

OK. So yeah, please. Good morning. And I'm N. Prasad from Nasa Langley. And today I'll discuss something about effects of processing parameters on sensing of chem-bio agents on surfaces. The interesting point is that some research is going on to develop new sensors, radiation sensors. And this is a very preliminary work. And after this very interesting talk on [INAUDIBLE]. This is on the other side of the spectrum of research. We are trying to explore new materials.

And this work was done at UMBC. Dr. Singh and Dr. Brian and their students did this work, and I'm presenting their work. So the overall objective is to indicate that the contact angle affects the absorption, which is a volume effect and adsorption, which is a surface effect. And this is for sensing agents.

And so the overall objective is to study the effect of contact angle on sensing agent for the detection of high energy radiations. And the secondary objective is to keep some degree of self-cleaning for reuse characteristics.

So there were approaches to use nanoparticle filled matrix which can sense the surface as well as remove the surface contaminants so that the cleaning is also an essential part of a sensor for reusing purposes.

So just going back to the materials that we would like to discuss. Here is an example of some of the basic materials like nickel oxide and nickel oxide with titania. And these things can be utilized as sensor materials and they can be re-casted.

And this work is also being presented in this conference. And, again, it only indicates that if we could-- so if we develop the materials, this could be re-casted into appropriate forms.

Coming back to our nickel oxide based technology, this work was done first with--  
- first by Professor Khalil Arshak. And the publication indicated that the possibility of using nickel oxide for various sensing purposes. And their data showed that the nickel oxide does work, but no attempt was made to understand and enhance the material parameters for device and sensor performance.

So the relevance of composite applications in our case is the significant difference in the I-

V characteristics or the resistance curves due to radiation source of a monolithic and composite based on ionic organics can be explained based on contribution of organic ionic matrix. And basically, the pair production due to ionization is the major mechanism in organic compounds. The gamma rays, the beta radiations easily break the chemical bonds because of the high energies that they carry and also prevent bond recombination.

Also the radiation gamma rays and other charged particles lead to ionization process and hence can alter the current-voltage characteristics compared to monolithic oxides. And this results in larger change in current high resistance for the composites compared to a monolithic film, and most of the film-based nickel oxide sensors involve growth of film by high temperatures and temperature process on desired substrates.

Here is our preliminary data on nickel oxide. And the left shows the resistance curves. There is electrical conductivity versus exposure change. And what it shows is the measurement of different exposure time showed feasibility, but the sensitivity with pure nickel oxide was low and higher exposure time was required.

And on the right, adding tin oxide to the nickel oxide composite or material component indicate there is a transparency change due to thickness. And measurement of different thicknesses did not show a pronounced enhancement of the material.

So basically, we concluded at this time that it shows the feasibility of nickel oxide based sensors, but the properties, process to control microstructures and fabricate diodes must be-- they have to be investigated further and also improved to make a practical detector. And basically the design of materials, reduction-oxidation state and understanding of that, film quality enhancements, and design of electrodes enhance the detector performances.

So coming back to our current work, the focus of our current work is to understand the nanoengineered coating. The self-cleaning and the cleaning is based on the lotus effect due to wettability. And the principle of lotus effect, which is generally known, is a natural phenomenon observed on the surface of the lotus plant leaves, and they refer to the high water repellence. And the structure of lotus leaves is used to understand surface sensing and surface behavior.

In the past we have presented papers related to DIC process using the lotus effect. And again, a hydrophobic polymer films were obtained by a solvent selective method composed of hydrophilic polymethylmethacrylate and hydrophobic polystyrene. And also we added a hydrophilic titania nanoparticles to that for understanding the contact angles.

The addition of titania nanoparticles changed the surface of the thin films from an anisotropic anisotropic morphology to a spherical isotropic surface due to hydrophobic

and hydrophilic repulsion. Again, the water contact angle measurements were done, and some of the results will be shown in coming slides.

Again, to recollect, the research has been performed on the flow of liquids on surfaces decorated with complex geometries indicating unusual wetting properties. Basically, the objective was to modify surface topographies with nanoengineered sensor materials embedded or embodied inside the polystyrene nanoparticles.

And the surface, morphological and optical characteristics of this composite that we are trying to target was prepared by wet and semi-wet techniques, and we determined contact angles. And its suitability for a sensor was also-- to-- we explored the possibility of using for sensor applications. So the approach for the development of sensing agent nanoparticles like titania is shown here. I don't want to go into detail of it. It's a great chemistry process.

And the synthesis was done in the lab, and the characterization was done. These nanoparticles were characterized with a differential light scattering instrument for size measurements, absorption, and IR measurement using appropriate spectrometers and [INAUDIBLE]. Spectrophotometers. And the ground state UV-Vis visible absorption spectra were measured using a double beam spectrometer. And again, infrared measurements were obtained with a thermo scientific FTIR instrument.

Here the picture shows how the titania was synthesized. And the technique, again, was explained in the past. And again, using the similar technique, nanoparticles of copper oxide were also synthesized. This graph shows the characteristics of the synthesized titania nanoparticles.

This is the absorption of titania in the particles in tetrahydrofuran solvent. And it shows that it is highly transmissive with no significant peaks within the UV-Vis in the UV visible region and the intensity decreases as the wavelength increases. And again, this graph shows the size distribution of the titania nanoparticles, varied from 10 to 100 nanometer range, and the average size of these particles were around 40 nanometers measured using DLS.

Again, we need to do the metrics. And the three different polymer mixtures were formulated using a polystyrene and polymethylmethacrylate to determine the effect of polymer mixtures effect on the hydrophobicity. And these are the three different representative [? weight ?] combinations we had chosen.

And the solution was prepared, and the process was repeated. This process was repeated for obtaining different mixtures and again, continued with our experiment.

This shows the synthesis of matrix material. And again, the only reason this picture is given is the temperature affected the color of this function as time. So the color changed over a period of time. Again, we want to-- we used the spin casting of a polymer solution using the lab made customized small system. And the polymer mixture were spin-cast onto glass substrates.

And the spin-cast material glass slide holder was developed in the lab. And the mixture was then spin-cast and were spun around like 400 rpm while applying the polymer mixture. And again, it was at a different-- higher rpm for 90 seconds to get the uniformity of the spin coat.

So this picture shows the dependence of morphology on aging. And on the left is the SEM image on a glass substrate without titania. On the right, the picture shows that our PMMA the polystyrene mixture with titania nanoparticles. And this is our own freshly prepared mixture composites.

And after aging of one week, the SEM SEM pictures were taken. And this shows that their spherical clusters were observed in the materials, so the aging cost aggregation of sensing nanoparticle. And this is a challenge that will be addressed in the future.

Then we went to measure the contact angles. And again, the contact angles were measured using NIH software available in the lab. There are two different plugins, one is Dropsnake and the other one is ContactAngle.

They do measure the contact angles in a little different way, and probably the Dropsnake plugin was a better one because you have a better control of the parameters and the user entry and user interface so that you get a better or more accurate angular measurement.

This is a system used to measure the contact angles, and this was developed or modified in the lab for measurements. These are representative pictures of droplets. And on the left, show-- you see these images about droplets of 50 microliter droplets on substrates.

And for the first combination of polystyrene and PMMA, the spray-coated substrate washed with cyclohexane showed that hydrophobic surface and the contact angles were greater than 90 degrees. On the right with a slightly different percentage of PS and polystyrene and PMMA, spin-coated on glass slide and washed with cyclohexane.

It demonstrated a hydrophilic surface because the contact angles were less than 90 degrees. And all of them demonstrated a superhydrophobic which is around 150 degrees or more. And so these both angles were averaged to get an average contact angle.

This only shows here that in using a different software the differences it makes. of-

- The left one has some difficulty because it has to be clear substrate and clear-
- the liquids are to be clear to get a better contact angles. On the right, the plugin used with a Dropsnake plugin that provided a better contact angle measurements, and it is user friendly.

This is a typical graph that will be obtained for each contact angle measurements. I

when we do the raw measurements, the washed, and the average measure-

- and annealed case. So this is a typical measurement graph. And in our case, as I said, we wanted to use titania nanoparticle filled composites.

So we did those studies. And after synthesizing on the glass particle or glass slides, we started measuring the hydrophobic or hydrophilic properties by using the contact angle method. This is the results that we obtained. This is a summary of contact angles when no cyclohexane wash was used. And we see that for the raw case, the contact angles were slightly-- they're greater than 90 degrees.

But none of these yielded thin film meeting the criteria of superhydrophobic of 150 degrees or more.

So with the raw contact angles as shown here with these combinations. This is for annealing at around 170 degrees centigrade, And this shows the contact angle with titania inside it. So there was not much difference here. The only thing probably that we see is that as the polystyrene decrease, the contact angle with the titanium increased. And that's what we observed so far.

Again, annealing was done at 170 degrees for one hour to observe the effects on angle. The boiling, we used-- because they were developed using tetrahydrofurans their boiling point is 66. And some of the-- there is some effect of these low boiling point compared to annealing temperatures. The solvent trapped in the thin film evaporates and creates pores within the thin film, increasing surface roughness. Again, the glass transition temperature is around 110 degrees centigrade, further roughening the surface.

So we observed that for the samples that were not subjected to cyclohexane wash, the effect of annealing significantly increased the contact angle, say, around 16 degrees increase. And the most significant increase was found in thin films with a greater amounts of polystyrene. And as polystyrene decreased, that also decreased.

So the overall effect of annealing is that the annealed samples that were subjected to cyclohexane wash, the effect of annealing was much less than those that had not been washed with cyclohexane. An average of 6.7 degrees increase was observed. And probably that's maybe due to the cyclohexane wash removed polystyrene from the matrix when annealed trapped solvent had more available paths to leave the thin film, resulting in a lesser effect in surface roughness.

So this is both promising as in both cases as the contact angle were similar. While annealing may not be practical for thin films, the cyclohexane wash is possible. And washing affected surface characteristics. Also, we saw some anisotropic effects of these titania filled sensors.

For example, in the case, isotropic distribution of water droplet on thin film coating with this combination showed no cyclohexane wash, annealed, and titania nanoparticles present. Whereas in this, it was isotropic in this case, and there was also an isotropic behavior with the cyclohexane wash, annealed, and titania present.

So basically, what we are trying to say is that these materials when we develop these characteristics could provide a better sensing possibilities for radiation purposes. Again, also that we wanted to keep the transparency within the range. And saw that for our case, the visibility was-- the transmission spectra, a and spectra here, was within the visible range and design matrix. This is also one of the parameters that we were be interested in.

Again, to summarize, the nanoparticles of sensing agents like titanium oxide, and also--  
- also-- lab students did copper too. And they were synthesized, and were--  
- their sizes were characterized using the DLS, that is the light scattering instrument, and then we did absorption spectroscopy. And IR spectroscopy there to understand their absorption, as well as the wavelength transmission over the visible range.

And the various polymer thin films were developed, spin-cast, annealed and measured, the contact angles were measured. And we have seen that the transmission of thin films did not show any significant effects due to low energy radiation. Again, to-- what we are continuing to do is to understand the nanoparticle behavior with the scheme that we are trying to use.

And the challenges are the clustering and lot of other issues which are there to remove them so that we could make a good sensor, a smart sensor for the radiation sensing, especially for high energy particles. Thank you.