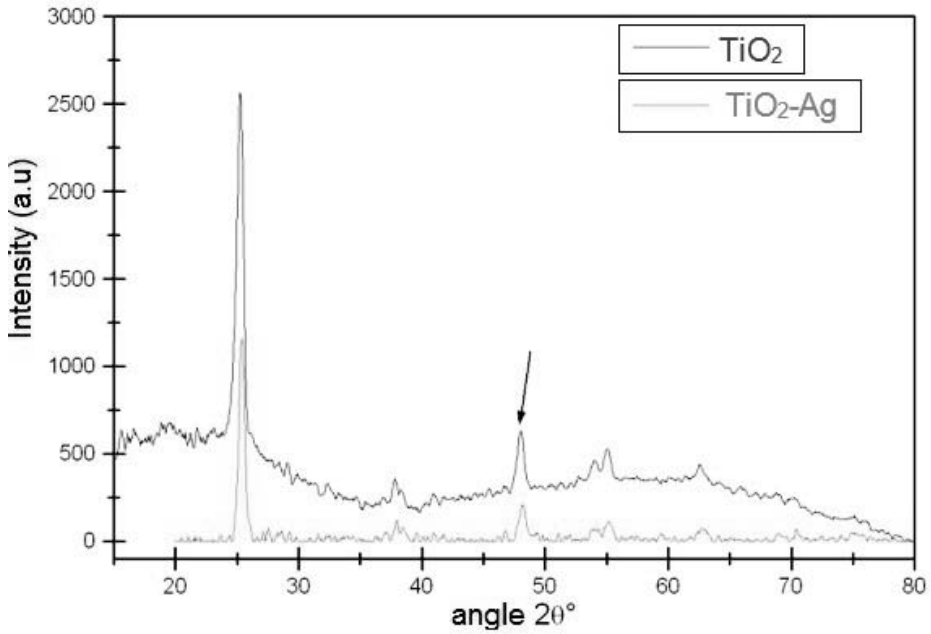

Figure 4 : XRD patterns for the mixed 24 h TiO_2 and 0.15%Ag/TiO_2 films

Crystallite size was calculated by using Scherrer´s formula [25]:

$$D = \frac{0.9\lambda}{\beta cos\theta}$$

where D is the crystallite size, λ the X-ray wavelength used, β the broadening of diffraction line measured as half its maximum intensity and θ the corresponding angle. Values of 14 nm and 12 nm were found for the mixed 24 h TiO_2 and 0.15% Ag/TiO_2 films, respectively. So, the grain size decreases with the Ag doping. Researchers [15,17] have explained this behavior as follows: the radius of Ag^+ (ca. 126 pm) is bigger than that of Ti^{4+}(ca. 68 pm), then Ag^+ ions introduced by the sol-gel process would not enter the lattice of the anatase phase to form a stable solid solution. During the calcination step these dispersed Ag^+ ions would gradually migrate from the volume of TiO_2 grains to their surfaces and further to the surface of TiO_2 film. Those Ag^+ ions then would compete with Ti^{4+} and O^{2-} ions for the diffuse and reorganization, and the anatase crystallization and grain growth is hindered. Furthermore, because the Ag^+ ions gradually moves along with the anatase grain boundaries to the surface of TiO_2 film, the energy necessary for the movement of anatase grain boundary would increase and the driving force for the anatase grain boundary migration consequently decreases.

The effect of Ag doping on the TiO_2 photo catalytic activity was also analyzed. The results, depicted in Figure 5, showed that the photo degradation of phenol using the 0.15%Ag/TiO_2 film was around 25% higher when compared to the mixed 24 h TiO_2 film. Similar behavior has been observed by other investigators [17] and according to them the increase in the photo catalytic activity using the modified silver TiO_2 film can be related to the fact that the Fermi level of TiO_2 is higher than that of silver. By this reason, electrons would transfer from film to metallic silver particles resulting in a space charge layer at the boundaries between Ag and TiO_2. The electric field drives electrons to the interior and abstracts holes to the interfacial region of TiO_2 film, which enhances the photo generated electron-hole pair separation and inhibits their recombination. The charge separation efficiency and lifetime of photo generated charge carriers then increases. Relatively larger amount of generated hole can reach the interfacial region of TiO_2 film producing higher densities of reactive radicals such as HO_2, ·OH and H_2O_2,

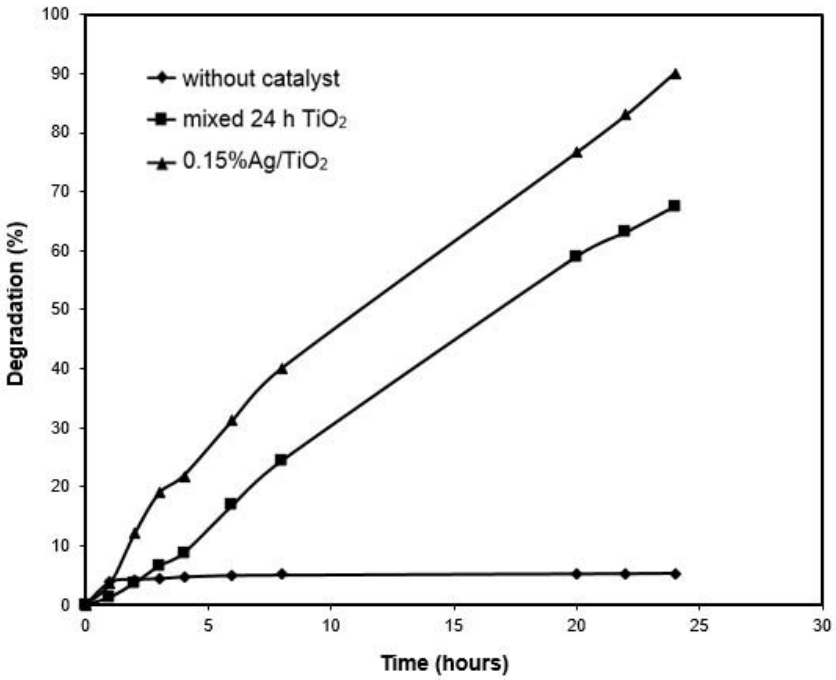

Figure 5 : Photodegradation of phenol as a function of irradiation time in the absence and presence of catalyst: mixed 24 h TiO_2 and 0.15%Ag/TiO_2 films under UV illumination, oxygen atmosphere and stirring (pH 5)

etc. For the TiO_2 photocatalytic oxidation proceeding mainly through the oxidization of photogenerated holes and its carriers, i.e. the reactive radicals, the photocatalysis is then facilitated. Besides, owing to the electrostatic interaction between the relatively positively charged TiO_2 film and negative ^{-}OH and H_2O, absorption of hydroxyl groups and water on doped TiO_2 film would be improved. This also leads to the increase in the contents of surface hydroxyl groups on doped TiO_2 films, which facilitates the charge transfer and the TiO_2 photocatalysis.

IV. Conclusions

The photocatalytic activities of the following films: pure titania, modified by addition of TiO_2 P25 and doped with Ag at different loadings and immobilized in borosilicate substrate were investigated. The results, tested for phenol degradation, showed that the most promising were the Ag-doped films deposited at 0.4 mm s^{-1} withdrawing rate and two dipping. Under these conditions, around 90% of the organic pollutant was photodegraded after 24 h of irradiation.

V. Acknowledgments

The authors gratefully acknowledge the financial support of FAPESP, CAPES and FUNDIBIO (Brazil).

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Results:

The principle of a results segment is to present and demonstrate your conclusion. Create this part a entirely objective details of the outcome, and save all understanding for the discussion.

The page length of this segment is set by the sum and types of data to be reported. Carry on to be to the point, by means of statistics and tables, if suitable, to present consequences most efficiently.You must obviously differentiate material that would usually be incorporated in a study editorial from any unprocessed data or additional appendix matter that would not be available. In fact, such matter should not be submitted at all except requested by the instructor.

Content

- Sum up your conclusion in text and demonstrate them, if suitable, with figures and tables.
- In manuscript, explain each of your consequences, point the reader to remarks that are most appropriate.
- Present a background, such as by describing the question that was addressed by creation an exacting study.
- Explain results of control experiments and comprise remarks that are not accessible in a prescribed figure or table, if appropriate.
- Examine your data, then prepare the analyzed (transformed) data in the form of a figure (graph), table, or in manuscript form.

What to stay away from

- Do not discuss or infer your outcome, report surroundings information, or try to explain anything.
- Not at all, take in raw data or intermediate calculations in a research manuscript.
- Do not present the similar data more than once.
- Manuscript should complement any figures or tables, not duplicate the identical information.
- Never confuse figures with tables - there is a difference.

Approach

- As forever, use past tense when you submit to your results, and put the whole thing in a reasonable order.
- Put figures and tables, appropriately numbered, in order at the end of the report
- If you desire, you may place your figures and tables properly within the text of your results part.

Figures and tables

- If you put figures and tables at the end of the details, make certain that they are visibly distinguished from any attach appendix materials, such as raw facts
- Despite of position, each figure must be numbered one after the other and complete with subtitle
- In spite of position, each table must be titled, numbered one after the other and complete with heading
- All figure and table must be adequately complete that it could situate on its own, divide from text

Discussion:

The Discussion is expected the trickiest segment to write and describe. A lot of papers submitted for journal are discarded based on problems with the Discussion. There is no head of state for how long a argument should be. Position your understanding of the outcome visibly to lead the reviewer through your conclusions, and then finish the paper with a summing up of the implication of the study. The purpose here is to offer an understanding of your results and hold up for all of your conclusions, using facts from your research and generally accepted information, if suitable. The implication of result should be visibly described. Infer your data in the conversation in suitable depth. This means that when you clarify an observable fact you must explain mechanisms that may account for the observation. If your results vary from your prospect, make clear why that may have happened. If your results agree, then explain the theory that the proof supported. It is never suitable to just state that the data approved with prospect, and let it drop at that.

- Make a decision if each premise is supported, discarded, or if you cannot make a conclusion with assurance. Do not just dismiss a study or part of a study as "uncertain."
- Research papers are not acknowledged if the work is imperfect. Draw what conclusions you can based upon the results that you have, and take care of the study as a finished work
- You may propose future guidelines, such as how the experiment might be personalized to accomplish a new idea.
- Give details all of your remarks as much as possible, focus on mechanisms.
- Make a decision if the tentative design sufficiently addressed the theory, and whether or not it was correctly restricted.
- Try to present substitute explanations if sensible alternatives be present.
- One research will not counter an overall question, so maintain the large picture in mind, where do you go next? The best studies unlock new avenues of study. What questions remain?
- Recommendations for detailed papers will offer supplementary suggestions.

Approach:

- When you refer to information, differentiate data generated by your own studies from available information
- Submit to work done by specific persons (including you) in past tense.
- Submit to generally acknowledged facts and main beliefs in present tense.

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Introduction	Containing all background details with clear goal and appropriate details, flow specification, no grammar and spelling mistake, well organized sentence and paragraph, reference cited	Unclear and confusing data, appropriate format, grammar and spelling errors with unorganized matter	Out of place depth and content, hazy format
Methods and Procedures	Clear and to the point with well arranged paragraph, precision and accuracy of facts and figures, well organized subheads	Difficult to comprehend with embarrassed text, too much explanation but completed	Incorrect and unorganized structure with hazy meaning
Result	Well organized, Clear and specific, Correct units with precision, correct data, well structuring of paragraph, no grammar and spelling mistake	Complete and embarrassed text, difficult to comprehend	Irregular format with wrong facts and figures
Discussion	Well organized, meaningful specification, sound conclusion, logical and concise explanation, highly structured paragraph reference cited	Wordy, unclear conclusion, spurious	Conclusion is not cited, unorganized, difficult to comprehend
References	Complete and correct format, well organized	Beside the point, Incomplete	Wrong format and structuring

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ISSN 9755896