The 1<sup>st</sup> International Conference on Advanced Materials for Printed Electronics and Sensors (ICAMPS2020)

**E-Conference** 

# **ABSTRACT BOOK**

10-11 September 2020 Faculty of Science, Chiang Mai University, Thailand









# Welcome Address

CHIANG MAL THAILAND 10-11 September 2020

# Assistant Professor Dr. Winita Punyodom

# Head of Materials Science Research Center Faculty of Science, Chiang Mai University



On behalf of the organizing committee, it gives me great pleasure to wish a very warm welcome to all participants to: The 1st International Conference on Advanced Materials for Printed Electronics and Sensors (ICAMPS2020). This conference is intended to provide the recent exciting updates on advanced materials, science and technology in printed electronics and sensors. We hope that this will be the crucial starting point and a good opportunity for new research ideas, scientific and technological innovation development as well as strengthened collaboration among academic and research institutions both at national and international levels. The conference is also a part of the global efforts to encourage students to improve research and communication skills with opportunity to publish in peer-reviewed journals/proceedings. We believe that these future-to-be young scientists will contribute greatly to the printed electronics and sensor research community.

The themes of this conference cover the following research fields: Gas Sensors and Biosensors for Sustainable Life, Printed Electronics and Devices for Sustainable Energy, and Advanced Materials for Biomedical Applications. The first theme includes the sensing materials, characterizations, mechanisms, modeling and simulation for chemical sensors, biosensors, diagnostics and healthcare, flexible, stretchable and wearable sensors for security and safety applications, and applications and manufacturing of chemical sensor systems. The second theme involves perovskite and emerging solar cell technologies, photocatalysts for sustainable energy, thermoelectric materials and flexible thin films for energy conversion devices, printed electronics technology for energy storage, flexible IoT devices for medical and agricultural applications. The last theme includes the recent developments in polymer design and synthesis, polymer processing and printing, polymer nanostructures and nanocomposites, bioplastics and polymers for sustainability, biomedical polymers and medical devices.

Although this conference is held during the challenging time, we wish that some of the participants who can visit Chiang Mai will enjoy the local culture and traditions. Those that are overseas, we wish to have a chance to welcome all of you in Chiang Mai in the near future. In this way, the organizing committee aims to make this conference both an outstanding scientific event and a memorable experience. Your participation will help us to achieve this. In closing, may I thank Chiang Mai University and our co-organizer the National Electronics and Computer Technology Center (NECTEC), National Science and Technology Development Agency (NSTDA) for their respective contributions. Thanks are also due to the various committees whose hard work and dedication have brought us to this point and made this conference possible. Last but not least, thanks to all participants, both from within Thailand and from overseas, for supporting this event by sharing your knowledge and experience.

I wish all of you would have a rewarding and enjoyable time participating in this first online international conference and we welcome any valuable suggestion.

Assistant Professor Dr. Winita Punyodom

Conference Chair

Head of Materials Science Research Center Faculty of Science, Chiang Mai University

# Welcome Address

CHIANG MAI, THAILAND 10-11 September 2020

# Associate Professor Dr. Pisith Singjai



Head of the Center of Excellence in Advanced Materials for Printed Electronics and Sensors

On behalf of the Center of Excellence in Advanced Materials for Printed Electronics and Sensors, which has been established from the partnership between the Thai Organic & Printed Electronics Innovation Center, National Electronics and Computer Technology Center (NECTEC), National Science and Technology Development Agency (NSTDA) and the Materials Science Research Center (MSRC), Faculty of Science, Chiang Mai University, may I add my own very warm welcome to the Plenary Lectures, Invited Speakers and all participants, especially to those from overseas who are joining the conference online and those who are visiting Chiang Mai, to this international conference entitled: The 1st International Conference on Advanced Materials for Printed Electronics and Sensors (ICAMPS2020).

It is an honor to host this first important conference at which such a distinguished group of internationally recognized speakers has been assembled. In recent years, Materials Science in the area of printed electronics, sensors and flexible devices at Chiang Mai University and our partnered institutions has become an important part in raising the research profile, both nationally and internationally, through attracting research grants and producing journal publications. This conference will focus on fundamental and applied researches and current developments related to materials science, physics, chemistry, engineering and other related fields. I would like to thank the Materials Science Research Center, Chiang Mai University, and the National Electronics and Computer Technology Center, National Science and Technology Development Agency, for their great contributions and efforts to co-organize this event. I believe that organizing an online international conference of this importance during this challenging time requires months of planning and preparation. I therefore commend the organizing committees from Chiang Mai University and our partnered institutions for their efforts in bringing us to this point. Hopefully, these efforts will be rewarded and the conference will meet everyone's highest expectations. All that remains now is for me to wish you all a rewarding conference with fruitful outcomes and, for anyone visiting Chiang Mai, an enjoyable stay and relaxing time.

Associate Professor Dr. Pisith Singjai

**Conference** Chair

Head of the Center of Excellence in Advanced Materials for Printed Electronics and Sensors

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10-11 September 2020, Faculty of Science, Chiang Mai University, Chiang Mai, Thailand

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# Development of Gas Sensor Technology for Safety and Health

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#### Abstract

We have been studying the development of gas sensor technology to fabricate high-performance gas sensors for a safe and secure society. Controlling the nanoscale microstructure of the gas sensing materials and introducing the new concepts to the gas detection are our strategies for sensitive and selective detection of harmful gases, such as NOx, CO and VOCs (volatile organic compounds). In addition, we now focus on the detection of the specific very low concentration gases in the breath or emitted from the skin, in order to apply gas sensors to healthcare monitoring.

The following three topics will be delivered in my presentation.

- High-performance semiconductor gas sensors by meso- and macro-porous structural control of metal oxides Surfactants and polymethyl metacrylate microspheres were used to control meso- and macro porous structure of metal oxides, respectively. The improvement of H<sub>2</sub> and NO<sub>2</sub> sensing properties by the porous structural control will be reported.
- Solid electrolyte CO gas sensors operable at room temperature by strict design of electrode materials The important role of metal oxides added to the Pt sensing and reference electrodes of a potentiometric NASICON (Na<sub>3</sub>Zr<sub>2</sub>Si<sub>2</sub>PO<sub>12</sub>) gas sensor will be reported.
- 3) MEMS (micro electro mechanical systems) gas sensors useful for healthcare applications by controlling the target gas adsorption and combustion behavior of sensor materials. The potential of adsorption/combustion-type MEMS gas sensors, in detecting selectively a low concentration of VOCs, which are possible biomakers for diagnosis of specific diseases and health checking, will be reported.

Keywords: Semiconductor gas sensors, Solid electrolyte gas sensors, MEMS gas sensors, CO, VOCs

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# Graphene Technology for Energy Storage Applications

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#### Abstract

2D and 3D Graphene has received increasing attention due to its unique physicochemical properties including high surface area, excellent conductivity, high mechanical strength, and ease of functionalization and synthesis. Graphene has recently applied in the area of energy storage applications including battery and supercapacitor. In our research group, electrochemical-exfoliated 2D graphene and reduced Graphene Oxide (rGO) are widely used for enhancing performance in various types of battery and supercapacitors. Moreover, 3D hollow graphite nanotetrapods by Vapor Phase Transport and In-situ Chemical Vapor Deposition/Etching is the technique used to fabricate 3D graphene for high-performance lithium-sulfur batteries based on 3-D graphene foam electrodes. Free standing of 3 layers graphene-sulfur cathode for Li-S battery is also demonstrated. For high power density applications, novel surfactant-stabilized graphene-polyaniline composite nanofiber for supercapacitor applications is successful achieved at 640 F/g. Moreover, ammonia strengthened graphene/CNT-wrapped polyaniline-nanofiber composites loaded with palladium nanoparticles supercapacitors is demonstrated at 611.8 F/g.



Keywords: Graphene, Printed Electronics, Supercapacitor, Battery, Energy Storage

## Gas Sensors Based on Semiconducting Metal Oxides: Fundamentals and Applications

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#### Abstract

Chemoresistive gas sensors based on semiconducting metal oxides (SMOX) are cost-efficient, small-sized and easy to integrate devices, which enable the detection and, sometimes, the quantification of the concentration of various reducing or oxidizing gases and vapors present in the ambient atmosphere. Because of their advantages they are in the focus of fundamental and applied research since almost 70 years with SnO<sub>2</sub>, WO<sub>3</sub>, In<sub>2</sub>O<sub>3</sub> and ZnO being the most widely studied and used in science and industry. Their applications are ranging from safety in households and industrial process control over in-cabin air quality to medical ones. Their fabrication technology evolved from individual devices built as ceramic pellets, which needed approx. 5W to reach their operation temperature, to the current state of the art miniaturized four-sensors-array with integrated driving and evaluation electronics, whose power consumption is only 20 mW and can be integrated even in mobile phones. The understanding of their sensing mechanism was initially based on ex-situ and surface science methods, thus leaving large gaps between investigation and application conditions and materials and, as a consequence, produced little useful information for the developers, who relied mostly on empirical optimization of their devices. With increasing availability of in-situ and operando investigation methods it became possible to bring fundamental understanding and sensor development together. Some examples of significant advances with practical applications in the improvement of gas sensor performances are: the understanding of the water vapor interference in gas sensing; the elucidation of the impact of conduction mechanism on the performance of n and p-type SMOX; the understanding of the chemical and electronic impact of noble metal additives on gas sensing performance. The talk will review the evolution of gas sensor technology and understanding of sensing with SMOX materials and will also present some of the most important current applications and most promising future ones.

Keywords: Semiconducting Metal Oxides; Gas sensors; Sensing Mechanism; Operando Investigations

### Flexible electronics at ITRI

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#### Abstract

Printed Electronics has been developed over the past two decades. There are lots of applications and products that have been brought to the market. Each major market region has deployed its own strategies to support the development of printed electronics.

Taiwan has strategically invested in flexible electronics since 2005. With the government's support, ITRI has established the first flexible electronics pilot laboratory in Asia in 2007. The purpose is to facilitate the development of flexible electronics with global partnerships. During these years, many Taiwanese companies have entered the stage of mass production in flexible electronics, including applications such as E-paper, printed touch panels, large area pressure sensors as well as material for solar cells. These companies have taken on leading roles in their respective areas.

With growth opportunities for flexible electronics being limited within the display industry, With the display industry becoming mature, printed electronics is seeking for a new drivers. And with the effects of the Covid-19 pandemic spread widely, new opportunities in the medical device area are becoming apparent. However, only and integrated product-solution approach and versatile business model will ensure successful. Hence, ITRI has established two key service offerings to smooth the stony road from lab to fab, available to all global partners.

1. Our versatile roll-to-roll and printing lab is designed for new process/equipment and material evaluation.

2. A one-stop shopping IoT integrated service center (IisC) is to assist the partners to bring early prototypes all the way to a market-ready product.

Through these services, we are able to speed up and smoothen the path from lab to fab (TRL 3~4 all the way to TRL 6 or higher) for our partners.

Keywords: Flexible electronics; Printed Electronics; E-paper; Printed touch panels; Solar cells.

# Development of Multi-dimensional Metal-oxide Nanostructures for Environmental Gas-Sensing Applications



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#### Abstract

Metal oxide composites comprising multi-dimensional nanostructures are attractive for various gassensing applications due to their large effective specific surface area, unique electronic/chemical characteristics and high environmental stability. Over the past several years, we have developed highly sensitive gas sensors based on multi-dimensional metal-oxide nanostructures prepared by different methods. For instance, 1D carbonnanotubes (CNTs)-0D metal oxide (SnO<sub>2</sub>, WO<sub>3</sub> and MoO<sub>3</sub>) nanocomposites are developed by means of powder mixing and electron beam evaporation. Appropriate compositions of the nanocomposites lead to enhance responses towards gases such as NO<sub>2</sub>, ethanol, acetone and H<sub>2</sub>. In addition, carbon-doped 1D metal oxide nanostructures such as WO<sub>3</sub> nanorods were produced by sputtering with glancing angle deposition and demonstrated to offer a significantly higher gas-sensing response compared with conventional undoped dense thin film prepared by conventional sputtering method. Moreover, carbon-coated 3D ZnO nanotetrapods fabricated by two-step vapor phase transport shows considerably enhanced response towards NO<sub>2</sub> compared with uncoated ZnO nanotetrapods. Additionally, graphene-metal oxides (SnO<sub>2</sub>, WO<sub>3</sub>, CuO and Bi<sub>2</sub>WO<sub>6</sub>) composite thick film gas sensors are fabricated based on one-step flame spray pyrolysis (FSP), electrolytic exfoliation and spin coating. The gas-sensing characteristics towards ethanol, acetone and NO<sub>2</sub> gases of the composite were found to be significantly improved with optimal graphene loading concentrations in the range of 0.5-5 wt%. Furthermore, 1D WO<sub>3</sub> nanorods-0D SnO<sub>2</sub> nanocomposites produced by fluidized bed reaction and FSP followed by thermal oxidation offered high selectivity and sensitivity towards NO<sub>2</sub>.

Keywords: Metal oxide nanostructures; Multi-dimensional nanocomposites; Gas sensor

# Label-free colorimetric aptasensor utilizing cationic perylene probe and localized surface plasmon resonance of gold nanoparticles for rapid detection of Aflatoxin B1



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#### Abstract

A label-free colorimetric aptasensor for rapid detection of aflatoxin B1 (AFB1) has been developed using specific aptamer, cationic perylene probe (CPP), and unmodified citrate – stabilized AuNPs. In the absence of AFB1, the free specific aptamer forms complex structures with the CPP, resulting in the solution remaining red without aggregation of AuNPs. Conversely, in the presence of AFB1, the aptamer competes for interaction with AFB1 and CPP. Notably, the aptamer is strongly specific to AFB1, leading to CPP free in the solution. When unmodified citrate – stabilized AuNPs are supplemented, the color turns blue as a result of AuNPs aggregation induced by CPP. The color change of AuNPs comes from localized surface plasmon resonance absorption, which relates to the size, shape, and agglomeration of the nanoparticles. This phenomenon can be measured through colorimetric detection. In this work, we presented two approaches, i.e., by using a spectrophotometer and a homemade colorimeter as a detector, which provided the limit of detections of 0.36 and 0.18 ng/mL, respectively. The application for the determination of AFB1 in rice and peanut samples was carried out. The proposed colorimetric aptasensor offers advantages in terms of simplicity, rapid detection, and high selectivity. Furthermore, the sensitivity for the real application is practical when coupled with an affinity column as sample preparation.



Keywords: Aflatoxin; Aptamer; Cationic perylene probe; Gold nanoparticles; Colorimetric detection

# Digitalizing biosensor: Next generation of bionanoelectrochemical based sensing



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#### Abstract

The integration of bionano-electrochemical biosensors and mobile technology is more in demand as efficient and affordable analytical tools for various analytes detection in many fields. Such platforms are very useful for pointof-care applications that facilitate personalized health care management since patients can monitor their health at home and data can be easily transmitted to the cloud for collecting and sharing with clinicians and caregivers. Additionally, low cost and portable biosensors integrated with smartphones could become a real-time monitoring device in resourcelimited settings for food safety and control, environmental monitoring and clinical diagnostics. Therefore, our research group, in collaboration with Silicon Craft Technology Public Company Limited, is developing the electrochemicalbased device containing a microchip for electrical analysis and a near-field communication (NFC) antenna for mobile phone connection. The NFC air-interface is selected to be a connection between the microchip on the test strip and smartphone to simplify measuring units and make the test strip become a passive operated device, running without battery. The miniaturized devices were designed to detect aging and stress biomarkers reported in biological age and stress index. We also integrate health sensors such as vital signs, periodic blood, urine test, physical fitness and motion test to monitor the health status of the community members. With all these health data, the local healthcare facility will know in advance the health status and design plans along an appropriate lifestyle for each member. Furthermore, we are developing the biosensor based on the urease enzyme extracted from soybean for heavy metal detection. The developed biosensors provide high sensitivity and selectivity, low cost and easy to use which could be an alternative device to detect the heavy metal contamination in water, food and cosmetics.

Keyword: electrochemical biosensor; near-field communication; biomarkers; heavy metals

# Recent progress in the development of non-MEMS MOS gas sensing platform at NECTEC-NSTDA



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#### Abstract

Since late 2017, we had publicly presented a non-MEMS gas-sensing platform, namely GASSET, which can support a variety of metal oxide semiconducting (MOS) gas-sensing materials for the real-world applications. The device has many advantages including low operating power, maskless sensing film coating process and no need of expensive MEMS facilities, which is presently the state-of-the-art technology for fabricating the commercial low-power MOS-type gas sensors. However, there were some limitations of the low maximum operating temperature of 250 °C, limited temperature uniformity on sensing area and limited temperature processability due to the chip holding material. In this work, we report the solutions to these problems via the use of different titanium nitride (TiN) sputtering process and electrode redesign. The quality of titanium nitride heater-film has been improved with the use of upward sputtering process and the optimization of sputtering process parameters. The resulting TiN films display substantially improved quality with ability to operate at a temperature of 400 °C for long period with no change in electrical properties. In addition, the electrode was redesign to obtain better temperature uniformity over the sensing area. Therefore, the improved GASSET platform will allow wider utilization of MOS gas-sensing materials in commercial applications.

Keywords: GASSET; gas sensor; metal oxide semiconductor; platform

# Rapid and Highly Selective Detection of H<sub>2</sub>S by Ag and Ni doped CaCu<sub>3</sub>Ti<sub>4</sub>O<sub>12</sub> Film Sensors



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#### Abstract

The detection of toxic gases such as  $H_2S$  is relevant in the food industries in the maintenance of the sewage system and health science. We have achieved sub-ppm sensitivity toward  $H_2S$  by using Ag and Ni doped CaCu<sub>3</sub>Ti<sub>4</sub>O<sub>12</sub> (CCTO) thin films prepared by a cost effective sol-gel method. Ag and Ni doped CCTO films were found to be remarkable sensors towards  $H_2S$  in the concentration range of 0.2-10 ppm. When compared with undoped CCTO sensors, Ag and Ni dopants cause a dramatic improvement of the response towards  $H_2S$  gas up to 10 times higher. These film sensors typically operated at ca. 250 °C and showed much shorter response times than that of undoped one. The operating principle of gas sensing mechanisms have been proposed.

Keywords: Ag-doped CaCu<sub>3</sub>Ti<sub>4</sub>O<sub>12</sub>; Ag-doped CaCu<sub>3</sub>Ti<sub>4</sub>O<sub>12</sub>; H<sub>2</sub>S sensor

# Semiconductor Nanomaterial for Low-Cost and Stable Perovskite Solar Cell Fabrication



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### Abstract

Development of electron transport layer ETL is one of the most important keys to improve performance and stability of the planar perovskite solar cells (PSCs). Titanium dioxide (TiO<sub>2</sub>) has been widely used as an effective electron transport layer. However, its poor conductivity and low electron mobilities limit the performance of planar PSCs. In this work we demonstrate the use of semiconductor nanoparticle SNPs such as zinc cadmium sulfide and silver-indium-sulfide as dopant in TiO<sub>2</sub> electron transport layer in planar PSCs structure. We also develop convective deposition technique to deposit the ETL layer to reduce material consumption and waste obtained by the conventional spin coating. The TiO<sub>2</sub>:SNPs electron transport layer shows the improvement of surface morphology, electron mobility, and electron extraction compared with pristine TiO<sub>2</sub>. The optimum TiO<sub>2</sub>:SNP in FTO/TiO<sub>2</sub>:SNPs/Perovskite/Spiro-OMeTAD/Au solar cell structure perform better in terms of power conversion efficiency and stability compared to the one with conventional ETL. In addition, the convective deposition is used to demonstrate film deposition for upscale fabrication of PSCs on rigid and flexible substrate.

Keywords: Perovskite; Electron transporting layer; AgInS<sub>2</sub>; ZnCdS; TiO<sub>2</sub>; Convective Deposition

# WO<sub>3</sub>/BiOBr heterojunction photocatalysts: Activity improvement and mechanistic study on selective oxidation of benzylamine



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#### Abstract

To improve the photocatalytic activity of materials, several approaches including metals/non-metals doping, introducing oxygen vacancy and forming heterojunction have been employed. In this work, we focused on enhancing the photocatalytic performance of bismuth oxybromide (BiOBr) for the oxidative coupling of benzylamine by forming heterojunction with WO<sub>3</sub>. The reaction was carried out at room temperature under an O<sub>2</sub> flow using a white LED as a light source. BiOBr alone provided only 40% yield of *N*-benzylidenebenzylamine, however the yield increased to > 80% by coupling with WO<sub>3</sub>. Mechanistic studies based on radical quenching experiment, EPR trapping study and Hammett plot were carried out to reveal a possible surface reaction mechanism in those heterostructure systems. Transient photocurrent and EIS were used to evaluate the electron-hole transfer and separation efficiencies of the materials. The insightful information on the structure-activity relationship and the involvement of reactive oxygen species in benzylamines transformation elucidated in this work lay an important background for the material design and encourage a further development of highly efficient photocatalysts toward organic fine chemical syntheses.



Keywords: BiOBr; WO<sub>3</sub>; Visible light; Heterojunction; Benzylamine

### Fabrication of Wearable Thermoelectric Power Generator

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#### Abstract

Thermoelectric generator (TEG) is proposed as a solid state energy harvester from waste heat sources. TEG produces the electrical power from a temperature differences applied across the device. Over the last decade, the human body has been considered as a heat source for the wearable TEGs. It may become attraction for an alternative power generation technique compared to other conventional ones used for many wearable devices. The energy conversion of the wearable TEGs has been demonstrated in order of micro watt range or less. It is therefore becomes a challenge to generate sufficient amounts of energy from body heat source to gain more energy intensive devices. The amount of generated electrical power being produced from TEG depends on material properties (ZT) and temperature different between two sides of module ( $\Delta T$ ). Bismuth telluride (Bi<sub>2</sub>Te<sub>3</sub>) and antimony telluride (Sb<sub>2</sub>Te<sub>3</sub>) alloy are the most commonly used as TE materials because of their high ZT value at room temperature. Moreover, these materials are also easily deposited in thin films to make the flexible devices. In this work, the deposition processes of n-type Bi<sub>2</sub>Te<sub>3</sub> and p-type Sb<sub>2</sub>Te<sub>3</sub> thin films on flexible substrate will be investigated. It has been shown that the highly (00l)-oriented Bi<sub>2</sub>Te<sub>3</sub> and Sb<sub>2</sub>Te<sub>3</sub> structures are expected to create a higher thermoelectric performance (both electrical and thermal properties) than ordinary films. The dc magnetron sputtering conditions such as sputtering power, working pressure and substrate temperature were optimized. The high thermoelectric performance of thin films was achieved. With relationship between thermoelectric properties and microstructure/stoichiometric composition, the designed structure of layered compact feature with stoichiometry provides the relatively high thermoelectric efficiency.

Keywords: Wearable Thermoelectric; Power Generator; Bi<sub>2</sub>Te<sub>3</sub> film; Sb<sub>2</sub>Te<sub>3</sub> film

# Recent Progress in Printed Electrode for Energy Storage Applications



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#### Abstract

There is an increasing interest in the development of miniaturized and flexible devices for applications in wearable and portable electronics today. However, one obstacle that hindered was the relatively large size and weight of energy storage. Therefore, the development of thin energy storage supply is an important solution for making lightweight wearable devices. Printed electronics refer to a process in which printing technology is that can be used to prepare thinner electronic devices and leads to a lightweight design. In addition, printing process is very simple and inexpensive, with the use of traditional printing techniques that are highly accurate, such as inkjet printing, screen printing, roll-to-roll printing. For this reason, printed electronics technology is suitable solution to prepare thinner energy storage devices. In this section, we are going to present the advancement of research on printed energy storage namely supercapacitor and batteries in terms of the printing method being applied and development of electrode materials and electrolyte that is suitable for printing process. In the last part, additionally, the recent progress of our researching in printed graphene-based electrode for supercapacitor and battery is presented.

Keywords: Printed energy storage devices; Battery; Supercapacitor

# Digitization of Human Body Odor – Method, Devices and Application in Digital Health

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#### Abstract

Recently, internet-of-things (IoT) health monitoring systems have become of unprecedented interest owing to their versatile functionalities such as being point-of-care, easy-to-use, low-cost and real-time. So far, various wearable devices based on measuring of physiological signals and biokinetics have been proposed but only a few if not at all have existed based on measurement of the body odor. In this lecture, we will present the benefits of human body odor detection based on our research experience on the past several years. We will show the results to support the notion that volatile organic compounds as released from the human body can give some information about diseases, behavior and health status of a person. In addition, it was found that body odor is one of the physical characteristics of a human that can be used to identify people. We have invented various platforms to make electronic nose (e-nose) wearable, including armband e-nose, sniffing shoes and smelling shirts. These novel devices can collect human body information based on the volatiles released from the armpits and feet of human and transmit the data wirelessly, showing a viable potential for real-time personal healthcare monitoring.

Keywords: Body odor; Gas sensor; Chemical sensor; Electronic nose; Health monitoring

# Surface Modification for Improved Biocompatibility of Absorbable Nerve Guides Fabricated by Electrospinning and 3D Printing



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#### Abstract

The principal aim of this project is to develop an absorbable nerve guide for use as temporary scaffold in peripheral nerve repair. New approaches in surface modification of biodegradable copolyester scaffolds are showing great promise. Specialty medical grade poly(L-lactide-co-ε-caprolactone) (PLCL) and poly(L-lactide-co-glycolide) (PLGA) copolymers according to ASTM F1925-17 guidelines (Standard Specification for Semi-Crystalline Polylactide Polymer and Copolymer Resins for Surgical Implants) were synthesized and characterized to meet the specific requirements of nerve guide applications. In addition, conductive polymers (CPs) such as polypyrrole (PPy) have been demonstrated as having the ability to provide electrical stimulation for neurons in guided axonal extension. In this research, a combination of 3D printing and electrospinning has been used to fabricate biodegradable, conductive scaffolds by dispersing PPy particles on PLCL and PLGA scaffolds. The fabricated scaffolds were characterized using X-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM), as well as the measurements of surface wettability and conductivity test were performed. It was found that a highly electrospun fiber is clearly embedded within the 3D printed construct which exhibited uniformly oriented channels and pores having micrometer resolution. Furthermore, PLCL and PLGA scaffolds were successfully deposited of the PPy particles on fibers. Scaffolds deposited with PPy particles showed significantly greater hydrophilicities which were a consequence of nitrogenated carbon functional groups in PPy. Cells cultivated of both the PLCL and PLGA scaffolds showed high viability (>95%). Furthermore, attachment and proliferation of Mouse fibroblast cells (L929) on PPy-coated and uncoated of 3D printed scaffolds with electrospun fibers were increased when compared to those without the fibers. The results of this study demonstrate the potential to create scaffold prototypes by combining 3D printing and electrospinning techniques. Moreover, PPy-coated and uncoated of 3D printed scaffolds with electrospun fibers of polymers enhanced both cell viability and growth without incurring any cytotoxic effects.

Keywords: Surface modification; poly(L-lactide-co- $\varepsilon$ -caprolactone); poly(L-lactide-co-glycolide); 3D printing; electrospinning; conductive polymers; biocompatibility

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## Trimethylated Chitosan-based Nanoparticles: Their Syntheses and Role as DNA/RNA Delivery Carriers



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#### Abstract

Quaternized chitosan, a quaternary ammonium derivative of chitosan, has receiving much interests in the recent decade, due to the improved water solubility in a wider pH range, compared to a native chitosan itself. Thus, with some inherent advantages (biocompatibility, biodegradability, and mucoadhesive ability) and the additional properties (e.g. water solubility in a wider pH condition, enhanced transfection efficiency, and better antimicrobial activity), quaternized chitosan has emerged to solve the limited applications for native chitosan. Meanwhile, quaternized chitosan in the form of nanoparticles are also extensively used for diverse applications, especially biomedical application, because of their small size and large specific surface area. So, research woks in the field of syntheses and applications of the quternized chitosan particles: 1) ionic gelation of chitosan with anionic gelators (e.g. tripolyphosphate or protein) and followed by quternization of the remaining  $-NH_2$  or -OH on the chitosan structure 2) ionic gelation of quaternized chitosan with anionic gelator, and 3) self-assembly of amphiphilic quaternized chitosan.

Herein, we would like to introduce alternative routes to quaternized chitosan particles through an emulsifierfree emulsion polymerization. Trimethylated chitosan (TMC), a quaternized chitosan prepared via methylation of chitosan, is used as a starting materials. TMC itself has different types of amino groups on it structure, including 1°, 2°, 3°, amines and quaternary ammonium group. The emulsifier-free emulsion polymerization makes use of some of those amines with different activators to generate free-radicals capable of initiate a polymerization of vinyl monomers, resulting in colloidally stable TMC particles without the addition of surfactants. Some of synthetic routes illustrated here consist of 1) thermally activated 1° amine/t-butyl hydroeroxide system 2) photo-induced 3° amine/camphorquinone system, and 3) photo-induced 3° amine/riboflavin system. The effects of important polymerization parameters are emphasized, as well as key characteristics (e.g. chemical, physical, biological properties) of the resulting particles are illustrated.

Since these particles possess cationic nature, their ability as DNA/RNA delivery carriers is also presented. TMC-based nanoparticles were developed to use as a carriers of gene in delivery systems. Not only the high transfection efficiency, but also the controlled release of the DNA/RNA into cells are expected. Moreover, several studies have shown that TMC particles exert adjuvant-like effects on dendritic cells and can be used as potent adjuvants and delivery systems capable of inducing mucosal immunity. Therefore, 2 examples as potential DNA/RNA delivery application of TMC particles are also presented. The first one is the vaccine development from TMC particles prepared by TMC/tripolyphosphate ionic gelation as an adjuvant and carriers for HA2 and NP proteins of influenza virus. The *in vitro* study of these influenza particles against primary human intranasal epithelium cells (HNEpCs) and human monocyte-derived dendritic cells (MoDCs) is conducted. The second investigation is the *in vitro* transfection study of TMC/PHEMA particles prepared by an emulsifier-free emulsion polymerization in shrimp (*Litopenaeus vannamei*).

Keywords: Quaternized Chitosan; Trimethylated chitosan; Gene Delivery; Vaccine; Adjuvant.

# Trash to Treasures: Chemical Recycling of Polyesters for Use as Value-added Functional Materials



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### Abstract

Rapid increase in plastic consumption rate leads to many serious environmental problems. Although mechanical recycling of post-consumer plastics has been practiced, deterioration in properties of the resulting products limits their use to only low-value products. Chemical recycling of plastic wastes into their smaller-sized constituents is vitally important in converting these into starting materials for other value-added products, which can accelerate global transition towards the circular economy for sustainable development. In our research center, processes for effective chemical recycling of post-consumer polyester products, especially the most widely-used poly(ethylene terephthalate) (PET), polylactide (PLA), and polybutylene succinate (PBS), utilizing alcoholysis reaction have been developed. Microwave irradiation and suitable catalyst systems are employed, in comparison with conventional heating sources. Effects of chemical structures and number of hydroxyl functional groups of the alcohol reagents on the reaction efficiency, production yield, and structures of the resulting alcoholyzed products have been examined. Chemical structures of the products and insights into mechanisms of the reactions were investigated by gel-permeation chromatography (GPC), 2-dimensional nuclear magnetic resonance (2D-NMR), and Fourier transform infrared (FTIR) spectroscopy. Efficiency of the alcoholysis process, types of the alcohol reagents, and the process conditions have been successfully optimized to generate products with desired chemical structures and properties. Feasibility of scaling up the process has been assessed. These resulting products have high potential for use as starting materials for value-added products, such as, metal organic framework (MOF) materials, polyester-cured natural rubbers, aliphaticaromatic copolyesters, degradable polyester-based adhesives, polyurethane products, and polymeric 3D- printing filaments.

Keywords: Chemical recycling, Polyesters, PET, PLA, Alcoholysis

# Cellulose-based Smart Materials : Modifications, Industrial Applications and Sustainability



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#### Abstract

Limited resources and a growing world population require new ways of thinking. Worldwide, the bioeconomy is gaining traction. But despite considerable successes, a lot of research and development still needs to be done. Increasing recycled content in plastics is an important way to reduce dependence on virgin fossil carbon resources. However, alternative feedstocks, such as bio-based and renewable, should also be encouraged in order to decrease the dependency of the plastics industry on finite fossil carbon resources, and the future demand should be met by the most sustainable options available. It was remarkable to note that the utilization of cellulose was versatile in many areas of research. It exhibited in many outstanding properties including high stiffness, high thermal and dimensional stability, high chemical resistance as well as high aspect ratio. The use of cellulose was therefore gained many interests on many areas of research. It was important to note that with the growth of human population, the development on functional materials from bio-based resource was one of the key factors in order to fulfill the need of life. Cellulose was one of the most abundant occurring bio-based materials. It can be therefore gained many interests in order to develop for serving and facilitating of human life. With the long term of experience and many collaborative on cellulose research, it was further developed on its conductivity. The application of conductive cellulose was therefore versatile in many areas of research such as active packaging, electronic substrate with additional feature of flexibility, functional membrane as well as chemical sensor. A number of cellulose based material for active packaging has been exponentially increasing. Numerous approaches of cellulose packaging gained many interests due to excellent properties of cellulose such as mechanical properties, thermal stability, chemical resistance and optical properties. The emergence of cellulose based packaging was also encouraged to be developed for environmentally friendly purpose.

Keywords: Cellulose; Smart Materials; Bio-based; Modifications; Industrial Applications

# Portable graphene-based electrochemical genosensor for rapid Mycobacterium tuberculosis detection



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#### Abstract

This work present a novel integrated point-of-care platform to detect a *Mycobacterium tuberculosis* (Mtb), Our approach employs loop-mediated isothermal amplification (LAMP) to amplify Mtb-DNA and the screenprinted graphene electrode (SPGE) for a label-free electrochemical analysis of DNA amplicons. When used with a portable potentiostat device developed in-house, the system (LAMP-EC) can simultaneously analyze and report the results in qualitative analysis. Under optimized conditions, LAMP-EC showed a comparable detection limit to the previously developed LAMP assay with a lateral flow readout. This highly specific technique could also determine the presence of TB in 104 blinded clinical samples with 100% accuracy. Beside these merits, our technique can be easily adopted due to its affordability (~USD2.5/test) and rapidity (<65 min turnaround time), a practical incentive that appeals to users inboth high- and low-resource settings.

Keywords: Mycobacterium tuberculosis; mini-potentiostat; graphene; screen-printed electrode; LAMP-EC

### Flame-made AgO-loaded LaFeO3-based Acetylene Gas Sensor

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#### Abstract

Power transformers are a vital component of energy distribution. In order to prevent costly transformer failure such as overheating of insulation, arc discharge, low energy sparking and bubble discharge, identification of acetylene gas in transformer oil via dissolved gas analysis can easily and quickly provide information about these potential failure, which relates to the safety of the transformer system. In this present work, AgO functionalized-LaFeO<sub>3</sub> *p*-type semiconductor has been synthesized using flame spray pyrolysis (FSP) in a single step for the first time and systematically evaluated for acetylene detection. The structural characterizations of X-ray and microscopy analyses revealed that AgO particles dispersed on orthorhombic LaFeO<sub>3</sub> supporting materials. The sensing films were fabricated to homogenously form by a screen-printing process and were evaluated towards low concentration of acetylene, while 0.1 wt% Ag-loaded LaFeO<sub>3</sub> sensor exhibited the highest sensor signal of 40 towards 50 ppm acetylene, which is about 10 times higher than those of unloaded sensor at 200 °C. Moreover, the sensor displayed high acetylene selectivity against C<sub>2</sub>H<sub>4</sub>, CH<sub>4</sub>, H<sub>2</sub>, CO and CO<sub>2</sub> in both dry air and humidity. Therefore, Ag-loaded LaFeO<sub>3</sub> was possible candidate to attribute for responsive and selective detection of acetylene at low ppm levels and may be practically useful for power transformers and industrials applications.

Keywords: Flame spray pyrolysis; AgO; LaFeO<sub>3</sub>; acetylene detection

### Flame-made Ru-functionalized WO<sub>3</sub> Nanoparticles for H<sub>2</sub>S Sensing

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#### Abstract

Hydrogen sulfide (H<sub>2</sub>S) is a toxic, odorous, colorless, flammable gas and used in industrial processes, so it can be dangerous to human health. The exposure to H<sub>2</sub>S can cause a wide range of effects to health including eyes irritation, disruption of olfactory receptors and respiration systems, paralysis, and death. Hence, it is important to have a monitoring system to detect the H<sub>2</sub>S exposed in the air. In the present work, 0-1 wt% Ru-functionalized WO<sub>3</sub> nanoparticles have been synthesized by flame spray pyrolysis (FSP) in one step and studied for H<sub>2</sub>S detection. The structural characterizations by X-ray spectroscopies and electron microscopies confirmed that the dimension of monoclinic WO<sub>3</sub> nanoparticles was around 5-20 nm with spherical shape. The sensing films were fabricated by spin coating technique on Au/Al<sub>2</sub>O<sub>3</sub> substrates and systematically tested towards H<sub>2</sub>S at working temperatures ranging from 200–400 °C. For the sensing result, the optimal 0.2 wt% Ru-functionalized WO<sub>3</sub> performed the highest response of 61.9 towards 10 ppm H<sub>2</sub>S at the optimal working temperature of 350 °C. Moreover, the sensor were selective to H<sub>2</sub>S against CH<sub>3</sub>SH, CH<sub>3</sub>SCH<sub>3</sub>, C<sub>3</sub>H<sub>6</sub>O, C<sub>2</sub>H<sub>5</sub>OH, CH<sub>3</sub>OH, CH<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, C<sub>8</sub>H<sub>10</sub>, CH<sub>2</sub>O, CH<sub>3</sub>COOH, C<sub>3</sub>H<sub>6</sub>O<sub>3</sub>, NO<sub>2</sub>, NO, H<sub>2</sub> and CH<sub>4</sub>. Therefore, the Ru-functionalized WO<sub>3</sub> is a promising sensing material as for H<sub>2</sub>S detection and to be benefit for breath analysis and industrial applications.

Keywords: Flame spray pyrolysis, Ruthenium, WO<sub>3</sub>, Hydrogen sulfide, Sensors

## Crucial role of gold nanoparticles functionalized zinc oxide nanorods for a non-enzymatic glucose detection

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#### Abstract

Investigation and monitoring of glucose levels in the blood, food, and beverage are crucial for diabetic patients. Now a day, high sensitivity and realizable glucose sensor rely on an immobilized enzymatic system. However, the stability, lifetime, and the cost of the enzyme are the massive drawbacks of the immobilized enzymatic glucose detection. To overcome the above issues, we propose an alternative way to detect glucose without enzyme immobilization by utilizing the nanostructure materials. The chemical functionalization of gold nanoparticles (Au-NPs) on zinc oxide nanorod arrays (ZnO-NRs) was accomplished as a label-free electrode for electrochemical glucose detection via electrocatalytic oxidation. The functionalized Au-NPs conversed to Au(OH)<sub>ads</sub> and generated hydroxide anion by chemisorption at the surface. Then, the glucose molecules were adsorbed on Au(OH)<sub>ads</sub> and transferred electron from the adsorbed species to Au. After that, the glucose oxidized to be gluconolactone (glucose + Au(OH)<sub>ads</sub>  $\rightarrow$  gluconolactone + Au + electron + H<sup>+</sup>). The prepared electrodes demonstrated the superior activity of glucose detection in a wide range between 0.5 – 10 mM. Glucose sensitivity and the limit of detection (LOD) of 157.34  $\mu$ A/cm<sup>2</sup> mM<sup>-1</sup> and 0.055 mM are obtained via controlling the density of Au-NPs. Moreover, the interference effect investigated in ascorbic acid (AA) and uric acids (UA) indicated the high specificity of the prepared electrode.

Keywords: Non-enzymatic glucose detection, ZnO nanorods, Au nanoparticles.
# Synthesis of few layers graphene–sodium dodecylbenzenesulfonate (SDBS) via electrolytic exfoliation for screen printed supercapacitor electrodes

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#### Abstract

Electrolytic exfoliation of graphene is a promising method for fast, green, and low cost graphene production. In this work, a common anionic surfactant in aqueous solution, namely sodium dodecylbenzenesulfonate (SDBS), has been used to prepare graphene by exfoliation of graphite. Conductive ink of SDBS-graphene was fabricated by ballmilling with carbon black as conductive filler and polyvinylidene fluoride (PVDF) as binder in N-Methyl-2pyrrolidone (NMP). The ink was screen printed onto carbon fiber paper in order to serve as supercapacitor electrode. Then, a symmetric coin-cell (CR2032) supercapacitor with SDBS-graphene in 1 M H<sub>2</sub>SO<sub>4</sub> was assembled. In electrochemical measurements, cyclic voltammetry, galvanostatic charge-discharge, and electrochemical impedance spectroscopy (EIS), the specific capacitance obtained from GCD of SDBS-graphene was150 Fg<sup>-1</sup> at 0.25 Ag<sup>-1</sup> and showed a superior cycling stability of 95% capacitance retention after 10,000 cycles at 3 Ag<sup>-1</sup>, compared to that of reduced graphene oxide (rGO) synthesized from Hummer's method was 76.5 Fg<sup>-1</sup> at 0.25 Ag<sup>-1</sup>. This suggests that the exfoliated SDBS-graphene has possible applications for high performance and long life EDLC supercapacitors.

Keywords: supercapacitor, electrochemical exfoliation; graphene; sodium dodecylbenzenesulfonate

# Copper(II) and palladium(II) complexes with 4-[(2hydroxybenzylidene)amino]benzoic acid: synthesis, characterization and potential application in non-enzymatic detection of hydrogen peroxide

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## Abstract

A bidentate Schiff base, 4-[(2-hydroxybenzylidene)amino]benzoic acid and its metal complexes containing copper(II) and palladium(II) have been synthesized and characterized. The molecular structures of the Schiff base and its complexes were elucidated by elemental analysis and spectroscopic techniques (FT-IR, <sup>1</sup>H-NMR and <sup>13</sup>C-NMR). The CHN microanalytical results for the Cu(II) and Pd(II) complexes agree with the metal:ligand ratio as 1:2, while the FT-IR and NMR data support the formation of metal complexes through the coordination of the phenolic O and the azomethine N atoms of the bidentate Schiff bases to the central Cu(II) and Pd(II), respectively. One of the notable features is that the presence of Cu(II) and Pd(II) complexes exhibit potential application for non-enzymatic detection of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>). The result can be evident by observing the electrochemical behavior of H<sub>2</sub>O<sub>2</sub> on the modified glassy carbon electrode using cyclic voltammetry and amperometry.

*Keywords:* 4-[(2-hydroxybenzylidene)amino]benzoic acid; copper(II) and palladium(II) complexes; spectroscopic techniques; non-enzymatic; hydrogen peroxide

# Label-free Electrochemical Biosensor for Detection of microRNA Cancer Biomarker based on Signal Amplification using Redox Probe Methylene blue and a Gold Nanoparticles/ Polypyrrole/Graphene Electrode

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## Abstract

MicroRNAs belong to a class of small non-coding RNAs that regulate numerous biological processes by targeting a broad set of messengers RNAs. Various recent studies showed a correlation between changes in miRNA levels with genetic diseases like cancer. Therefore, our aim is to develop a label-free microRNA detection approach for breast cancer diagnosis. We selected microRNA-21 as a biomarker of breast cancer for this study. In this work, a highly sensitive and label-free sensor was developed for the detection of microRNA-21 based on a graphene (GP)/ polypyrrole (PPY)/gold nanoparticles (AuNPs) nanocomposite to increase electron transfer of methylene blue at the electrode resulting in signal amplification. The detection was performed using differential pulse voltammetry (DPV) and the oxidation peak current of the redox probe, methylene blue (MB), under the optimal condition was determined to monitor the hybridization of capture DNA-21 and microRNA-21. The proposed biosensor reveals a linear range from 0.0010 pM to 1.0 nM with a low detection limit of 0.020 fM. In addition, the microRNA-21 biosensor provides good sensitivity, selectivity, reproducibility, and stability which had a promising application in clinical research and diagnostic applications.

Keyword: microRNA-21; graphene; polypyrrole; gold nanoparticles; methylene blue

# A colorimetric aptasensor for chlorpyrifos detection based on gold nanoparticles aggregation and cationic polymer

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## Abstract

Chlorpyrifos is one of toxic organophosphate insecticides that widely used to prevent and control a wide range of insects on crops. The contamination of chlorpyrifos in crops brings a risk to human health. Therefore, the monitoring of chlorpyrifos residues in an agricultural product is important. In this present study, a colorimetric aptasensor for chlorpyrifos detection based on gold nanoparticles aggregation using cationic polymer, polyethylenimine (PEI), was investigated. In the absence of chlorpyrifos, the negatively charged phosphate backbone of the aptamer can interact with the cationic PEI. The addition of chlorpyrifos, the aptamer can specifically bind to the target analyte and the cationic PEI remain free, leading to aggregation and dispersion condition as so called, localized surface plasmon resonance (LSPR) phenomenon, which can be measured by colorimetric detection. Under the optimized condition, the proposed aptasensor provided a linear range of 10 - 300 ppb with a detection limit of 7.74 ppb. The aptasensor is quite simple and the analysis time is in a few minutes without needing of complicated instrument. Moreover, this sensor performed good selectivity that can be applied for chlorpyrifos detection in real sample.

Keywords: Aptamer; Chlorpyrifos; Gold nanoparticle aggregation; Colorimetric detection

# Label-free Electrochemical Immunosensor based on Polyaniline /Poly(acrylic acid) Film for Detection of Human immunoglobulin G

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## Abstract

An label-free electrochemical immunosensor based on polyaniline (PANI)/poly(acrylic acid) (PAA) film on a screen-printed carbon electrode (SPCE) has been developed for the detection of human immunoglobulin G (IgG). An PANI/PAA films on electrode were prepared by the electrochemical polymerization. The PANI/PAA film has the sufficient conductivity and high density surface carboxylic groups, on which can be covalently conjugated with capturing anit-IgG antibodies. Subsequently, the PANI/PAA film-modified SPCE was employed for construction of electrochemical immunosensor for detecting IgG without external or additional redox probe. The electrochemical signal from PANI/PAA film in signal amplification was obtained by differential pulse voltammetry (DPV). Under the optimal condition, the anodic peak current linearly responded to the logarithm of IgG concentration in a linear range from 0.5 to 50 ng mL<sup>-1</sup> and a limit of detection of 0.37 ng mL<sup>-1</sup>. Our proposed immunosensor exhibited satisfactory sensitivity and selectivity.

*Keywords: Label-free electrochemical immunosensor; polyaniline /poly(acrylic acid); Human immunoglobulin G; Differential pulse voltammetry* 

# Hybrid electrocatalytic nanocomposites based on carbon nanotubes/nickel oxide/nafion toward an individual and simultaneous determination of serotonin and dopamine in human serum

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## Abstract

Serotonin (ST) is a monoamine neurotransmitter that helps control emotional system and biological functions such as sleeping, eating, and digesting, its abnormal concentrations are associated with different diseases, such as depression, migraine, bipolar disorder and anxiety. Thus, the development of simple, fast, and sensitive electroanalytical methodologies for the determination of ST is currently needed in clinical and biomedical fields. However, detection of ST is generally interfered with by other compounds in biological samples, such as dopamine (DA). In this work, the electrochemical sensor for the simultaneous determination of ST and DA in human serum was developed. The sensor-based on electrochemically pre-treated materials of hybrid electrocatalytic nanocomposites composed of carbon nanotubes/nickel oxide/carbon black/Nafion modified screen-printed carbon electrode (CNT-NiO-CB/Nafion/SPCE) was proposed for the development of the sensor. A linear calibration graph in the range of 0.08-19.84  $\mu$ M DA and 0.23-14.60  $\mu$ M ST were achieved. An electrochemical sensor exhibits superior analytical performance such as wide linear range, low limit of detection (11 nM ST and 11 nM DA), acceptable reproducibility (RSD = 5.74% ST and 7.74% DA) indicating excellent selectivity, stability, sensitivity along with good recovery range.

Keywords: Serotonin, Dopamine, Electro-catalyst, Electrochemical sensor

# Label-Free Electrochemical Immunosensors Based on Redox Gold Nanoparticles for Simultaneous Detection of Cancer Biomarkers

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## Abstract

In this work, we describe a new multiplexed electrochemical immunosensors based on different redox gold nanoparticles for the simultaneous detection of three tumor markers of breast cancer including mucin1 (MUC1), cancer antigen 15-3 (CA15-3), and human epidermal growth factor receptor 2 (HER2). The device is designed using antibody-conjugated polyethylenimine gold nanoparticles (PEI-AuNPs) containing redox species for simultaneous detection of multiple biomarkers in a single run. The redox-active species, anthraquinone-2-carboxylic acid (AQ), thionine chloride (TH), and AgNO<sub>3</sub> (Ag<sup>+</sup>), are utilized for the detection of MUC1, CA15-3, and HER2, respectively. The PEI-AuNPs in this experiment plays an important role in improving the conductivity and loading of the capture antibodies. Under the optimization, the immunosensor displays good linear relationships in range from 0.10-100 ng mL<sup>-1</sup> for MUC1, 0.10-100 U mL<sup>-1</sup> for CA15-3 and 0.10-100 ng mL<sup>-1</sup> for HER2, respectively. This designed immunosensor offers simple, low cost, fast detection, high selectivity, and good reproducibility. Meanwhile, the immunosensor is successfully applied to detect three tumor markers in blood serum samples with good recoveries, which can be used as diagnostic device for point-of-care testing applications.

Keywords: Breast cancer; Multiplex label-free; Electrochemical immunosensor; Polyethyleneimine-coated gold nanoparticles

# A label-free electrochemical aptasensor using platinum nanoparticles on carboxylated-graphene oxide for

## alpha-fetoprotein detection

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## Abstract

A label-free electrochemical aptasensor for alpha-fetoprotein (AFP) detection was fabricated using carboxylated-graphene oxide (GO-COOH) modified screen-printed carbon electrode (SPCE). Amine functionalized AFP aptamer was used as the bio-recognition element for selective detection of AFP. The GO-COOH modified could increase the surface area and amounts of aptamer immobilized on the electrode. In addition, platinum nanoparticles (PtNPs) were loaded on GO-COOH to improve the electrical conductivity and the signal of electrochemical probe. The interaction between aptamer and AFP resulted in a decrease of current because the layer of binding biomolecules on the modified electrode can affect the electron transfer. The effect of some parameters was studied such as aptamer concentration and incubation time. The currents of redox probe before and after AFP incubation were investigated using a square wave voltammetry. Under the optimum conditions, the developed aptasensor showed a linear range from 3.0–30 ng mL<sup>-1</sup> with a detection limit of 1.5 ng mL<sup>-1</sup>. The aptamer immobilized electrode exhibited high specific binding to AFP molecules with good stability. Moreover, the cost effective aptasensor has simple preparation and detection procedures, and it could be used for determination of AFP in real sample.

Keywords: Alpha-fetoprotein; Aptamer; Electrochemical sensor; Graphene oxide; Platinum nanoparticles

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# Label-Free Electrochemical Immunosensor Based on Methylene Blue adsorbed a 2D Molybdenum disulfide/Graphene Oxide Nanocomposite for Detection of Matrix Metalloproteinase-7

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## Abstract

In this study, methylene blue (MB) adsorption onto 2D molybdenum dioxide (MoS<sub>2</sub>)/graphene oxide (GO) nanocomposites are used to develop the electrochemical immunosensor for the detection of matrix metalloproteinase-7 (MMP-7) cancer biomarker. MB/MoS<sub>2</sub>/GO nanocomposite is modified on the screen-printed carbon electrode (SPCE) to amplify the electrochemical signal in the immunosensor. The modification process of the nanocomposite is monitored by cyclic voltammetry (CV). The nanocomposite platform shows a large specific surface area, high electrical conductivity, good electron transfer property, and good biocompatibility. The differential pulse voltammetry (DPV) is applied to investigate the specific interaction between anti-MMP-7 and MMP-7. Under the optimized condition, the label-free immunosensor exhibits a wide linear range of 0.01-75 ng mL<sup>-1</sup>. The sensor provides a simple platform for low cost, sensitivity, selectivity, good reproducibility, and stability. This device can be applied for early clinical diagnosis of liver and colorectal cancer.

Keywords: Metrix metalloproteinase-7; Nanocomposites; Label-free; Electrochemical immunosensor

# Flow injection amperometric sensor for expedient determination of orthophosphate in soil and water

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#### Abstract

In-line coupling of a flow injection manifold with an amperometric sensor is herein proposed for reliable and expeditious determination of orthophosphate in environmentally relevant samples. The analytical method is based on the in-line formation of 12-molybdophosphate, which is subsequently reduced electrochemically at a glassy carbon electrode. Standard/sample solutions were injected into a stream of 0.1 mol/L potassium chloride and then merged downstream with 0.5% (w/v) molybdate in 2.5% (v/v) sulfuric acid solution to form the measurand, which is 12molybdophosphate complex, in a reaction coil. Electrochemically reduction of the complex at a glassy carbon electrode at 0.20 V vs Ag/AgCl produced electrical current which was directly proportional to the concentration of orthophosphate in the injected solution, and the current was continuously recorded as a transient signal. Linear calibration graphs spanned the ranges of 10-100 µg L<sup>-1</sup> P, 0.1-1.0 mg L<sup>-1</sup> P, and 1-10 mg L<sup>-1</sup> P based on selected experimental conditions with a limit of detection of 3 µg L<sup>-1</sup> P. Relative standard deviations for 11 replicate injections of both 0.5 mg L<sup>-1</sup>P and 5 mg L<sup>-1</sup> P were 0.8%. A sample throughput of 40-60 h<sup>-1</sup> was achieved. The proposed method was validated by analysis of certified reference materials of soil and water and was successfully applied to real-life environmental samples as well. Amperometric detection might be a straightforward alternative to the conventional spectrophotometric phosphomolybdenum blue method. Besides, the analytical method does not suffer from interferences such as particulates and coloured substances, and refractive index effect (Schlieren's effect), and can tolerate concentrations of silicate and chloride as high as 1 g L<sup>-1</sup> and 40 g L<sup>-1</sup>, respectively.

Keywords: Flow injection; Amperometric sensor; Orthophosphate; Soil; Water

# Screening of Coffee Impurity Using a Homemade NIR Sensor System

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## Abstract

Coffee is among the economically important beverage plants. In each year, a great amount of this agricultural product has been world-widely traded. For this reason, inspection of coffee bean quality to match the desired level of the customers is a crucial step. Near infrared (NIR) spectroscopy is a non-destructive detection based on the measurement of the electromagnetic radiation in the range between 750-2500 nm. With the detection using a reflectance mode, a number of solid samples can be easily and quickly detected making NIR preferably suitable for the measurement of the agricultural products especially coffee. In this research, NIR spectra of green coffee been samples were recorded using a homemade NIR system. The Arabica coffee samples were obtained from Chiang Rai province in the north part of Thailand. Three types of impurity were tested, including physically broken (11), insect damaged (I2) and dried cherry (I3). The coffee samples were prepared to have 0, 3, 5, 7, 10, 15, ...., 50 %w/w of the impurity resulting in different 13 levels for each impurity test. Therefore, with the three types of the impurity test, a total of 39 contaminated coffee samples were obtained where the NIR spectra were recorded with 20 replicates to provide the average spectrum of each sample. The spectral data were exploratorily analyzed using principal component analysis (PCA) to investigate the difference among the samples. After that, partial least square (PLS) calibration models were established to estimate the impurity levels of the coffee samples. From the PCA score plot, the developed NIR sensor system could be well employed to identify the contaminated coffee. The PLS models could be used to accurately quantify the impurity level with acceptable degree of error demonstrating that the developed NIR sensor system could be used for screening the impurity in the coffee bean products.

Keywords: Coffee; Homemade NIR sensor; Quality control; Principal component analysis (PCA)

# Fabrication of Low-Cost NIR Sensor for Detection of Agricultural Products

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## Abstract

Near infrared (NIR) spectroscopy is an analytical technique that detects the absorbance in the region of 700–2500 nm, providing spectral information from the overtone and combination bands of the infrared (IR) absorptions. The detection of NIR has several advantages such as requiring less detection time and providing more specific information of the chemical samples. In addition, this detection technique is the non-destructive method with less or no sample preparation which is suitable for the measurement of various agricultural samples such as coffee, rice, soy and mung bean. However, at the present, the commercial NIR spectrometer is relatively expensive and most of them require specific condition for the optimal measurement such as large space and stability in the experimental conditions. In this research, a portable NIR spectrometer was developed. The use of a single element detector and inexpensive light sources (LEDs) allowed for the reduction in the price of the developed NIR spectrometer with a light weight of approximately 2 kg. The NIR spectra were recorded in the region of 900 to 1700 nm. To demonstrate the performance of the developed NIR spectra were exploratorily analyzed using principal component analysis (PCA). It was found that the developed NIR spectra were exploratorily analyzed using principal component analysis (PCA). It was found that the developed NIR spectra were exploratorily analyzed using principal component analysis (PCA). It was found that the developed NIR spectra were or orbined with PCA analysis can successfully characterize the difference among the studied samples as well as their combinations. In addition, the potential tendency to classify coffee according their origins and to predict the percentage of Thai jasmine rice adulteration were presented.

Keywords: Low-Cost NIR sensor; Chemometrics; Agricultural products; Principal component analysis

# SnO<sub>2</sub> Quantum Dots Prepared by Electrochemical Process for Perovskite Solar Cell Application

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## Abstract

Tin dioxide Quantum dot  $(\text{SnO}_2 \text{ QDs})$  can be applied as electron-transporting layer for efficiency enhancement of conventional perovskite solar cell (PSCs). Here, SnO<sub>2</sub> QDs are synthesized by electrochemical process and investigated for the perovskite solar cell application. The electrochemical process is performed in distilled water and in citric acid (0.1 M) at applied voltage of 6V by using tin foil as electrodes. It is found that the obtained solutions for all conditions show clear color but a Tyndall effect can be observed when shining laser light in the solutions. The observed Tyndall effect indicates a light scattering of the fine particles dispersed in the solutions as a colloid. The hydrodynamic size of colloid from dynamic light scattering (DLS) technique is calculated to be  $62.9\pm0.9$ nm for water solution and  $123.3\pm5.6$  nm for citric acid solution. From TEM images, the diameter of colloid particles is  $3.35\pm0.15$  nm for water solution and  $3.14\pm0.11$  nm for citric acid solution suggesting quantum property. These SnO<sub>2</sub> QDs are used in the conventional structure of perovskite solar cells. The photo conversion efficiency (PCE) of solar cells based on SnO<sub>2</sub> QDs is 13.59% for water solution and 13.24% for citric acid solution compared to 12.63%for the reference cell which is based on SnO<sub>2</sub> nanoparticles. The enhancement of PCE by using QDs may be explained by the smaller size of QDs. Therefore, SnO<sub>2</sub> QDs can be successfully used for efficiency enhancement of PSCs.

Keywords: SnO<sub>2</sub>; Quantum dot; Electrochemical process; Perovskite solar cells

# Low-temperature Carbon Electrode with Solvent Exchange Process for Planar Hybrid Perovskite Solar Cells

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## Abstract

Among the next-generation photovoltaic technologies, perovskite solar cells (PSCs) have attracted significant attention and interest. Hybrid organic–inorganic metal halide perovskites have recently emerged as excellent light absorbing materials for low-cost and high-throughput solar energy harvesting technology. The reported power conversion efficiency (PCE) of the perovskite device has progress significantly from 3.8% in 2009 to over 24.2% presently, in laboratory-scale devices. The perovskite materials and the carrier (electron and hole) transporting layers in PSCs involve an effective role in boosting their efficiency and long-time stability. However, PSCs typically use high-vacuum processed noble metal as a cathode, which is considerably costly. Furthermore, this process results in dramatic performance degradation after halide ion migration, continuous bending and thermal stress. Therefore, replacing metal electrodes with carbon electrodes is a promising way to address the stability issues of PSCs. This is because carbon materials possess unique qualities, such as; low cost, high stability, good conductivity and inherent water resistance. In contrast, some solvents in commercial carbon paste damage the structure of perovskite film. Herein, low temperature carbon films have been successfully prepared with solvent exchange process via time optimization. The carbon films were then applied as working electrodes in PSCs based on TiO<sub>2</sub> nanoparticles electron transporting layer. By optimizing time toward solvent exchange process, an excellent power conversion efficiency (PCE) of these devices has improved up to 12.2%

Keywords: Carbon electrode; Low temperature; Perovskite solar cells; Solvent exchange; TiO<sub>2</sub> nanoparticles

# Slot-die-coated Zinc Tin Oxide Film for Carbon-based Methylamonium-free Perovskite Solar Cells

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## Abstract

On a laboratory scale, spin coating technique is commonly used for depositing an electron transporting layer (ETL) in perovskite solar cells (PSCs). However, this technique is unsuitable for scaling up production. To enable a large-scale deposition, slot-die coating, which is a promising scalable technique and low operational cost processing, has been proposed for depositing thin and uniform films across large areas. In this work, scalable slot-die coating processes of zinc tin oxide (ZTO) as ETLs have been illustrated for carbon-base methylamonium-free PSCs. ZTO was selected because of its excellent optical and electronic properties such as high electrical conductivity, high electron mobility and optical property. The slot-die coated ZTO thin films was prepared from a homemade slot-die bead. The morphology and photovoltaic performance of PSCs using these films are reported. The device using slot-die coated ZTO films exhibits the best device power conversion efficiency of 7.85%, which is comparable to that of the device using spin-coated ZTO film. This work demonstrates the potential of slot-die coating technique to replace the conventional spin-coating method for fabricating high efficiency and scalable PSCs.

Keywords: Slot-die coating technique; perovskite solar cells; zinc tin oxide; scale up

# Nitrogen-doped TiO<sub>2</sub> via Thermal Hydrolysis and Photocatalytic Performance in Selective Oxidation Under Visible Light

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## Abstract

TiO<sub>2</sub> is a promising photocatalysts in photoreaction due to its exceptional stability in various conditions, high capability to generate charge carrier and low cost. Unfortunately, the photocatalytic performance of TiO<sub>2</sub> is limited by its wide band gap energy (3.1 eV) which inhibits its photoactivity under visible light region. Doping TiO<sub>2</sub> with nitrogen is one of alternative ways to overcome this challenge because a new band level above the valence band of TiO<sub>2</sub> is created, resulting in extended light absorption range, hence an enhanced photocatalytic activity under visible light. Nitrogen-doped (N-doped) TiO<sub>2</sub> has been synthesized via simple thermal hydrolysis of TiOSO<sub>4</sub> at room temperature using NH<sub>4</sub>OH as a nitrogen source and a precipitating agent. Compared with typical titanium alkoxide precursors, the inorganic titanium precursor is low-cost and non-sensitive to air and moisture, thus making the material preparation process cost-effective and easy to handle. The existence of nitrogen in TiO<sub>2</sub> exhibits better visible-light-driven photoactivity (>85% conversion and >95% product selectivity) in the benzyl alcohol and benzylamine oxidation than the undoped TiO<sub>2</sub>, clearly indicating the advantage of nitrogen doping. Therefore, the synthesized TiO<sub>2</sub> by the facile thermal hydrolysis route and adjusting pH by NH<sub>4</sub>OH is one of the promising methods to create N-doped TiO<sub>2</sub> material in order to apply as a useable photocatalyst in the photoconversion of organic reaction.

Keyword: Photocatalyst; Nitrogen doping; Oxidation; Benzylamine; Benzyl alcohol

# Effect of humidity on physical property of TiO<sub>2</sub> nanoparticulate films prepared by facile sparking process

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## Abstract

In our previous works, some metal oxide nanoparticulate (NP) films were successfully deposited on glass substrate by a simple and low-cost sparking process under atmospheric pressure. Some techniques have been resolved and developed in the process to improve the film's properties, such as electroplating, electric and magnetic field assisted in the process. In this work, we aim to study the effect of humidity on physical property of titanium dioxide  $(TiO_2)$  NP films. The high DC voltage of 4.0 kV and limited current of 3 mA were applied and sparked at Ti tips which were used as a starting materials. A very small particles in the range of <20 nm was then deposited on the glass substrate to form the NP films for 30 min under various humidity. The NP films were characterized in the terms of physical property. Interestingly, the effect of humidity on morphology will be reported and discussed.

Keywords: TiO<sub>2</sub>, sparking process, nanoparticulate, films, humidity

# Development of Bismuth Oxyhalide Solid Solutions

# for Selective Organic Synthesis

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## Abstract

Bismuth oxychloride (BiOCl) is regarded as an interesting photocatalyst for several photocatalytic applications. However, the performance of BiOCl is limited due to its wide band gap energy, so this material shows rather low activity under visible light. In this work, the photocatalytic performance of BiOCl toward the selective oxidation of benzylamine to N-benzylidenebenzylamine was improved by forming solid solution with iodide to reduce the band gap energy and improve activity under visible light. Results from X-ray diffraction spectroscopy (XRD) and scanning electron spectroscopy (SEM) studies indicated that sheet-like BiOIxCl1-x solid solutions were successfully synthesized. The BiOI0.1Cl0.9 sample provided the highest benzylamine conversion and N-benzylidenebenzylamine yield of ca. 81% and 74% within 4 h irradiation due to a narrower band gap energy and higher separation efficiency of photoexcited electrons and holes under visible-light illumination which were confirmed by UV-visible diffuse reflectance spectroscopy (UV–vis DRS), photocurrent response and electron paramagnetic resonance (EPR) and active species quenching experiments indicated that singlet oxygen (102), molecular oxygen and electron were the main active species.

Keywords: BiOI<sub>x</sub>Cl<sub>1-x</sub>; Benzylamine; Photocatalyst; Amine coupling; Solid solution

# Thermoelectric Properties of Copper Iodide Transparent Thin Films

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## Abstract

Copper Iodide (CuI) transparent thin films were prepared on glass slide substrates by using dc magnetron sputtering method from the Cu target. The as-deposited Cu thin film was dipped in Iodine solutions for 15, 30 and 60 s, and then dried by the hot blower. Microstructure, morphology and thermoelectric properties were studied by using X-ray diffraction (XRD) techniques, scanning electron microscopy (SEM) and ZEM-3 method, respectively. The optical property of the film samples was determined from measured percent transmittance using a UV-visible spectrophotometer. The results showed that the microstructure of as-deposited thin film showed the phase of Cu at (111) peak, while the thin film as-dipped in iodine solution for 15, 30 and 60s contained the phase of Cu at (111), (200), (220), (311) and (222) reflections. The surface morphology of the thin films showed the crystallite as agreed with the XRD results. Thermoelectric properties measurement results of CuI thin film at room temperature had the power factor of  $1.471 \times 10^{-5}$ ,  $1.749 \times 10^{-5}$  and  $1.748 \times 10^{-5}$  W m<sup>-1</sup> K<sup>-2</sup> for the thin film as-dipped in iodine solution for 15, 30 and 60 s, respectively.

Keywords: Thermoelectric thin film; copper iodide; dc magnetron sputtering

# A combination of point defects and nano-sized grains to minimize lattice thermal conductivity of Sn and Se co-doped CoSb<sub>3</sub> via mixed ball milling and SPS

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## Abstract

Effective strategies to minimize main thermal conductivity pathways in skutterudite materials are creating point defects along with turning grain sizes down to nanoscales. Here, we utilized these strategies by synthesizing Sn and Se co-doped CoSb<sub>3</sub> materials through the combination of mixed ball milling and spark plasma sintering techniques. Their phases, microstructure, and thermoelectric properties were investigated under the variation of Sn and Se content in CoSb<sub>3</sub> bulk samples. The experimental results from the X-ray diffraction, electron microscopes, and thermoelectric properties reveal that the existence of point defects by co-doping and intense phonon scattering by nano-grains dramatically reduced the lattice thermal conductivity down to 2.4 W/mK at 25°C. Extra doping of Sn and Se in CoSb<sub>3</sub> could further minimize the lattice thermal conductivity. The lowest value is 1.8 W/mK at 25°C for  $CoSb_{2.70}Sn_{0.150}Se_{0.150}$  composition which is lower than the pure CoSb<sub>3</sub> more than 100%. Moreover, the increment of Sn and Se contents also gradually increased the electrical conductivity of doped samples in the whole measurement temperature contrary to the negative Seebeck coefficient trend. Although the electrical conductivity of doped samples was increased and the Seebeck coefficient values were high, the increment of electrical conductivity was still low as well as the substantial reduction of the Seebeck coefficient at high measurement temperature resulting in low power factors and ZT values. The highest power factor of 880 µW/mK<sup>2</sup> was found at 250°C for CoSb<sub>2.65</sub>Sn<sub>0.175</sub>Se<sub>0.175</sub> composition which dominates the highest ZT value of 0.29 at 300°C among the Sn and Se co-doped samples. Ball milling under dry and wet conditions not only allowed a longer milling time resulting from non-sticking of powder in a milling jar but also generated a small amount of porous which was a part of the reduction in the lattice thermal conductivity.

Keywords: Co-doped CoSb<sub>3</sub>; Microstructure; Thermoelectric properties; semiconductors; Point defects

## Thermoelectric Properties of Pr-substituted YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-y</sub> Ceramics

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## Abstract

The thermoelectric properties of Pr-substituted YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-y</sub> ceramics were investigated from room temperature to 773 K. The ceramics of  $(Pr_xY_{1-x})Ba_2Cu_3O_{7-y}$  (where x = 0-1) were successfully prepared by a conventional sintering method at 920 °C for 12 h in the normal air atmosphere. Phase identification was carried out by X-ray diffraction (XRD) technique. The microstructure of samples was studied using scanning electron microscopy (SEM). The thermogravimetric and differential thermal analysis (TG/DTA) were used to examine the thermal analysis of samples. In addition, Seebeck coefficient, electrical resistivity and thermal conductivity were investigated and discussed. The average phonon velocity was measured using Pulse-echo technique at room temperature to discuss the substitution effect on the lattice thermal conductivity. It was observed that the substitution of Pr in YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-y</sub> improved the Seebeck coefficient ( $\alpha$ ) and showed high figure of merit (*ZT*) at wide temperature range from 300 to 773 K, compared to pure YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-y</sub> and other thermoelectric oxide ceramics. These results suggested that Pr substitution is a promising approach for improving thermoelectric properties of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-y</sub> system and these compounds show high potential as a novel material for intermediate and high temperature thermoelectric applications.

Keywords: Pr-substitued YBCO; YBCO; PrBCO; Thermoelectric

# Carbon Conductive Paint For Manufacturing Energy Storage Devices Using Integration Of 3D Printing Techniques

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### Abstract

Energy storage has become a main focus of world powers and the scientific community. More efficient energy storage devices have attracted great attention. One such device, the supercapacitor, has increased in use significantly over the last decade and has the potential to facilitate significant advances in energy storage. The fabrication methods of supercapacitors have been intensively researched and have successfully developed various techniques, such as dip coating, doctor blade coating, screen printing, and inkjet printing. More recently, 3D printing techniques have also been developed and used to manufacture supercapacitors. The purpose of this research is to determine the optimal concentration of carbon conductive paint for the fabrication of current collectors and electrodes in a small cuboidal 3D-printed supercapacitor. The carbon conductive paint, CCP, used in this study was Bare Conductive Electric Paint. This CCP offers the possibility of creating electrically conductive areas onto objects using 3D printing. The CCP is a water-based material which is non-toxic, organic, solvent free, and may be used for making low-current circuits and sensors. Different concentrations of CCP were prepared in order to create 3D-printed supercapacitors (EDLCs). Several 3D-printed energy storage supercapacitors were made and their electrochemical performances were characterised. Different concentrations of carbon conductive paint provided different electrical performance and printability, i.e. the suitable activated carbon (AC) slurry for 3D printing should not be too viscous in order to extrude easily in the system. The AC slurry made with 3.0 g CCP and 30 g distilled water was found to deliver the best electrical performance. Please limit your abstract to 300 words.

Keywords: Carbon Conductive Paint, Fused Deposition Modelling, Paste Extrusion System, Electrical Performance, Energy Storage Devices.

# Aqueous asymmetric supercapacitors based on rGO/PANI/MnO<sub>2</sub> anodes and rGO/MoS<sub>2</sub>/CuO-NiO cathodes

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### Abstract

Aqueous electrochemical energy storage devices, such as supercapacitors and batteries, offer potential advantages compared to devices based on organic electrolytes, including environmental-friendliness/low toxicity, better safety and lower cost. In this work, we demonstrate an aqueous asymmetric supercapacitor based on composites of reduced graphene oxide, manganese dioxide and poly-aniline (rGO/MnO<sub>2</sub>/PANI) as negative electrode as well as rGO, molybdenum disulfide and copper/nickel mixed oxide particles (rGO/MoS<sub>2</sub>/CuO-NiO) cathodes with KOH as electrolyte. GO was prepared by modified Hummers' method, PANI was interfacially polymerized and rGO/PANI/MnO<sub>2</sub> composite was produced by hydrothermal process. For rGO/MoS<sub>2</sub> and rGO/MoS<sub>2</sub>/CuO-NiO composites, a combination of wet-chemical and hydrothermal processes were used. Electrodes were fabricated by screen printing material onto carbon paper current collectors. SEM and XRD showed successful composite preparation. CR2032 coin cell asymmetric supercapacitors were constructed and examined by cyclic voltammetry and galvanostatic charge-discharge measurements. The developed ASCs delivered specific capacitance of up to 174.9 F g<sup>-1</sup> and energy and power densities of 35 Wh kg<sup>-1</sup> and 4.4 kW kg<sup>-1</sup> at a current of 1 A g<sup>-1</sup>.

Keywords: energy storage; asymmetric supercapacitors; aqueous electrolytes; graphene oxide; graphene composites

# Synthesis and Characterization of SbSi on N-doped Graphene Composites as Anode Materials for Li-ion Batteries

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## Abstract

Li-ion batteries have begun commercialized technologies for modern and future world, but commercial batteries using graphite still have low specific capacity and concerned safety issue. Sb-Si composites have been tended to synthesize as anode materials which can be a solution for the mentioned problems. This work describes the methods and characterization of Sb and Si on N-doped graphene (SbSi/NrGO) composite materials by simple chemical routes, magnesiothermic reduction and NaBH<sub>4</sub> reduction. Si used in this study was obtained by recrystallization of SiO<sub>2</sub> from rice husk, waste from agricultural process. All prepared samples were characterized phases, particle distribution and morphologies by XRD, SEM equipped with EDS and TEM, respectively. The XRD patterns showed phases of c-Si, t-Si, Sb and shifted peaks of expanded C which causes by NrGO synthesis. EDS mappings and TEM images displayed distribution of Si and Sb particles of NrGO surface. Morphological identifications can be observed in TEM images that mainly compose of Si, Sb and C for all samples, but the residual SiO<sub>2</sub> can be found in some prepared samples. All compositions found in XRD patterns were confirmed their phases by SAED patterns in TEM technique. The method in this report is appropriate to prepare SbSi/NrGO composites with high Si content.

Keywords: Si; Sb; Li-ion Batteries (LIBs); Graphene; Anode Materials

# Two-Dimensional FTIR Correlation Spectroscopy for Investigation of Self-folding Structure of Polyampholytes

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## Abstract

Chitosan polyampholytes (CPAs) have been widely used in various applications, especially in biomedical, cosmetics, catalyst, and environmental fields. CPAs possess both positive and negative charges in their backbone, which can cause self-folding of the chains to form a globular shape. Therefore, extensive understanding of the complex CPAs structure for proper characterization techniques becomes a priority and essential work. Although Fourier transform infrared (FTIR) spectroscopy is a powerful technique for characterization of polymers, it has not been fully exploited for investigation of self-folding structures of polyampholytes. In this study, the self-folding structures of CPAs are deeply analyzed by FTIR spectroscopy in both ATR and transmission modes. 2D FTIR correlation spectroscopy is applied to gain insights into the structure formation mechanisms. In addition, X-ray photoelectron spectroscopy (XPS), transmission electron microscopy (TEM), and dynamic light scattering are employed in the characterization. FTIR results are in good agreement with TEM observations for self-folding structure of CPAs to form the globular shape structure. FTIR results also provide detailed information about functional group distributions, i.e., a high distribution of quaternized groups on the globular's surface, whereas carboxylate in hydrogen bonds and electrostatic interactions are densely located in the bulk structure of CPAs. The results are also confirmed by those observed from XPS and zeta potential results. In addition, trapped water molecules and impurities are observed in the bulk structure of CPAs by transmission FTIR measurements. The insights into these structure formation mechanisms are essential in assessments of the materials applications.

Keywords: FTIR; 2D FTIR; polyampholytes; self-folding structure; functional group distribution.

# Ring-Opening Polymerization of ε-Caprolactone Using a Soluble Tin(II) *n*-Butoxide-L-lactide Macroinitiator

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## Abstract

The novel soluble tin(II) n-butoxide-L-lactide macroinitiator ((Sn(OnBu)<sub>2</sub>-LL) was synthesized from ringopening polymerization (ROP) of L-lactide initiated by 4.0 mol % and 8.0 mol % of tin(II)-n-butoxide (Sn(OnBu)2) in order to improve the aggregation of  $Sn(OnBu)_2$ . Chemical structure of SnLL4 and SnLL8 macroinitiators were characterized by infrared spectroscopy (IR) and proton nuclear magnetic resonance spectroscopy (<sup>1</sup>H-NMR), then their thermal analysis were studied by differential scanning calorimetry (DSC) and thermogravimetric analyser (TGA). The Kinetic study of ROP of  $\varepsilon$ -caprolactone ( $\varepsilon$ -CL) initiated by a synthetic Sn(OnBu)<sub>2</sub>-LL using non-isothermal DSC and the activation energy (Ea) was calculated by two iso-conversional methods of Friedman and Kissinger-Akahira-Sunose. The Sn-O bond active species was found in both SnLL4 and SnLL8 macroinitiators structure. The longer SnLL4 gave higher crystallization temperature ( $T_{c}$ ), melting temperature ( $T_m$ ) and the exotherm range of ROP of  $\varepsilon$ -CL at the same heating rate used than the shorter SnLL8. Whereas SnLL8 gave faster reaction rate and lower  $E_a(E_a \approx 32.4 - 48.1 \text{ kJ/mol})$ than SnLL4 ( $E_a \approx 54.9 - 98.1 \text{ kJ/mol}$ ). Increasing the LL chain length of macroinitiator resulted in decrease the ROP rate of E-CL. The efficiency for initiating the bulk ROP of E-CL at 150 °C and 48 hrs of SnLL4 and SnLL8 macroinitiators were compared with another initiating systems such as stannous octoate (Sn(Oct)<sub>2</sub>), Sn(Oct)<sub>2</sub>/n-butanol and Sn(OnBu)<sub>2</sub>. The result showed that SnLL4 can controlled the synthesis of poly(*e*-caprolactone) (PCL) more efficiently, when the molecular weight of PCLs were decreased with increasing SnLL4 concentrations with high % yield (> 77 %) and % conversion (> 88 %). It is concluded that the novel  $Sn(OnBu)_2$ -LL macroinitiator has the potential for use as an effective initiator for ROP of cyclic esters and easy to handle even use and store in an atmosphere.

*Keywords:* Soluble tin(II) n-butoxide-L-lactide macroinitaitor; ε-Caprolactone; Ring-opening polymerization; *Kinetic study*; Non-isothermal differential scanning calorimetry

# Solvent-infusion cold drawing of poly(L-lactide) filaments

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## Abstract

Post treatments of polymeric products after fabrication, especially improvement of their crystallinity, are commonly applied to further enhance properties of the materials. In this study, a cold drawing process in various solvents is developed for poly(L-lactide) (PLLA) filaments, and its solvent-infusion mechanism is examined. Multiple necking behavior was observed when the filaments were drawn in water and ethanol, compared to a single-neck deformation when the experiment was conducted in air. The number of necks was also dependent on the drawing conditions, in which the number of necks decreased with an increase in the draw ratio (DR). The number of necks of the filaments drawn in water and ethanol lead to a decrease in the yield and drawing stress on the filaments. The results also indicate that ethanol is effectively infused into the PLLA filaments, leading to further chain arrangements. The process has high potential for promoting the crystallization of filament at room temperature conditions, leading to enhancement in the mechanical properties.

Keywords: Poly(L-lactide), Solvent infusion, Cold drawing, Filaments, Post treatment

# Fabrication of Poly(L-lactic acid)-Based Colorimetric Humidity Sensors by Printing Technology

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#### Abstract

Nowadays, biodegradable polymer development for smart packaging has been interested in using more environment-friendly materials such as poly(L-lactic acid) (PLA) due to their biodegradability. However, some properties of PLA such as its brittleness, poor thermal stability and hydrophobicity limit its applications. In this research work, polymer-based humidity sensors have been fabricated from blends of PLA with poly(ethylene oxide) (PEO) via melt mixing to improve the properties of PLA. Three-dimensional (3-D) printing technology was used to fabricate biodegradable substrates using 30% w/ v PLA/PEO blend solutions in chloroform. Inkjet printing successfully printed cobalt chloride (CoCl<sub>2</sub>) solution as the sensing colorimetric humidity indicator onto the 3-D substrate. The various properties of the PLA/PEO blend relevant to its use as a 3-D substrate were determined by a combination of tensile testing, differential scanning calorimetry, thermogravimetric analysis and contact angle measurements. In terms of sensitivity, the cobalt chloride indicator demonstrated fast sensing times of within 60 secs for color changes ranging from blue to pink when exposed to moisture (% RH = 60-70), as confirmed by image analysis and total color difference (TCD) as a function of time. The fabrication of a colorimetric humidity sensor based on a biodegradable polymer by 3-D and inkjet printing technology represents a new challenge to make the most use of these new technologies with a non-toxic polymer to detect moisture and be safe for the environment.

Keywords: Poly(L-lactic acid); poly(ethylene oxide); colorimetric humidity sensor; 3-D printing; inkjet printing

# Assessment of Rheological Properties in Relation to Fabrication Conditions of Medical-grade Poly(L-lactide-*co*-glycolide) for Use as Resorbable Bone Fixation Devices

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#### Abstract

Nowadays, resorbable polymer bone fixation devices can be used as alternatives to metal bone fixation devices in orthopedic surgery for bone repair. One of the most promising materials is poly(L-lactide-*co*-glycolide) (PLG) due to its biocompatibility in the human body. However, PLG's main drawback is its low melt strength which negatively influences its ease of fabrication. In this study, medical-grade PLG 80:20 (mole %) was synthesized according to ASTM F1925-17 specifications and its melt strength improved using Joncryl<sup>®</sup> ADR-4368 as a chain extender. Rheological and thermal properties were studied in order to understand the melting characteristics and to determine suitable fabrication conditions. The PLG was then fabricated in the form of bone screws by micro-injection molding at injection temperatures from 170 to 190 °C. The results showed that the PLG resin with 1.0 wt% of Joncryl<sup>®</sup> ADR-4386 had an increased initial thermal degradation temperature of 280 °C compared to the 270 °C of PLG alone. The results of oscillatory rheology testing indicated that the PLG melt exhibited a liquid-like state characteristic of a viscoelastic material. In addition, the chain-extended PLG showed remarkable increases in both zero-shear viscosity and thermal stability. The PLG bone screws fabricated at 190 °C demonstrated mechanical properties comparable to those of commercial PLG bone screws. These results indicate that the fabricated PLG bone screws have the potential to be used as low-stress implantable bone fracture fixation devices.

Keywords: Poly(L-lactide-co-glycolide); resorbable bone fixation devices; melt rheology; micro-injection molding

# Effect of Rheological Properties on the Melt Extrusion of Poly(Llactide-*co-\varepsilon*-caprolactone) Monofilament Absorbable Surgical Sutures

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## Abstract

In the production of poly(L-lactide-*co-e*-caprolactone) (PLC) monofilament sutures by single-screw melt extrusion, rheology has been used extensively to understand the viscoelastic behaviour of the polymer since its rheology is critical for producing identical products from run to run. In this work, the rheological properties of the PLC melt were studied in both oscillatory and steady shear testing modes in order to determine how temperature and shear rate can be tuned to tailor the melt behaviour for melt extrusion. The appropriate combination of temperature and shear rate in an extruder is essential for controlling the polymer's melt behaviour and achieving good material properties by determining the polymer's optimum "processing window". The results indicated that the PLC copolymer in the melt state is pseudoplastic due to the linearity of shear viscosity at low shear rate. Comparisons were made with when triphenyl phosphine (TPP) was added as a chain extender and with a commercial PLC. The results showed that the commercial PLC had maximum values of elastic modulus (G'), viscous modulus (G'') and complex viscosity ( $\eta^*$ ) in a stress sweep of 4.47 kPa, 19.7 kPa and 3.2 kPa respectively. These values were higher than those of the synthesized PLC and PLC with TPP. However, the addition of TPP could increase the length of the linear region of thermal stability. The tensile properties of the PLC fibers were determined using a universal testing machine. The maximum tensile strengths of the commercial PLC, synthesized PLC and PLC with TPP fibers were found to be 322.3, 169.2 and 92.2 MPa respectively.

Keywords: Poly(L-lactide-co- $\varepsilon$ -caprolactone), melt extrusion, rheological properties

# Transparent Cellulose Nanofiber Reinforced Composites with Superior Performances for Electronic Applications

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## Abstract

Flexible and transparent composite substrates have been greatly studied to replace glass. Here, we successfully prepared tough and transparent composites reinforced with cellulose nanofibers. Thanks to superior properties of cellulose nanofibers, the composites showed outstanding performances. Similar thermal expansion to glass  $(7 - 10 \text{ ppm K}^{-1})$  was observed for the composites, and more than 90 % of light could pass through the composite films. These superior performances of the cellulose nanofiber reinforced composites also come with high mechanical properties and flexibility. With increasing a concentration of cellulose nanofibers, tensile strength, modulus and toughness of the composite films increased considerably. The prepared composites in this work could be used as a promising candidate for flexible optical electronic applications.



Fig. 1 Flexibility of the transparent cellulose nanofiber reinforced composite.

Keywords: Transparent; Composite; Cellulose nanofiber; Mechanical properties; Electrical applications

# A comparison of thermal and functional group characterization of cellulose between sugarcane bagasse and hemp

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#### Abstract

This research has focused on the comparison of properties between cellulose nanofibrils made from sugarcane bagasse and hemp: thermal and functional group analysis. In this work, cellulose was extracted from sugarcane bagasse and hemp using chemical treatment with sulfuric acid, sodium hydroxide and hydrogen peroxide. Thermal analysis was studied by using Differential Scanning Calorimeter (DSC), and functional group characterization was investigated by using both Fourier transform infrared spectrophotometer (FTIR) and X-ray diffraction (XRD). The result of FTIR showed that the evidence of hemicellulose and lignin from sugarcane bagasse and hemp was removed by chemical treatment. In addition, the result of XRD showed that crystalline structures of both cellulose nanofibrils were in a form of cellulose  $I_{\beta}$  structure. It was also found that the treated cellulose by chemical treatment had a higher crystallinity than untreated cellulose.

Keywords: cellulose; chemical treatment; characterization

# Effects of Fabrication Method on Ionic Conductivity, Crystallinity and Thermal Properties of PEO-LiCF<sub>3</sub>SO<sub>3</sub>-HNTs Composite SPE

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#### Abstract

The composites of solid polymer electrolyte (SPE) membranes were fabricated by two different methods, hot-pressing and solution casting. The ionic conductivity of SPE which was fabricated by hot-pressing method showed higher ionic conductivity than that of solution casting. The halloysite nanotubes (HNTs) were added at 1,5 and 10 wt% into the SPE membranes to improve the ionic conductivity of PEO-20%LiCF<sub>3</sub>SO<sub>3</sub>. The ionic conductivity, crystallinity and thermal properties of SPEs were investigated by electrochemical impedance spectroscopy (EIS), x-ray diffraction (XRD) and differential scanning calorimetry (DSC), respectively. The morphology of composites was investigated by scanning electron microscope (SEM) images. The highest ionic conductivity at room temperature of  $5.151 \times 10^{-5}$  S.cm<sup>-1</sup> was found in the composite that contained 1 wt% of HNT that fabricated by the hot-pressing method.

Keywords: Solid Polymer Electrolyte; Hot-pressing; Solution Casting; Halloysite nanotube; Poly (ethylene oxide)

# Fabrication and Properties of Hollow Poly(lactic acid-co-glycidyl methacrylate) Particles via Phase Inversion Emulsification

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## Abstract

Overexposure to ultraviolet (UV) light can deeply cause severe negative impacts to human's health. UVshielding materials have been effectively used in personal care products to cope with this serious issue. Due to their excellent light scattering characteristics and low density, hollow polymeric particles have attracted vast attention for use in this application. In this work, hollow particles of poly(lactic acid-co-glycidyl methacrylate) (P(LA-co-GMA)) are prepared via a phase inversion emulsification process. As the degradable and biocompatible copolymer contains an abundant of unsaturated functional groups, this can be crosslinked by employing either thermal- or light-curing reactions. The influence of polyvinyl alcohol (PVA) stabilizing agent, on colloidal stability, particle size and degree of hollow-structure formation was investigated. During the reaction, the movement of PVA from the oil droplet to the oil–water interface can entangle the crosslinked P(LA-co-GMA) copolymer, resulting hollow structure formation. By using 3 wt% PVA, hollow P(LA-co-GMA) particles, with average size of approximately 0.7  $\mu$ m, monodispersity, and good colloidal stability were obtained. The as-prepared hollow P(LA-co-GMA) particles (5 wt%) were then applied as UV-shielding agent in PVA film. The films exhibit high UV-shielding capabilities. The results from cytotoxicity tests indicated that the hollow particles had high cell viability (>90%), when high amount of the materials was employed (up to 200 µg/mL). The materials have high potential for use as UV-shielding filler in cosmetic applications.

*Keywords: Hollow polymeric particles; Poly(lactic acid-co-glycidyl methacrylate); Degradable polymer, UV shielding, Cosmetic* 

# Preparation, Characterization and Property Testing of Biocomposites of PLA and Modified Longan Shell Powder

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#### Abstract

In this research, preparation, characterization and property testing of biocomposites of poly(lactic acid) (PLA) and modified longan shell powder were studied. Longan shell powder, which is the waste from agriculture in the north of Thailand, was modified by alkaline treatment, silane treatment and alkaline-silane treatment. The surface morphology of modified powder was investigated by using scanning electron microscope and optical microscope. The chemical composition of the modified powder was characterized by a Fourier transform infrared spectroscopy. The spectra showed that hemicelluloses and lignin can be extracted by alkaline and silane treatment. The biocomposites composed of PLA and different amounts of unmodified or modified powder (0%, 10%, 20% and 40% wt.) were fabricated into thin sheets by compression molding. The mechanical properties, in terms of hardness, were studied. It was found that the modification and adding of the powder affect directly to the hardness of the composites. It was in the range of 12 - 20 HV. The thermal properties determined from differential scanning calorimetry and thermogravimetric analysis confirmed that the modification decreased the glass transition temperature and degradation temperature of the biocomposites. Moreover, the wettability of the surface was evaluated to determine the hydrophilicity. It showed that the modifications increased the wettability of the samples. It can be concluded that the biocomposites of PLA and modified longan shell powder can be successfully prepared and controlled the properties by varying the amount of longan shell powder and modification method. However, further investigations have to be carried out to determine other properties that relevance to the applications.

Keywords: Biocomposite; Poly(lactic acid); Natural Fiber; Alkaline treatment; Silane treatment

# Synthesis and Characterization of Chemically-Modified Cassava Starch Grafted with Poly(2-Ethylhexyl Acrylate) for Blending with Poly(Lactic Acid)

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## Abstract

Native cassava starch (CS) was chemically modified by grafting with 2-ethylhexyl acrylate (EHA) monomer to make it more hydrophobic for improved blending with poly(lactic acid) (PLA). Grafting was carried out using CS:EHA weight ratios of 2:1, 1:1, 1:2, and 1:3 in a methanol-water solvent mixture at 45 °C for 48 h. L-Ascorbic acid and hydrogen peroxide were used as the redox initiating system. Following purification, the poly(2-ethylhexyl acrylate)-grafted starch, starch-g-PEHA, was obtained either as a finely divided powder or as a slightly tacky solid with % grafting values in the range of 13–26% by weight. The main objective of this chemical modification was to improve the interfacial adhesion between the starch particles and the PLA matrix through the hydrophobic PLA-PEHA interactions. PLA/starch-g-PEHA blends were prepared in the form of solution-cast films with weight ratios ranging from 100/0 to 60/40. Tensile testing of the films shows a marked increase in extensibility and toughness up to a loading of 10% starch-g-PEHA above which the properties deteriorated rapidly due to starch particle aggregation. Thus, for potential use as biodegradable film packaging, the best properties were obtained for the PLA/starch-g-PEHA 90/10 blend.

*Keywords:* Synthetic biodegradable; Cassava starch grafted with poly(2-ethylhexyl acrylate); Biodegradable film; *PLA*
### Functionalised pH Responsive Hydrogels for Controlled Release

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#### Abstract

This work is concerned with the fabrication of pH responsive hydrogels. This was achieved by producing hydrogels that contain the vinylamine (VAm) functional group. We first prepared a poly(N-vinyl formamide)-copoly(N-hydroxyethyl acrylamide) (poly(NVF-co-HEA)) hydrogel via a photo-polymerization (UV-LED) technique using N'N-methylenebisacrylamide (MBAAm) as the cross-linking agent. This gel was then hydrolyzed to convert the vinylformamide groups in the polymer to vinylamine, the hydrolysis was carried out under acidic conditions, at 80 °C using 0.01 M aqueous HCl. These pH sensitive hydrogels were characterized by EWC, DSC and swelling behavior in water and ethanol. The hydrogels swelling at different pHs was observed between pH 2 and pH 12, nonhydrolyzed hydrogels (poly(NVF-co-HEA)) swelling was consistent at across the entire pH range but after hydrolysis the hydrogels show different behavior with reduced swelling ratios observed at acidic and basic conditions (pH2 and pH 10 and 12). For the release studies, three different dye were used; Orange II Sodium salt, Crystal Violet and Congo Red. The amount of each dye incorporated and released from the hydrogel was measured. Both uptake and dye release showed pH dependence, this is related to the functional groups present within the hydrogel's polymer network, the charge and characteristics of each dye molecule also effect both the dye uptake and release. For example, the anionic Orange II Sodium salt dye were complete incorporated into the hydrolyzed hydrogel but not the non-hydrolyzed gel. The release of this dye shows very little release at low pH values (pH 2) and large release at pH 12. Whereas, when comparing the release of Congo Red between the hydrolyzed and non-hydrolyzed gel, the hydrolyzed gel only sees significant release at pH 7. All the results show that the functionalised hydrogel was successfully prepared to form a hydrogel of poly(NVF-co-HEA-co-PVAm) and this gels exhibits pH dependent behavior.

Keywords: poly(vinylamine) (PVAm); pH responsive hydrogels; controlled release

### Fabrication of core-shell nanofiber scaffolds of PVA/SS - PLGA for skin tissue engineering applications

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#### Abstract

Core-shell nanofiber scaffolds of poly(vinyl alcohol)/silk sericin (SS) and poly(lactide-co-glycolide) (PVA/SS - PLGA) were developed for tissue engineering applications targeted especially for chronic wounds. The scaffolds were produced via a coaxial electrospinning technique. The morphology of scaffold was observed by scanning electron microscopy (SEM). Confirmation of the core-shell structure was observed by transmission electron microscopy (TEM) and attenuated total reflection - Fourier transform infrared spectroscopy (ATR-FTIR). Contact angle (CA) was used to study the hydrophilicity properties of the scaffolds. All parameter conditions for electrospinning, such as polymer concentration, flow rate, applied voltage and distance between needle and collector were studied. In the core section, PVA/SS was fabricated at appropriate conditions of 7 %w/v PVA, 2 %w/v SS, flow rate of 0.02 mL/min, applied voltage of 25 kV and distance to collector of 15 cm. In the shell section, PLGA was fabricated with appropriate conditions of 10 %w/v PLGA, flow rate of 0.3 mL/h, applied voltage of 25 kV and distance to collector of 15 cm. This provides fibers with good smooth surfaces and a small number of beads. The average fiber diameters were measured and found that for PVA/SS is between 230 to 500 nm, PLGA is 700 to 1200 nm and PVA/SS - PLGA core-shell scaffolds is between 800 to 1300 nm. The successful fabrication of core-shell structure was confirmed by TEM images and ATR - FTIR spectrum, as shown in the figure below. The core-shell scaffolds show hydrophobic behavior with a CA of 139.1°. The cell culture studies with primary normal human dermal fibroblast (NHDF) cell line proved that this scaffold is non-cytotoxic. Therefore, this core-shell structure of PVA/SS - PLGA scaffolds can be potentially used for skin tissue engineering applications.



Figure 1. TEM image of core-shell nanofiber scaffolds of PVA/SS - PLGA.

Keywords: Coaxial electrospinnig; Poly(lactide-co-glycolide); core-shell nanofiber; tissue engineering scaffolds

### Design and Fabrication of Biomedical-grade Polymer Membranes for Use in Oral Surgery Applications

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#### Abstract

Because of their biocompatibility and biodegradability, resorbable materials such as aliphatic polyesters are used in many biomedical applications such as sutures, screws, stents, scaffolds, and in oral surgery. Their continued development can be attributed to the advancements that have taken place in polymer synthesis, processing technologies, implant design, and innovative surgical techniques. In this work, the main aim has been to improve medical-grade polyesters currently being produced in Thailand for use as barrier membranes for oral bone regeneration. At present, this treatment requires the use of expensive imported materials from abroad. Therefore, this research has focused on reducing medical expenses by not having to rely on imported materials. In the first phase of this research, biodegradable barrier membrane prototypes, poly(L-lactide-*co-&*-caprolactone) modified with 37-long chain polypeptide, have been fabricated by electrospinning technique with a view to being effective in inducing oral bone cell growth. The results have shown that a modified poly(L-lactide-*co-&*-caprolactone) prototype membrane can release protein from polypeptide in amounts 0.1-0.7  $\mu$ M in 7 days that were non-cytotoxic for cells and evaluated the wound healing. The various polypeptide is suitable releasing amounts for inducing cell growth. If this type of resorbable barrier membrane can be successful in promoting the healing of dental wounds and bone regeneration in oral surgery, it will help to create new treatment guidelines for the dental industry in Thailand.

Keywords: Barrier membrane; bone regeneration; medical-grade polymer; poly(L-lactide-co- $\varepsilon$ -caprolactone); polypeptide

### Glancing Angle Deposition of Nanocolumnar NiWO Film Gas Sensor by Reactive Co-magnetron Sputtering with Glancing Angle Deposition

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#### Abstract

In this work, the nanocolumnar nickel tungsten oxide (NiWO) films were successfully developed by reactive co-magnetron sputtering with glancing angle deposition technique. The morphologies, crystallinities, atomic concentration in the nanocolumnar NiWO films analyzed by glazing incidence X-ray diffraction, field emission scanning electron microscopy, and X-ray photoelectron spectroscopy. The results indicated that as-deposited sample exhibit an amorphous structure with vertically aligned nanocolumnar, which depends on the shadowing effect. After annealing treatment at 500 C in air ambient for 2 hr leads to crystalline structure with nanocolumnar structure. Among the tested H<sub>2</sub> sensing, the nanocolumnar NiWO films were found to be higher than that of nanocolumnar NiO and WO<sub>3</sub> film. The results indicate that reactive co-magnetron sputtering with GLAD technique is a promising method for developing the mixed oxide nanostructure for gas sensor applications.

Keywords: Glancing angle deposition; NiWO, Nanocolumnar; Co-Sputtering

### Hydrogen Sulfide Gas Sensor Based on Cadmium and Zinc Ferrite Nanoparticles

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#### Abstract

Cadmium and zinc ferrite nanoparticles were prepared by co-precipitation. The crystal structure and morphology of the samples characterized by mean of X-ray diffraction (XRD), transmission electron microscope (TEM). The corresponding X-ray diffraction peaks confirm the formation of cubic spinel structure of  $CdFe_2O_4$  and  $ZnFe_2O_4$ . The  $CdFe_2O_4$  and  $ZnFe_2O_4$  and  $ZnFe_2$ 

Keywords: Cadmium; Zinc ferrite; Nanoparticles; Hydrogen sulfide

### Simple preparation of porous gold modified gold screen-printed electrode for non-enzymatic hydrogen peroxide detection

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#### Abstract

A simple one-step electrodeposition of porous gold so-called "dynamic hydrogen bubble template" (DHBT) method was used to prepare a porous gold modified on a gold screen-printed electrode (porous Aumodified AuSPE) for H2O2 detection. The surface morphologies of porous Au on AuSPE surface was characterized by using scanning electron microscopy (SEM), and the electrochemical behavior of the high surface area electrode was analyzed by using cyclic voltammetry (CV). The modified electrode exhibited improved current response compared with that of unmodified electrode. The porous Au-modified AuSPE was evaluated with a flow injection amperometry (FI-Amp). The proposed FI system was able to detect  $H_2O_2$  in the range of 0.01 to 1.0 mM. The relative standar deviation (RSD) of 10 replicates of 100 mM  $H_2O_2$  was 1.0%. The proposed porous Au-modified AuSPE showed a potential for non-enzymatic sensitive detection of hydrogen peroxide in real samples.

Keywords: Hydrogen peroxide; Non-enzymatic; Porous gold; Flow injection analysis; Amperometry

### Development of Screen-Printed Graphene Electrode by Overoxidation for Simultaneous Determination of Sunset yellow and Tartrazine

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#### Abstract

Sunset yellow (SY) and Tartrazine (TAR) are common azo dyes which are added into various food products such as candy, soft drinks, and carbonated drinks to improve their physical appearance. They are most widely used as colorants in many beverages. However, the receiving of their overdose can lead to high toxicity such as carcinogenicity, allergies and asthma due to the presence of azo group. Therefore, the simultaneous detection of SY and TAR need to be developed in order to control the quantities of these synthetic dyes in foods and drinks. In this work, we first proposed the in situ improvement of the electrocatalytic properties of screen-printed graphene surface using overoxidation process for the simultaneous detection of SY and TAR. 0.1 M phosphate buffer solution at pH 7 was used as a non-toxic overoxidizing reagent. After oxidizing process, the prepared electrode was employed to investigate the electrochemical properties of SY and TAR. The well-defined oxidation peaks of SY and TAR were observed at 0.5 V and 0.75 V (versus Ag/AgCl). Under the selected experimental variables, the calibration curves of SY and TAR were obtained in the ranges from 0.01  $\mu$ M to 5  $\mu$ M and 0.1  $\mu$ M to 10  $\mu$ M with detection limits of 0.01  $\mu$ M and 0.1  $\mu$ M, respectively. The developed electrodes were successfully applied to the simultaneous determination of SY and TAR in carbonated drinks with the recovery values were in the range of 97.51 to 105.07%, which can be acceptable. This new finding offers the simplicity and cost-effectiveness for electrode fabrication. Moreover, this procedure could be considered as an alternative method for the routine analysis of food colorants.

Keywords: Sunset yellow; Tartrazine; Overoxidation; Screen-printed graphene electrode

### Artificial Olfactory System Based on a Combination of Metal Oxide and Nanostructure Gas Sensors for Applications in Agricultural Quality

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#### Abstract

A combination of metal oxide and nanostructure gas sensors used in the olfactory system for applications in agricultural quality detection is presented. The system consists of three metal oxide gas sensors and one nanostructure gas sensor. The nanostructure gas sensor was fabricated based on conductive ink prepared from a hybrid of poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS), multiwall carbon nanotubes (MWCNTs) and silver nanoparticles (AgNPs) by drop-casting technique on an interdigitated silver electrode. Then, the system was applied to predict cabbage quality during different storage days. To analyze experiment data, principal component analysis (PCA) and artificial neural network (ANN) were employed. The results showed that PCA could classify cabbage odors related to storage days. ANN showed good agreement with the PCA result. More details and sensing mechanism of gas sensors will be discussed. Based on PCA and ANN results, it indicates that the developed olfactory system with a combination of metal oxide and nanostructure gas sensors can be an alternative technique for applications in food quality control and owns many advantages over other methods including rapid, non-destructive and easy measurement with high relative accuracy.

Keywords: Olfactory system; Nanostructure gas sensor; Metal oxide gas sensor; Food quality; Plant quality control

### Photoinduced current generation and photocatalytic activity of TiO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> nanoparticles coated MWCNTs films prepared by sparking process

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#### Abstract

Titanium dioxide (TiO<sub>2</sub>) and iron oxide (Fe<sub>2</sub>O<sub>3</sub>) nanoparticles (NPs) were successfully deposited on multiwall carbon nanotubes (MWCNTs) films using a low-cost and simple sparking process. The as-deposited film was annealed at 350 °C for 2 h to improve their crystallinity. The results show the anatase-TiO<sub>2</sub> and hematite-Fe<sub>2</sub>O<sub>3</sub> NPs with the size of 5-10 nm are coated on MWCNTs. The energy band gap of the as-prepared and the annealed films were 2.3 eV and 2.7 eV. Photocatalytic activity of the annealed films under visible irradiation is greater than the as-prepared films. Moreover, TiO<sub>2</sub>:Fe<sub>2</sub>O<sub>3</sub> with the ratio of 3:1 was the optimized condition. Interestingly, the relative current of the annealed films increased to 0.75 when increasing the irradiation time for 5 h. This result confirmed that the excited electron from photocatalytic activity can be transferred through the MWCNTs. This is an alternative way to produce the electric current from photocatalysis in the future.

Keywords: Titanium dioxide; iron oxide; MWCNTs; photocatalyst; sparking process

### α-MnO<sub>2</sub> nanofibers decorated N&S-co doped reduced graphene oxide and carbon nanotubes as cathode by printing method for ionic liquid additive-aqueous rechargeable Zn/MnO<sub>2</sub> batteries

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#### Abstract

Aqueous rechargeable Zn/MnO<sub>2</sub> ion batteries are high safety, environmental friendly, low cost  $\alpha$ -MnO<sub>2</sub> nanofibers decorated sulfur co-doped reduced graphene oxide (N&S-rGO) and carbon nanotubes (CNTs) were synthesized by hydrothermal process using KMnO<sub>4</sub> and MnSO<sub>4</sub> followed by ball milling process and printing method (noted as  $\alpha$ -MnO<sub>2</sub>\_NF + N&S-rGO + CNT). The as-prepared materials are served as cathode for rechargeable Zn-ion batteries electrodes that are characterized by XRD, XPS, SEM, TEM, and Raman spectroscopy. The results shows that the as-synthesized  $\alpha$ -MnO<sub>2</sub>\_NF had 5 – 10 nm in diameter and 100-200 nm in length and well dispersed on N&S-rGO and CNTs flakes. Electrochemical performances of batteries using  $\alpha$ -MnO<sub>2</sub>\_NF+N&S-rGO+CNT with 2.5% (w/v) ionic liquid of 1-ethyl-3-methylimidazolium ethyl sulfate ([EMIM] [MS]) in 2 M ZnSO<sub>4</sub> + 0.2 M MnSO<sub>4</sub> possesses a maximum specific capacity reaching 140 Fg<sup>-1</sup> at 30 mAg<sup>-1</sup> with energy density of 182 Wh kg<sup>-1</sup>. The specific capacity of 2.5% (w/v) [EMIM][MS] exhibited better cycling stability with a slower capacity fade and possesses a specific capacity remaining 50 mAh g<sup>-1</sup> after 1,000 cycles, while the capacity of the without [EMIM][MS] in aqueous electrolyte is a promising electrolyte for high performance and stability of Zn-ion batteries due to numerous advantages including simple fabrication and remarkable electrochemical behaviors.

*Keywords:* Zn/MnO<sub>2</sub> ion batteries; nitrogen & sulfur co-doped reduced graphene oxide; 1-ethyl-3-methylimidazolium ethyl sulfate; hydrothermal process; printing method

# Tin(II) *n*-hexoxide as a new initiator for the ring-opening polymerization of $\varepsilon$ -caprolactone: Isoconversional kinetic analysis by non-isothermal DSC

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#### Abstract

The kinetics of the ring-opening polymerization (ROP) of  $\varepsilon$ -caprolactone ( $\varepsilon$ -CL) initiated by 1.0, 1.5 and 2.0 mol% of Sn(OnHex)<sub>2</sub> and SnOct<sub>2</sub>/n-HexOH were investigated by non-isothermal differential scanning calorimetry (DSC). The DSC thermograms showed a dependency of the polymerization exotherm on the heating rate. The polymerization rate increased with the heating rate for all initiating systems. The values of the activation energy (E<sub>a</sub>) obtained from the exotherm peak by the Kissinger method for the ROP of  $\varepsilon$ -CL initiated by Sn(OnHex)<sub>2</sub> (60-64 kJ/mol) were lower than for the SnOct<sub>2</sub>/n-HexOH system (68-78 kJ/mol). The variation of E<sub>a</sub> with monomer conversion was investigated by the Kissinger-Akahira-Sunose (KAS) and Starink isoconversional methods. It was found that the values of E<sub>a</sub> decreased with the increasing nucleophilicity of the initiator. The 0.1 mol% Sn(OnHex)<sub>2</sub> initiator produced the highest molecular weight and % yield of PCL at polymerization temperatures of 140, 160 and 180 °C. In the synthesis of PCL, the efficiency of the initiator depends on the mechanistic pathway as demonstrated by the kinetic results.

Keywords: Kinetics; DSC; ε-caprolactone; ring-opening polymerization; tin(II) hexoxide

### Synthesis and Characterization of Poly(methylenelactide-g-Llactide) Graft Copolymers via Ring-Opening Polymerization

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#### Abstract

Nowadays, poly(lactic acid) (PLA) is one of the most important bioplastics in current commercial production which can be produced from renewable resources . The main objective of this study has been to synthesize poly(methylenelactide-g-L-lactide), P(MLL-g-LL), graft copolymers via ring-opening polymerization (ROP) in solution using PMLL:LL composition ratios of 1:30, 1:50, 1:300, and 1:600 % w/w. Liquid tin(II) *n*-butoxide  $(Sn(OnBu)_2) 0.03 \text{ mol}\%$  was used as the initiator in 1,2-dichlorobenzene (*o*-DCB) as the solvent at a temperature of 120 °C for 72 hours. The P(MLL-g-LL) products obtained were characterized in terms of their chemical structure by a combination of nuclear magnetic resonance spectrometry (<sup>1</sup>H-NMR, <sup>13</sup>C-NMR) and Fourier-transform infrared spectroscopy (FT-IR). The results showed that the copolymer molecular weights ranged from  $\overline{M}_w = 3700-7800 \text{ g mol}^{-1}$  with dispersities D = 1.1-1.4. Melting temperatures ranged from 133-162 °C. The T<sub>m</sub> of P(MLL-g-LL) from a composition ratio of PMLL: LL of 1:300 % w/w was higher than that from the 1:600 % w/w ratio due to the limit of the LL: PMLL ratio. In addition, the thermal decomposition temperature of P(MLL-g-LL) of around 314-335 °C was higher than that of PLA (300°C) which indicated that the grating reaction had increased thermal stability.

Keywords: Bromolactide, methylenelactide, poly(methylenelactide), poly(methylenelactide-g-L-lactide), ring-opening polymerization

### Fabrication of Poly(L-lactide-*co-ɛ*-caprolactone) and Poly(L-lactide-*co*-glycolide) by Combined 3D printing and Electrospinning for Use as Absorbable Nerve Guides

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#### Abstract

Nowadays, synthetic biodegradable aliphatic polyesters are used in a wide range of biomedical applications. Of these polyesters, the most commonly used are polylactide (PL), polyglycolide (PG), poly(&-caprolactone) (PCL) and their copolymers. In addition, electrically conductive polymers such as polypyrrole (PPy) have been demonstrated as having the ability to provide electrical stimulation for neurons in guided axonal extension. In this research work, medical-grade poly(L-lactide-co-ɛ-caprolactone) (PLCL) and poly(L-lactide-co-glycolide) (PLGA) copolymers have been synthesized and characterized to meet the specific requirements of nerve guide applications according to ASTM F1925-17 guidelines (Standard Specification for Semi-Crystalline Polylactide Polymer and Copolymer Resins for Surgical Implants). Porous scaffold prototypes were then fabricated under optimized conditions in the form of sheets by 3D-printing and electrospinning. The most suitable electrospinning conditions to produce consistent and uniform nanofibers from PLCL and PLGA were from 10 and 14 % w/v solutions in dichloromethane (DCM) and N,Ndimethylformamide (DMF), respectively. The results of this study have demonstrated the feasibility of adjusting the printing parameters to print scaffold prototypes using optimum polymer concentrations of PLCL and PLGA of 22.5 and 30.5 % w/v, respectively. The pressure, printing speed, and platform and cartridge temperatures which gave the best printing quality were 3.0-4.5 bar, 25.0-35.0 mm/s, 25 °C and 25 °C, respectively. The combination of 3D printing and electrospinning produced fibrous scaffolds which were clearly embedded within the 3D printed construct. This in turn has provided uniform oriented channels and pores having nano/micrometer resolution.

Keywords: Poly(L-lactide-co- $\varepsilon$ -caprolactone); poly(L-lactide-co-glycolide); 3D printing; electrospinning; electrically conductive polymers

### Fabrication of Medical-grade Poly(L-lactide) by Microinjection Molding for Use as Resorbable Plates and Screws

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#### Abstract

Craniomaxillofacial fracture repair normally uses metal plates and screws for bone fixation. However, metals have higher strength than human bone which causes abnormal bone growth. Furthermore, a metallic fixation device needs to be removed in a second operation causing more trauma to the patient. Resorbable polymer plates and screws provide attractive alternatives to overcome the limitations of metal bone fixation devices. Thus, the main purpose of this study has been to synthesize medical-grade poly(L-lactide) for use as resorbable bone plates and screws .The poly(L-lactide) was synthesized by the ring-opening polymerization of L-lactide and characterized according to ASTM F1925-17 (Standard Specifications for Semi-crystalline Polylactide Polymer and Copolymer Resins for Surgical Implants). Rheological and thermal properties were studied to optimize the fabrication conditions. Thermal analysis showed glass transition temperatures (Tg) of between 59-64 °C with melting temperatures (Tm) of 178-192 °C .Dynamic shear rheological testing at 210 °C exhibited linear viscoelastic properties which indicated Newtonian flow behavior of a viscous liquid. Values of the complex viscosity from steady shear testing were found to be dependent on the polymer molecular weight. Poly(L-lactide) plates and screws were fabricated using a Wittmann Battenfeld Micropower 5 micro injection molding machine at an injection temperature of 185 °C and pressure of 900 bars. Plates of 3 mm width and 20 mm length with 3 holes of 2 mm diameter were fabricated, while the screws were of 3 mm diameter and 9 mm length. Mechanical testing showed that the average maximum bending load of the plates was  $5.17 \pm 0.38$  N. The results of this work will be utilized for the future development of resorbable poly(L-lactide) bone fixation devices for craniomaxillofacial surgery.

Keywords: Microinjection molding; poly(L-lactide); rheology; resorbable plates and screws

### Fibrous Membranes of Poly(lactic acid) and their Nanocomposites for Adsorption Application

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#### Abstract

Morphology of electrospun polymer fibers can be varied by different electrospinning conditions. In this work, the effect of solvent mixing ratio, applied voltage, polymer concentration, and additive content is investigated on morphologies of the poly(lactic acid) (PLA) electrospun fibers. It is found that smooth fibers without polymer beads can be obtained by using optimal electrospinning parameters. To explore a possible important use of the prepared fibrous membranes, PLA and nanocomposite membranes are used as oil adsorbents. The nanocomposite membranes offer oil adsorption capacity higher than that of the neat PLA membranes. The nanocomposite membranes based on biodegradable PLA here are thus alternatives to conventional plastic adsorbents. Moreover, the membranes may find their use in adsorption of oily fluid exudates from living cells for biomedical applications.

Keywords: Fibrous membranes; Poly(lactic acid); Oil adsorption

### Effect of Organo-modified Montmorillonite Nanoclay on Mechanical and Thermal Properties of Carbon Fiber-reinforced Phenolic Composites

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#### Abstract

This work aims to explore the effect of organo-modified montmorillonite nanoclay (O-MMT) on the mechanical and thermal properties of carbon fiber-reinforced phenolic composites (CFRP). The CFRP at variable O-MMT contents (from 0 to 2.5 wt%) were prepared. All components were mixed by dry ball milling. After mixing, the composites were fabricated into specimens by hot compression molding at 150 °C and 1800 psi and then the specimens were post-cured in an oven at 150 °C for 1 h and 180 °C for another 2 h. The mechanical, thermomechanical and thermal properties of the polymer composites were characterized. It was found that the addition of O-MMT can improve the tensile strength, bending strength, impact strength, storage modulus, glass transition temperature and heat deflection temperature of the CFRP. However, these properties showed the highest value at different O-MMT contents. In this work, the addition of 1.5 wt% O-MMT was found to proper for preparing the heat resistant polymer composite. The results showed that the CFRP with 1.5 wt% O-MMT provided the highest tensile strength which increased by 20% and the higher impact strength which increased by 51% but a little lower bending strength which decreased by 1%, compared to those of the CFRP which were about 54 MPa, 32 KJ/m<sup>2</sup> and 164 MPa, respectively. In addition, the CFRP with 1.5 wt% O-MMT showed the storage modulus at 30 °C of 6.4 GPa which was 64% higher than that of the CFRP, and the glass transition temperature of 171 °C. Moreover, the CFRP with 1.5 wt% O-MMT had the highest heat deflection temperature of 152 °C which was comparable to that of the CFRP. This polymer composite might be a good candidate heat resistant material for automotive part applications.

Keywords: Carbon fiber-reinforced polymer composite, Phenolic resin, Nanoclay, Thermo-mechanical property, Heat resistant composite

### Influence of annealing and etching on physical and wetting properties of acrylic surface

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#### Abstract

The objective of this research aims to study the surface modification of acrylic for superhydrophobic application. The acrylics were modified using annealing at 50, 75 and 100 °C for 1 h. The annealed acrylics were then etched by tetrahydrofuran acid (THF) with the concentration of 99.8 % v/v for 1 min. The effects of annealing and wet chemical etching on morphology, optical properties and wettability can be characterized using atomic force microscopy, scanning electron microscopy, hardness tester, UV/vis spectroscopy and water contact angle measurement. This result exhibits that the hardness of acrylic increases with the increase of annealing temperature. Furthermore, the wet chemical etching has direct affected to wettability of acrylics due to the increasing of surface roughness.

Keywords: acrylic; tetrahydrofuran; wettability; annealing; etching

### UCST-type Themoresponsive Nanocarriers for Synergistic Imageguided, Photothermal- and Chemo-therapy

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#### Abstract

Multifunctional stimuli-responsive pharmaceutical nanocarriers have been developed for cancer drug delivery to overcome the drawbacks from single therapy and enhanced the therapeutic efficiency. Herein, synergistic nanocarriers from triple combination of the upper critical solution temperature (UCST)-type thermo-responsive behavior, fluorescence imaging and hydrophobic anticancer therapy were fabricated. The thermoresponsive core/shell nanocarriers of quaternized chitosan and poly(2-hydroxyethyl methacrylate)(QC/PHEMA) were synthesized via surfactant-free visible light-induced emulsion polymerization using a riboflavin as a photoinitiator. The average diameter ranging from 100-150 nm with narrow size distribution and positively surface charge were obtained. The degree of quaternization (DQ) and the N,N'-methylenebisacrylamide (MBA) crosslinker content was varied, and found that this significantly affected the thermo-responsive behavior of the nanocarriers. The nanocarriers prepared from 40% DQ of TMC and 5 mol% of MBA crosslinker exhibited ~ 78% swelling from 20 °C to 70 °C in acetate buffer ph 5. For imaging and chemotherapy application, the sodium copper chlorophyllin (SCC) as a NIR-induced photothermal agent and fluorescent label molecules, and honokiol as an anticancer drug were loaded into the nanocarriers. The anionic SCC can be loaded on the outer shell of nanocarriers by electrostatic interaction whereas hydrophobic honokiol were loaded into the inner core of nanocarriers. The high loading content (LC) and encapsulation efficiency (EE) of honokiol into the QC/PHEMA nanocarriers were  $16.6 \pm 0.02$  % and  $99.8 \pm 0.16$  %, respectively. For SCC, LC of  $27.9 \pm 0.3$  % and EE of  $96.8 \pm 1.3$  % were attained.

Keywords: UCST-type thermoresponsive, quaternized chitosan/PHEMA nanocarrier, synergistic cancer therapy

### Preparation and Characterization of Biodegradable Film Based on Poly (lactic acid) Incorporate with Herbal Oil

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#### Abstract

This research studied the preparation and characterization of biodegradable films based on poly (lactic acid) or PLA incorporate with herbal oils (coconut oil, moringa oil, clove oil and lemongrass oil). Furthermore, in the PLA matrix, the herbal oil, acts as a natural plasticizer and is added in order to replace the synthetic plasticizer to improve the mechanical properties of the PLA film. In this study, PLA films with difference amounts of herbal oil (0, 0.25, 0.5, 0.75 and 1 wt %) were made by a twin-screw extruder and then blow film extruded was used to prepare PLA/herbal oil biodegradable films. The obtained films had thicknesses of around 15-20 microns. The obtained compound and biodegradable films were characterized for physical and mechanical properties as follows. The melt flow index of the compound was increased with increasing amounts of herbal oil. The morphology of biodegradable film was studied by SEM. The SEM photos illustrate that the films were smooth and miscible. Physical properties of the PLA film addition herbal oil have show that the water vapor permeability was higher than the neat PLA film. Moreover, for migration tests including plasticizer migration and overall migration, the results of the biodegradable films show in the range of 0 to 5%. The chemical functional groups of the biodegradable films were determined by FTIR. The spectrums of these films present the wavenumbers of functional groups of both PLA and herbal oil. The addition of herbal oil into the PLA film effects the films mechanical properties. It was found that the tensile strength and modulus at break decreases while percent elongation at break increases. The PLA film with coconut oil presented the largest improvement and showed an elongation at break around 40% greater than the neat PLA film. Finally, these films can be used for food packaging applications as can are biodegradable and environment friendly.

Keywords: Poly (lactic acid); herbal oil; coconut oil; Biodegradable film

### Development of the Mulch Film From Poly (lactic acid) Incorporated Natural Fillers

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#### Abstract

This research was interested in producing and developing mulch film from a biodegradable polymer (Poly (lactic acid)) incorporated with natural fillers include ginger powder and lemongrass powder. This project studied the effect of different contents of natural fillers on physical and mechanical properties. The natural filler contents were varied at 0, 2.5, 5.0, and 7.5 wt%. The biocomposite film was produced via twin-screw extruder and blow film extruder. The biocomposite films produced had a thickness of 70-80 micron. The compounds from the twin-screw extruder were characterized using melt flow index and then the biocomposite films were subsequently characterized for physical properties (Color parameters, and UV transmittance), chemical properties (Fourier Transform Infrared Spectroscopy; FT-IR), and mechanical properties (Tensile test). The higher natural filler contents increased the melt flow index of the compounds. The mechanical properties of the biocomposite film that indicated the addition of natural fillers alters the tensile strength and % elongation, showing a decrease in both with increasing contents of natural fillers. The highest contents of natural fillers have tensile strength approximate 11-13 MPa and %elongation have ranges around 3-4%. However, the modulus at break shows an increase. The natural fillers also effected the color of biocomposite films, with the biocomposite film having a yellow-brown shade when ginger powder was incorporated and a green shade when the lemongrass powder was used. From the color of biocomposite film this influences the light transmittance, which is seen when the natural fillers are adding into the PLA matrix. These films have reduced UV transmittance when compared with the neat PLA film. This shows that the modified biocomposite films can absorb UV light when greater the contents of the natural fillers are used, therefore, these films can help to prevent weed growth and also reduced the number insects. In the future, this biocomposite film based on Poly (lactic acid) and natural fillers can be used to produce biodegradable mulch film and reduces agricultural plastic waste.

Keywords: Poly (lactic acid); natural fillers; ginger powder; lemongrass powder; Biocomposite film

### Development of Poly lactic-co-glycolic acid and graphene composite for tissue engineering applications

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#### Abstract

Nowadays, 3D bioprinting technology is widely used in a field of medical engineering such as development of biomaterial and supporting components for the fabrication of functional living tissues. Poly lactic-co-glycolic acid (PLGA) is the most attractive polymeric material which used to fabricate a tissue engineering due to FDA-approved biocompatible and biodegradable material. However, PLGA is too fragile and not durable in some application. Thus, graphene is a nanocarbon material which has been recently used as a composite material due to its excellent high mechanical strength, high flexibility, electrical conductivity and good biocompatibility. In this study, we developed PLGA/Graphene composites for fabrication of scaffolds by using 3D bioprinting technology and studied the properties of the composites. Scanning electron microscope (SEM) were studied the characteristic of graphene on the scaffolds. Mechanical tests showed that 3D-printed graphene-based materials exhibit higher strength and elasticity than unloaded PLGA scaffolds. Moreover, PLGA/Graphene scaffold was compatibled with *Caenorhabditis elegans* as organism model. Thus, PLGA/Graphene was able to apply with tissue engineering.

Keywords: 3D-printing; Graphene; PLGA; PLA; Tissue engineering

### Silk-Fibroin Coated Liposomes for Sustained Release of Doxorubicin in Drug Delivery System

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#### Abstract

Sustained-release drug delivery system has been designed for long-term drug release at therapeutic range. One approach is to coat the surface of nanocarriers with polymeric materials, including chitosan, alginate or some fibrous proteins which are biodegradable and biocompatible. This study aims to develop a delivery system using liposomes coated with fibrous silk fibroin (SF) encapsulating with an anticancer drug. SF was extracted from silk cocoon by LiBr followed by dialysis, providing the SF concentration of ~10%w/v. Liposomes were mainly composed of soy phosphatidylcholine (soyPC) and cholesterol with addition of cationic stearylamine (SA) to provide electrostatic interaction for SF-coating. The study demonstrated that the optimal lipid composition was 10:1:3 of soyPC : cholesterol : SA. Liposomes with or without SA were coated with the extracted SF at various concentrations in the presence of methanol. Liposomes were characterized for their size, charges and morphology. Mean diameter of SF-coated SA liposomes was ~145 nm which was higher than uncoated liposomes. Zeta potential of SF-coated SA liposomes was ~+15.40 mV and decreased with increasing percentage of coating SF. However, transmission electron microscope imaging showed that the surface of SF-coated liposomes was uneven compared to the uncoated liposomes. In this work, doxorubicin (DOX) was loaded into the liposomes for demonstrating in vitro drug release. The encapsulation efficiency of DOX was ~93%. DOX could release from SF-coated liposomes over 20 days in a slower rate than that of the free drug and the uncoated liposomes without SF. Therefore, the SF-coated liposomes could be potentially used and further optimized for sustained drug delivery system for cancer treatment.

Keywords: Drug delivery; Doxorubicin; Liposomes; Silk fibroin; Sustained Release

### Development of Drug-loaded Biodegradable Electrospun Membranes for the Treatment of Periodontitis

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#### Abstract

Medical-grade poly(L-lactide-co- $\varepsilon$ -caprolactone) (PLCL) copolymers with LL:CL compositions of 70:30 mol % were synthesized via the ring-opening polymerization (ROP) of L-lactide and  $\varepsilon$ -caprolactone and characterized according to the requirements of ASTM F1925-09 (Standard Specification for Semi-Crystalline Polylactide Polymer and Copolymer Resins for Surgical Implants). The copolymers were characterized by nuclear magnetic resonance spectroscopy (NMR), dilute-solution viscometry, differential scanning calorimetry (DSC), gas chromatography (GC), inductively coupled plasma spectroscopy (ICP), Karl-Fischer titration, polarimetry and mechanical (tensile) property testing. In addition, the PLCL copolymers were loaded with tetracycline in various concentrations and fabricated by electrospinning in the form of porous membranes for use in guided periodontal tissue regeneration. The optimal electrospinning conditions for preparing the membranes were also determined. Fabrication by electrospinning has been shown to be capable of producing highly elastic copolymer membranes comprising 3- dimensional fibrous networks. The results showed that the amount of drug loading had significant effects on the fibre diameter, pore size and tensile properties of the membrane. It is hoped that this research work will lead to periodontal membranes which are of comparably high quality but much lower in cost than the currently imported commercial products for periodontal repair in Thailand.

Keywords: Poly(L-lactide-co- $\varepsilon$ -caprolactone); electrospun porous membrane; drug loading, guided periodontal tissue regeneration

## ศูนย์วิจัยวัสดุศาสตร์

คณะวิทยาศาสตร์ มหาวิทยาลัยเชียงใหม่ Meterials Science Research Center, MSRC

### movation Showcase 2020





### ศูนย์วิจัยวัสดุศาสตร์ คณะวิทยาศาสตร์ มหาวิทยาลัยเชียงใหม่

ศูนย์วิจัยวัสดุศาสตร์ เป็นหน่วยงานตามพระธาชบัญญัติ มหาวิทยาลัยเชียงใหม่ พ.ศ. 2551 สังกัดคณะวิทยาศาสตร์ มหาวิทยาลัยเชียงใหม่ เริ่มก่อตั้งเมื่อเดือน มีนาคม พ.ศ. 2553 โดยมีพันธกิจคือ ประสานงานวิจัยและ นวัตกรรมเพื่อน่ามหาวิทยาลัยเหียงใหม่ก้าวไปส่ความเป็นเลิศทางวัสดุศาสตร์และ เทคโนโลยีและสามารถนำพลงานไปใช้ประโยชน์ได้จริงทั้งในภาคอุตสาหกรรมและ RNRN

วิสัยทัศน์

ศูนย์วิจัยวัสดุศาสตร์ คณะวิทยาศาสตร์ มหาวิทยาลัยเชียงใหม่ เป็นศนย์ความเป็นเลิศที่ได้รับการยอมรับทั้งในระดับชาติและนานาชาติ

## พันธกิจ

ส่งเสริมและพลิตงานวิจัยและนวัตกรรมด้านวัสดุศาสตร์และ เทคโนโลยีไปสู่ความเป็นเลิศให้ได้รับการยอมรับระดับชาติและนานาชาติ

### โครงการวิจัยกายใต้การตำเนินงาน ศูนย์วิจัยวัสดุศาสตร์



คลัสเตอร์ศูนย์ความเป็นเลิศทางวัสดุศาสตร์ฯ โครงการมหาวิทยาลัยวิจัยแห่งชาติ (NRU) สกอ.



งบประมาณแพ่นตินพ่านมหาวิทยาลัยเชียงใหม่ แพนบูรณาการวิจัยและนวัตกรรม



โครงการการพลิตพลงานทางวิชาการ ศูนย์ความเป็นเลิศ ทางวัสดุศาสตร์ฯ มหาวิทยาลัยเชียงใหม่ (พลิตพลงานทางวิชาการ)



โครงการ ศูนย์ความเป็นเลิศด้านวัสดุศาสตร์และเทคโนโลยีวัสดุ มหาวิทยาลั้ยเชียงใหม่ (COE)



โครงการ Co Research อุทยานวิทยาศาสตร์ ภาคเหนือ ประจำปิงบประมาณ 2556



โครงการศูนย์ความรู้เฉพาะต้านวัสดุขั้นสูงสำหรับ

อิเล็กทรอนิกส์พิมพ์ได้และเซ็นเซอร์ ปี 2560 (CMU NECTEC)



โครงการวิจัยด้านพลาสติกชีวภาพโดยส่านักงานพัฒนาเศรษฐกิจ ຈາກฐานชีวกาพ (องค์การมหาชน) (BEDO)



งบประมาณการขับเคลื่อนยุทธศาสตร์ที่ 2 เชิงธุก : นวัตกรรมด้านอาหารและสุขภาพ และการดูแลพู้สูงอายุ



โครงการเทคโนโลยีวัสดุเพื่อพัฒนาคุณภาพชีวิตพู้สูงอายุ และพู้ป่วยยากไร้ งบบริการวิชาการ มหาวิทยาลัยเชียงใหม่





อาการ 40 ปี SCB2 คณะวิทยาศาสตร์ ชั้น 4 ห้อง 2408

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msrc.scicmu

Ins.053 941915

# ศูนย์วิจัยวัสดุศาสตร์

คณะวิทยาศาสตธ์ มหาวิทยาลัยเชียงใหม่

Meterials Science Research Center, MSRC

### งานประชุมวิชาการ

การประชุมนานาชาติอาเซียนของเซ็นเซอร์ทางเคมี ครั้งที่ 10 : เซ็นเซอร์ทางเคมีเพื่อสังคมที่ยั่งยืน (The 10<sup>th</sup> Asian Conference on Chemical Sensors (ACCS 2013) : Chemical Sensors for the Sustainable Society)

The 10<sup>th</sup> International Conference on the Physical Properties

and Application of Advanced Materials (ICPMAT2015)

วันที่ 11-14 พฤคจิกายน 2556 ณ โรงเรม ติเอ็มเพรส เชียงใกม่ จังกวัตเชียงใกม

ACCS 2013

วันที่ 17-21 พฤศจิกายน 2558

**ICPMAT2015** 

ณ โรงเรม ติเอ็มเพรส เชียงใกม่ จังกวัตเชียงใกม่

การจัดประชุมวิชาการนานาชาติ The First Materials Research Society of Thailand International Conference (MRS-Thailand 2017)

วันที่ 31 ตุลาคม – 3 พฤศจิกายน 2560 ณ โรงแรม ดิเด็มเพรส เชียงใหม่ จังหวัดเชียงใหม่



งานประชุมวิชาการ The 1<sup>st</sup> International Symposium on Applied Plasma Science and Engineering for Agro and Bio-industry : CMU-SKKU Joint Workshop on Nano and Bio Material Devices





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### Nanoscience and Nanotechnology Interdisciplinary Program



### Faculty of Science, Chiang Mai University



### Nanoscience and Nanotechnology Interdisciplinary Program

Chiang Mai University was founded in 1964 and was the first institution of higher education in the north and the first provincial university of Thailand as a center for academic and occupational knowledge in order to benefit the region and the country as a whole.

### Ph.D. Program in Nanoscience and Nanotechnology

### (International/Interdisciplinary Program)

is an interdisciplinary program administered by Materials Science Research Center (MSRC), Faculty of Science. Nanoscience and Nanotechnology is a broad field of research encompassing diverse disciplines including chemistry, biochemistry, materials science, physics, pharmacy, medicine, computer and electrical engineering, biomedical engineering, agro-industry, and environmental sciences. At CMU, faculty members from various backgrounds and research interests in various aspects of nanoscience and nanotechnology. This Ph.D. program provides opportunity for students to gain knowledge and skills through interdisciplinary collaborations.

### Degree Requirements:

- Qualifying examination to evaluate his/her ability before presenting a thesis proposal
- Participation in the nanoscience and nanotechnology
  graduate seminar series
- Oral presentation on the topic related to his/her thesis at the international conference(s)
- Publications and/or patents (2 publications for 3 year program and three publications for 4 year program)

### **Our Mission**

Nanoscience and Nanotechnology Doctoral program at CMU is a multidisciplinary program providing a platform to students to work in broad area of nanoscience and nanomaterials disciplines.

We aim to facilitate interaction among scientists across the disciplines toward the discovery beyond the traditional disciplines.

In this program, Ph.D. students are educated through research projects in broad disciplines and academic activities. Coursework is not required but students have freedom to participate in courses that are relevant to their research projects.



### Tuition Fee : 35,000 BAHT per semester Financial support/scholarships are available.

Financial support/scholarships are ava

### Curriculum Plans:

Doctoral Program in Nanoscience and Nanotechnology (International Program)

- 3-year doctoral degree
  (Student with Master's degree) : 48 credits
- 4-year doctoral degree
  (Student with Bachelor's degree) : 72 credits
- · Financial support/scholarships are available

### **Eligibility of the applicants**

#### Type 1.1 : Student with Master's degree

 Completion of Master's degree with GPAX greater than 3.00 in the field of science and technology or health sciences or related fields

#### Type 1.2 : Student with Bachelor's degree

 Completion of Bachelor's degree with GPAX greater than 3.25 in the field of science and technology or health sciences or related fields

OR

 Completion of Bachelor's degree with GPAX greater than 3.00 and additional requirement of publications.

### Contact information

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- Website: http://www.science.cmu.ac.t/
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## ้เครื่องมือวิเคราะห์ของศูนย์วิจัยวัสดุศาสตร์

คณะวิทยาศาสตร์ มหาวิทยาลัยเชียงใหม่



Analytical Tools of Materials Science Research Center

Faculty of Science, Chiang Mai University

ศูนย์วิจัยวัสดุศาสตร์คณะวิทยาศาสตร์ มหาวิทยาลัยเชียงใหม่ ได้ เปิดให้บริการงานวิเคราะห์ลักษณะเฉพาะและสมบัติของวัสดุ ให้กับ หน่วยงานทั้งภายในและภายนอกมหาวิทยาลัย ทั้งภาครัฐ และเอกชน เพื่อสนับสนุนผลงานด้านงานวิจัยและพัฒนาการค้นคว้าคิดค้น สิ่งประดิษฐ์และต้นแบบต่างๆ อันจะนำไปสู่การใช้เทคโนโลยีใหม่ๆ ใน การพัฒนาประเทศ

XRD

### **X-ray Diffractor**

เทคนิคเอกซเรย์ดิฟแฟรกชัน หรือ เทคนิค XRD เป็น เทคนิคที่นำรังสีเอกซ์มาใช้วิเคราะห์โครงสร้างผลึกของ สารประกอบและแร่ ผลการวิเคราะห์จาก XRD ทำให้สามารถ แยกแยะประเภทและชนิดของวัสดุที่พบในธรรมชาติว่ามีรูปแบบ ้โครงสร้างผลึกแบบใด หรือจำแนกได้ว่าวัสดุที่พบเห็นนั้นเป็นแร่ ชนิดใด โดยทำการวัดค่าความเข้มของรังสีที่สะท้อนออกมาที่มุม ต่างๆเปรียบเทียบกับข้อมูลมาตรฐานที่ทำการตรวจวัดโดย องค์กร JCPDs (Joint Committee on Powder Diffraction Standard)



DMA

**Dynamic Mechanical Analyzer** METTLER TOLEDO



METTLER-TOLEDO ŚU DMA1





### Differential Scanning Calorimeter



DSC เป็นเทคนิคที่ใช้วิเคราะห์ทดสอบวัสดุโดยการ วัดค่าพลังงานความร้อนและอุณหภูมิของสารตัวอย่าง เปรียบเทียบกับสารมาตรฐาน เมื่อมีการเปลี่ยนแปลง ทางกายภาพหรือการเปลี่ยนแปลงทางเคมี เช่น การ หลอมเหลว การเปลี่ยนสถานะ การเปลี่ยนรูปผลึก การ เกิดปฏิกิริยาเคมี เป็นต้น

METTLER TOLEDO

HORIDA

METTLER-TOLEDO șu DSC1

NanoSizer

### Nanoparticle Analyzer



เครื่องวัดขนาดอนุภาค ค่าความต่างศักย์ บนผิวอนุภาค และน้ำหนักโมเลกุลโดยอาศัย การเลี้ยวเบนของแสงเลเซอร์





#### **Surface Area and Pore Size** Analyzer

ใช้วิเคราะห์หาพื้นที่ผิว ขนาดของรูพรุน การกระจาย ขนาดของรูพรุน ปริมาตรรูพรุนทั้ง Mesopore และ Micropore และยังสามารถท่ำนายรูปร่างของรูพรุนเปิด ของวัสดุโดยใช้หลักการดูดซับทางกายภาพ

Quantachrome



### (Potentiostat / Galvanostat)

PGSTAT เป็นเครื่องมือวัดทางเคมีไฟฟ้าแบบโมดุลลา ควบคุมด้วยคอมพิวเตอร์ ประกอบด้วย ชุดควบคุมหลัก ที่สามารถทำงานด้วยระบบโพเทนทิโอสแตทและ กัลวานอสแตท อีกทั้งมีโมดุลลาที่ใช้สำหรับทำ

Ω Metr

Electrochemical impedance spectroscopy (EIS) โดยมีชุดโปรแกรมสำเร็จรูปควบคุมการ ทำงานชดควบคมหลัก ตัวอย่างการนำไปใช้ศึกษา เช่น ศึกษาการกัดกร่อนของชิ้นงาน, fuel cell, battery, energy, biotechnology และ sensor development เป็นต้น



### **Simultaneous Thermal Analysis**

() Rig STA เป็นเครื่องที่สามารถหาปริมาณความร้อนที่เปลี่ยนแปลง ไปของสารตัวอย่างเนื่องจากปฏิกิริยาทั้งที่เป็นแบบดูดความร้อน และคายความร้อน อีกทั้งสามารถหาปริมาณน้ำหนักของสาร ตัวอย่างที่เปลี่ยนแปลงไปอันเนื่องมาจากปฏิกิริยารีดักชันหรือ

Rigaku ŚU Thermo plus Evoz

### **3D** Printer

### **3D** printer



เครื่อง 3D Printer คือเทคโนโลยีการผลิตที่สามารถเปลี่ยนข้อมูล ดิจิตอล หรือแบบจำลอง 3 มิติ ที่สร้างขึ้น ให้กลายเป็นชิ้นงานจริง<sup>้</sup>ที่ สามารถจับต้องได้ โดยหลักการของเครื่องคือการเติมเนื้อ วัสดุ (additive) ทีละชั้น (layer by layer) จนได้ตามแบบที่ต้องการ ซึ่ง แตกต่างจากกระบวนการผลิตแบบดั้งเดิมเช่น เครื่องกลึง เครื่องกัด เครื่องตัด หรือเครื่อง CNC ที่มักใช้การตัด หรือนำเนื้อวัสดุ ออก (subtractive) ดังนั้นวัสดุที่ใช้และสูญเสียในการผลิตจึงน้อยกว่า

ออกซิเดชัน ขณะที่เพิ่ม/ลด และรักษาอุณหภูมิให้คงที่แก่สาร

ตัวอย่าง สารตัวอย่างที่สามารถวิเคราะห์ได้เช่น สารพอลิเมอร์

เซรามิค โลหะ อาหาร ยาปฏิชีวนะ ยางสังเคราะห์ เป็นต้น

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จาวน์โหลดเอกสาร เบบฟอร์มขอใช้บริการ ารื่องมือวิเคราะห์

ระบบจอง

เรื่องมือวิเคราะห์

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