



UPPER MIDWEST

SOCIETY OF PLASTICS ENGINEERS • UPPER MIDWEST SECTION

the

# SPECIALIST

VOLUME 45 APRIL 2019

## SPE UPPER MIDWEST

# MegaTech 2019:

### THE FUTURE OF QUALITY INSPECTION TECHNOLOGIES

## Wednesday, May 1<sup>st</sup>, 2019

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# HALL OF FAME INDUCTEE GREG HAMMOND



*Mr. Greg Hammond has been inducted into the Upper Midwest SPE Hall of Fame*

Mr. Gregory Hammond, plastics industry professional residing in Saint Paul, MN with wife Sonja and three children, Isabelle (17), Louisa (15) and Jamison (11).

## Industry Background

Mr. Hammond is currently a Sr. Global Account Manager for the Petrochemical Business Unit of SABIC, specializing in sales and marketing of polycarbonate, polyester, styrenic and polyolefin resins and compounds. He has been a plastics industry professional in Minnesota for 35 years. A graduate of Winona State University, with a Bachelor of Science in Chemistry, Mr. Hammond has held a variety of technical and commercial roles for RTP Company, EMPAK (now Entegris), LNP Engineering Plastics, GE Plastics and currently SABIC.

## SPE Upper Midwest Section Board Tenure

Mr. Hammond served as an SPE Upper Midwest board member for nine years in the starting in 1989 and held various roles including Secretary, Education Chair, Special Events Chair, Vice-President, President-Elect, President, Past-President. He organized educational seminars and special events, including annual golf outings (140+ golfers) and holiday parties (500+ attendees). Specifically, in 1992, Mr. Hammond led the effort to revive the Upper Midwest Section's annual education seminar

resulting in a leading edge, well attended (100+) event at HTC. As well, during his tenure as a board member, the Upper Midwest Section received multiple awards for New Member Growth and Newsletter Excellence.

## SPE Upper Midwest Section Presidency

During Mr. Hammond's 18-month tenure as President (1995-96), the Upper Midwest Section created two branch sections, Western Wisconsin and Southeast Minnesota, growing the membership to over 800 members and holding many meetings and events in the branch areas. With a dedicated focus on member growth, outreach and education, the Upper Midwest Section was awarded the prestigious SPE STAR Award in 1995. Mr. Hammond also initiated and sponsored Mr. David Erickson for the SPE Honored Service Member Award.

## Plastics Outreach

During his tenure on the board, with the goal of adding and retaining members, Mr. Hammond routinely met with local plastics business leaders to encourage them to support the Upper Midwest Section by offering reimbursement of SPE dues for employees. As well, Mr. Hammond often visited schools to present and discuss the benefits of plastics in the world. This included visits to grade schools with the "Plastics Kit" and speaking at career days. Currently, both Mr. Hammond and his employer SABIC are contributors to the America Chemistry Council.

## Giving Back

Mr. Hammond always makes giving back a priority whether for industry or community. He currently coaches youth hockey (since 2016) and youth baseball (since 2013) in Saint Paul and South St. Paul, MN. As well, he currently serves on the South St. Paul Youth Hockey Association Board as Director of Sponsorships (since 2017) with the focus on raising funds to make hockey accessible and affordable for underprivileged girls and boys ages 5-15.

## Serving Others

Since 2003, Mr. Hammond has been an active member of King of Kings Lutheran Church in Woodbury, MN where he serves with the music ministries as a guitar player for the worship bands. In 2007, he organized a traveling music ministry team that continues to volunteer several times per year as worship leaders at Union Gospel Mission/Saint Paul. This includes speaking to and ministering to the homeless, addicted and afflicted men in the community. In 2015, he also helped found and currently helps lead the men's ministry team at King of Kings which meets on a regular basis to complete various local mission projects for King of Kings and the surrounding community.

# PRESIDENT'S REMARKS



Eric Hall  
*SPE Upper  
Midwest  
President*

Another beautiful Minnesota spring is upon us, and another year of great SPE activities and events!

First, join me in congratulating Greg Hammond who was inducted into the SPE Upper Midwest Hall of Fame. This is an annual award, given to an SPE member who has shown exceptional leadership and accomplishments within SPE and the plastics industry. Greg was a long-time member of the Upper Midwest Section, who held just about every board position we have. Our award ceremony needed to be rescheduled to April 5th, so we can't include pictures of the event, but check out Greg's bio in the newsletter, and look to our website for pictures of this year's gala.

Our next event is the Spring Megatech, scheduled for May 1st, at Hennepin Technical College in Brooklyn Park, MN. This year's topic is The Future of Quality Inspection Technologies. This should be a useful Megatech for anyone involved in production and process control in a wide range of plastics industries. We have an impressive lineup of speakers on board, and as usual this will

be a great opportunity to meet, network and socialize with other local plastics professionals. Check out the section in this newsletter for more details, and we really hope to see you there.

We have some exciting news on the expansion of the Upper Midwest Section. Several years ago the Iowa section went dormant, leaving a gap in local SPE section coverage for this important geographical area. I'm happy to report our section has requested and been granted the opportunity to expand our sections geographical area to now include most of Iowa. We have enlisted the help of Jeremy Bland with PolySource to help kick this off and get some local section activity going in this area again. If you are based in, or do significant work in Iowa, and would like to be involved in getting this going, please give one of us a call we can get you involved at whatever level you are interested in contributing. Keep an eye open for our first event in 2019, likely a Minitech this fall.

As always, I would like to acknowledge the board and all the volunteers and sponsors who make

this local section of SPE work. We have a tremendously talented and hard-working group of folks who work behind the scenes to put our events together, interact with the national SPE organization, and get out this newsletter. We have people on our committees from all areas of the plastic's industry, including production, materials, R&D, testing, sales and marketing and academia. If you are interested in working with this fun and talented group of folks, and advancing your own network and career, get in touch with any of us on the board and we'll be happy get you more information.

Have a great spring, and hope to see you at the Megatech.

Eric Hall

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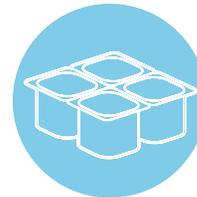
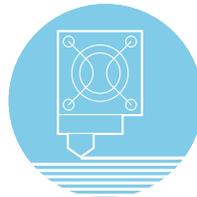
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# COUNCILOR'S CORNER

Michael Arney, Ph.D. COUNCILOR – UPPER MIDWEST SECTION

It has been a busy winter and spring in the Society of Plastics Engineers. We have a new President-Elect, a new President, several new Vice-Presidents and a new format for ANTEC. And this is just for starters. The Society has also begun to inform the public about the values that plastics bring to our everyday lives and how indispensable plastics are to a sustainable future.

## JAIME GOMEZ IS THE NEW PRESIDENT ELECT

In January, Dr. Jaime Gomez was elected as the new President-Elect. Jaime most recently served as our Vice-President of Events. Previously, he served as Vice-President—Treasurer, Vice President—Secretary, and Vice President—Marketing and Communications. Jaime has also served as a Councilor for many years. In 2014, he was awarded the President's Cup, which is SPE's highest service award. Jaime's career includes the founding of three different start-up companies, leadership roles in two mid-size companies including Novatec and K-Tron, as well as work in Fortune 500 companies.

## DR. BRIAN LANDES INAUGURATED AS PRESIDENT OF SPE

Dr. Brian Landes was officially inaugurated as the new President of SPE at the March 17 Council meeting in Detroit, MI. Dr. Landes is the Technology Leader at Dow Chemical in Midland, MI. During his inaugural address, Dr. Landes reminded us of our purpose

as a society: "To inspire plastics professionals through knowledge and networking". He proceeded to introduce a new strategy plan which better aligns our resources with our mission to produce better value by increasing knowledge and networking opportunities. If interested, please contact one of the Upper Midwest SPE Board members for details.

## THE UPPER MIDWEST CHAPTER OF SPE NOW INCLUDES IOWA

During the Council meeting of March 17, 2019, the Councilors unanimously approved our petition to include the state of Iowa in our geographic area. Previously, Iowa was served by the Iowa Section, which was dissolved in 2012. We would like to welcome the members of SPE who work / reside in the state of Iowa and welcome you to attend our many events and activities throughout the year.

## THE FUTURE OF ANTEC

Lastly, I would like to introduce you to some exciting changes to the Annual Technical Conference (ANTEC), that was introduced for ANTEC 2019 (Detroit).

- Tutorial Sessions for both Injection Molding and Extrusion were included in the Technical Program. The purpose of

these sessions is to provide the professionals who new to the extrusion or injection molding technologies and want to know more about the fundamentals.

- A series of mentoring sessions offered plastics professional the opportunity to obtain advice in the plastics industry from long-term recognized experts in their fields.
- Antec Insight session is a departure from the normal technical program. These presentations are intended to be future-looking, and not technical in nature. The topic for 2019 was "sustainability". We agree that everyone wants/needs to use plastics in our daily activities, but no one wants to deal with excessive plastic waste. The purpose of these discussions were to explore the ways in which we can find a balance between these two opposing ideals.
- An improved ANTEC social program was introduced. Everyone had fun at the "Fowling" (Football/Bowling) Tournament, which was held at the Fowling Warehouse in Detroit.
- Future ANTEC Locations were announced. ANTEC 2020 will be in San Antonio, TX, March 29 – April 2. ANTEC 2021 will be in Denver, Co, March 22-25, 2021.



# SPE EDUCATION COMMITTEE

Tom McNamara and Joshua Weed

We are pleased to announce that our Section has provided three students with scholarship awards and financial support towards their education. These students fulfilled all requirements for the awards and were selected based on their educational performance, contributions to their plastics programs, society involvement, and essays explaining their accomplishments and goals for continuation in the plastics industry.

Winner of the Jerome Formo Scholarship Award, given to a student in a 4-year Plastics Engineering program, is Laryssa Meyer from the University of Wisconsin – Stout. Laryssa has been the President of the UW-Stout Student Chapter of SPE since May of 2018. She is also the Vice-President of the Society of Women Engineers at UW-Stout. Laryssa will be interning at Medtronic this summer and has a keen interest in medical device manufacturing. Laryssa is very deserving of the Jerome Formo Scholarship Award.



Winners of the Tony Norris Scholarship Award, given to students in a 2-year Plastics Engineering Technology program, are Hanok Ayana and Ramiro Garcia of Hennepin Technical College.

Henok made the President's list in the Spring of 2018 and has been serving as Secretary of the SPE Plastics Student Chapter at HTC. Henok has worked at Tyco Plastics,

Boston Scientific, and Torax Medical in various roles. He has attended many of our MiniTechs and our last MegaTech as well as the MDM. Henok aspires to continuing his progress in our industry.



Ramiro was Vice-President of the SPE Plastics Student Chapter at HTC. He was also on the HTC President's list in the Spring of 2018. Ramiro has worked for Pro Ampac in flexible packaging, polymer processing, and multi-layer extrusion. He also worked at Fulcrum in Minneapolis. He aspires to working with plastics and helping people through innovation of better techniques and products.

Both Henok and Ramiro deserve the Award by embodying the values, skills, determination, and leadership that was exhibited by Tony Norris.



Our Upper Midwest Section will also be giving out both Jerome Formo and Tony Norris Scholarships this Spring. You can get forms on our Upper Midwest website [www.uppermidwestspe.org](http://www.uppermidwestspe.org).

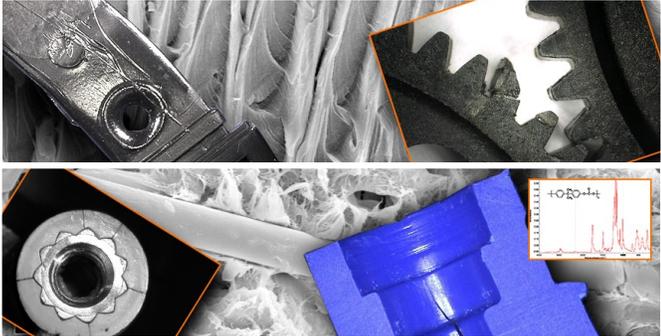


## Upper Midwest SPE Chapter has Officially Absorbed the Iowa Chapter

Our chapter has officially absorbed the Iowa Chapter adding to our growing group. Initially we will be working to alert local members of the change and moving forward will be looking to start putting together great events like the Mega and Mini Techs that we hold currently in Minnesota. To help with the transition Des Moines local Jeremy Bland from Polysource will become our local Iowa Ambassador. This is very exciting news and everyone apart of the board is looking forward to this adventure. If you have any questions, or are looking to lend a hand please contact Jeremy at [jeremy@polysource.net](mailto:jeremy@polysource.net).



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## INVESTIGATION OF MOLECULAR ENTITIES PRESENT IN MALEATED POLYPROPYLENE USING LSM

*Susnata Samanta<sup>1</sup>, Jeff Toke<sup>2</sup>, John Muzzy<sup>2</sup>, Charles L. Liotta<sup>1</sup>*

<sup>1</sup>*Chemistry and Biochemistry*

<sup>2</sup>*Chemical and Biomolecular Engineering*

*Georgia Institute of Technology*

*Atlanta, GA*

### Abstract

Maleated polypropylene (mPP) is often used as an adhesion promoter in polypropylene composites. Poly (maleic anhydride) (PMA) or oligomers of maleic anhydride are often present in maleated polypropylene, but they do not contribute to the adhesion of polypropylene to glass fiber reinforcements. PMA can be detected by confocal laser scanning microscopy.

### Introduction

Growing environmental concern has created a major thrust for using thermoplastic matrices in polymer composites due to their ease of recycling. Among different thermoplastics, polypropylene matrix composites attract considerable attention due to their low cost and ease of processing. But most of the reinforcing fibers, like glass fibers and natural fibers, are polar while polypropylene is non polar. In order to achieve efficient stress transfer from the matrix to the reinforcing fibers different coupling agents are used as adhesion promoters. Maleated polypropylene, the most widely used polymeric coupling agent for polypropylene composites, contains reactive maleic anhydride pendant groups capable of covalently bonding with the functional groups present on the fiber surface. As a result, the composites exhibit a stronger and tougher interface.

Although maleated polypropylene improves the mechanical properties of the polypropylene based composites, some limitations remain. First, a large excess (beyond optimal amount) of maleated polypropylene must be used to ensure grafting for interfacial adhesion. Second, maleated polypropylene often show heterogeneous structure due to the difficulty in controlling the introduction of the maleic anhydride units and due to the presence of low molecular weight fractions of maleated polypropylene, which do not create effective entanglements with matrix polypropylene.

Indeed, these undesired outcomes result from the competitive reactions occurring during reactive

extrusion of polypropylene(1) in the presence of organic peroxides and maleic anhydride(3). The grafting of a single unit of maleic anhydride is difficult because of the extremely reactive nature of the radical involved in the mechanism. The polypropylene radical is known to undergo chain degradation by  $\beta$ -scission during reactive extrusion in the presence of organic initiators. The objective of this research is to investigate the distribution of maleated polypropylene in polypropylene composites and blends to understand the mechanism of adhesion and compatibilization in these materials. Confocal microscopy appears to be a useful technique for this purpose as maleated polypropylene exhibits auto-fluorescence when excited with a 488 nm laser and observed at wavelength  $>530$  nm(3). This research should lead to better use of maleated polypropylene coupling agents.

In recent research(4,5), there has been debate about the nature of grafting of maleic anhydride onto the propylene chain, which is still not well understood. The principal disagreement resides in whether the maleic anhydride exists as single unit, as an oligomeric unit along the backbone of the polymer, or at the chain ends. Not only that, there is discussion of how much of the measured maleic anhydride content in mPP is bound, unbound, or oligomeric maleic anhydride(6) species.

### Experimental

In previous research(3) the distribution of maleated polypropylene in mPP/PP blends was examined in a confocal microscope with excitation light of 488nm and observed at a wavelength  $> 530$  nm. Interestingly, a strong fluorescence signal was recorded. The observed auto-fluorescence volume (AF) was correlated to the amount of maleic anhydride present in the maleated polypropylene samples as well as to the amount of maleated polypropylene present in the mPP/PP blends(3). However, the molecular species which are responsible for this phenomenon are not clearly identified. It is important to point out that no auto-fluorescence is observed from pure maleic anhydride or succinic anhydride samples. The confocal laser-scanning microscope (LSM) can be a powerful tool to determine the distribution of maleated polypropylene in composites and blends. However, to efficiently use LSM as an analytical tool it is critical to

understand what is generating the observed auto-fluorescence.

This problem was approached by separating each chemical entity present in the maleated polypropylene and analyzing their fluorescence characteristics as well as investigating their molecular structures.

It is mentioned in the literature(6) that unreacted maleic anhydride and/or the unbound oligomeric maleic anhydride can be extracted from maleated polypropylene (mPP) by acetone. Consequently, soxhlet extraction of the solid mPP with acetone was used. Both the acetone soluble and insoluble fractions obtained from maleated polypropylene after extraction were studied. Their fluorescence characteristics were monitored and their structures were investigated using FTIR, and liquid and solid state  $^{13}\text{C}$  NMR in order to identify and gain a better understanding of the fluorescence phenomena /structure relationship. Three commercially available maleated polypropylene samples were examined, designated as MP-1,2 and 3 respectively. Their anhydride contents were reported to be 10, 2.4 and 1.6 % by weight respectively. Fluorescence characteristics of the maleated polypropylene were observed by a Zeiss LSM 510 scanning confocal microscope using a 100x oil immersion objective. 2-D images of cross sectional slices of the samples were obtained using 488 nm excitation light and the fluorescence signal was recorded at a wavelength  $> 530\text{nm}$ . All three commercial maleated polypropylene samples were extracted with acetone for 24 hours in a soxhlet apparatus and each fraction was dried at  $120^\circ\text{C}$  for 4 hours under vacuum prior to analysis. Maleated polypropylene as well as their acetone soluble and insoluble portions were melted on glass slides, covered with cover slip and used for confocal studies. For comparison, fluorescence characteristics of pure poly (maleic anhydride) were studied as a control and its molecular characteristics were examined with FTIR and  $^{13}\text{C}$  NMR.

## Results and Discussion

Pure polypropylene, maleic anhydride and succinic anhydride were tested and did not exhibit any auto-fluorescence when excited with 488 nm laser and observed at wavelength  $> 530\text{ nm}$ . However all the commercial maleated polypropylene samples (e.g. MP-1, 2 and 3) exhibited strong auto-fluorescence when excited with the same conditions. Both the acetone soluble fraction and the insoluble fraction were analyzed by confocal microscopy. The insoluble fraction exhibited a very faint auto-fluorescence at similar experimental set up (Fig 1-C). But interestingly, the acetone soluble fraction

showed very strong auto-fluorescence (Fig.1-D). The solid-state  $^{13}\text{C}$  NMR study revealed that the acetone soluble fraction (Fig. 1-D) has a high content of maleic anhydride units characterized by the presence of a peak at 175 ppm (C=O)(7). The  $^{13}\text{C}$  NMR of the commercial maleated polypropylene (Fig.1-A) exhibits a peak at 175 ppm as well. However, this signal is absent from the spectrum of the insoluble fraction (Fig.1-C), which indicates that most of the maleic anhydride derivatives present in the original commercial samples were extracted into the acetone soluble fraction. In addition, since only the acetone soluble fraction exhibited fluorescence, the maleic anhydride derivatives seem involved in the fluorescence phenomena.

The acetone soluble fraction was investigated in more detail. The fluorescence characteristics, FTIR spectra and solid-state  $^{13}\text{C}$  NMR spectra (Table 1) of the acetone soluble fraction were compared with the pure polymaleic anhydride (PMA). Pure polymaleic anhydride exhibited very similar auto-fluorescence (Fig.1-B) characteristics to the one observed for the acetone soluble fraction. In addition, the similarity of the  $^{13}\text{C}$  NMR (Fig.1-B, D) and FTIR (Fig.2) spectra of the acetone soluble fraction and PMA samples strongly indicates that the acetone soluble fraction is composed of maleic anhydride oligomers. This argument is consistent with the literature, which reported that oligomers of maleic anhydride could be formed during reactive extrusion of polypropylene(4,7). Based on the presence of characteristic peaks of polypropylene (1460, 1378 &  $2860\text{-}3000\text{ cm}^{-1}$ ) in the FTIR spectra of the acetone soluble fraction (Fig. 2), it is likely the oligomers of maleic anhydride are attached to low molecular weight polypropylene chains. Moreover, the  $^{13}\text{C}$  solid-state NMR spectra of acetone soluble portion (Fig.1-D), show a peak at about 10-50 ppm which is consistent with the presence of  $sp^3$  carbon peaks arising from the polypropylene.

The origin of the fluorescent phenomenon from poly (maleic anhydride) is not fully understood yet. In order to explain the molecular basis of the observed fluorescence from poly (maleic anhydride) the keto-enol tautomerism of the maleic anhydride polymer or oligomer (Scheme 1) was considered. The enol form (succinic anhydride unit) of the poly (maleic anhydride) is a conjugated system and may be the source of the observed auto-fluorescence. To support this argument, we performed a FTIR study of the acetone soluble portion of mPP at different stages of drying. It is mentioned in literature that drying mPP samples at  $120^\circ\text{C}$  for 24 hrs evaporates the unreacted maleic anhydride and induces complete cyclization of any diacid into the cyclic anhydride form(4). Based on the FTIR data the concentration of the diacid decreased as the acetone soluble portion was dried with a corresponding increase of cyclic anhydride concentration in it. The appearance of a new peak around

1640  $\text{cm}^{-1}$  in the dried sample indicates the presence of C=C adjacent to a polar group, which might be the OH group shown in the conjugated structure (Scheme 1). The proposed conjugation is further supported by the noticeable change in the nature of the OH peak in FTIR spectra of the acetone soluble portion of mPP before and after drying. Similar spectral changes were observed in the FTIR studies of pure PMA samples before and after drying.

The acetone soluble fraction of mPP is not expected to contribute favorably to interfacial adhesion in polypropylene composites. This fraction has a low molecular weight and a relatively high concentration of anhydride groups. This fraction is probably not miscible with polypropylene. This fraction does migrate to glass interfaces based on observations made by confocal microscopy.

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**Keywords:** Maleated polypropylene, LSM, maleic anhydride, autofluorescence

### Conclusions

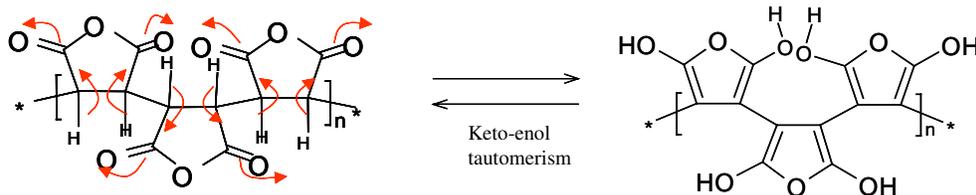
A strong fluorescence signal was observed from the acetone soluble portion of all three commercial maleated polypropylene samples. Faint fluorescence was observed from the acetone insoluble portion of all three commercial maleated polypropylenes. The similarity of FTIR and  $^{13}\text{C}$  NMR spectra of the acetone soluble portion of maleated polypropylene and pure poly (maleic anhydride) indicates that maleic anhydride oligomers, which forms during reactive extrusion of polypropylene in the presence of maleic anhydride and peroxide initiators, is responsible for the observed fluorescence. Keto-enol tautomerism of poly (maleic anhydride) might be responsible for the observed auto-fluorescence.

**This paper first appeared in the Proceedings of the Annual Technical Conference of the Society of Plastics Engineers, 2005, and was Reprinted by permission. Copyright Society of Plastics Engineers. All rights reserved.**

**ATTEND ANTEC 2020  
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Table 1. Characteristic FTIR,  $^{13}\text{C}$  NMR<sup>6,8</sup> peaks and fluorescence property of maleated polypropylene (MP-1) before and after soxhlet extraction and pure poly(maleic anhydride).

| Sample                                  | FTIR peaks ( $\text{cm}^{-1}$ )   | Solid state $^{13}\text{C}$ NMR (ppm)                               | Fluorescence characteristics |
|---|---|---|------------------------------|
| Intact Maleated PP (MP-1)               | 1784, 1850 (C=O, anhydride), 1460, 1378, 2860-3000 ( $\text{CH}_2$ , $\text{CH}_3$ )  | $\sim 170$ (C=O), 20-50 ( $\text{sp}^3$ -C)                         | Strong                       |
| Acetone soluble portion of mPP (MP-1)   | 1710 (C=O, acid), 1784, 1850, 1460, 1378, 2860-3000 ( $\text{CH}_2$ , $\text{CH}_3$ ) | $\sim 170$ (C=O), 20-50 ( $\text{sp}^3$ -C)                         | Very strong                  |
| Acetone insoluble portion of mPP (MP-1) | 1784, 1460, 1378, 2860-3000   | 20-50 ( $\text{sp}^3$ -C)   | Weak                         |
| Pure poly(maleic anhydride)             | 1710 (C=O, acid), 1784, 1850,   | $\sim 170$ (C=O), 140 ( $\text{sp}^2$ C), 20-50 ( $\text{sp}^3$ -C) | Very strong                  |



Scheme 1. Keto-enol tautomerism of poly(maleic anhydride)

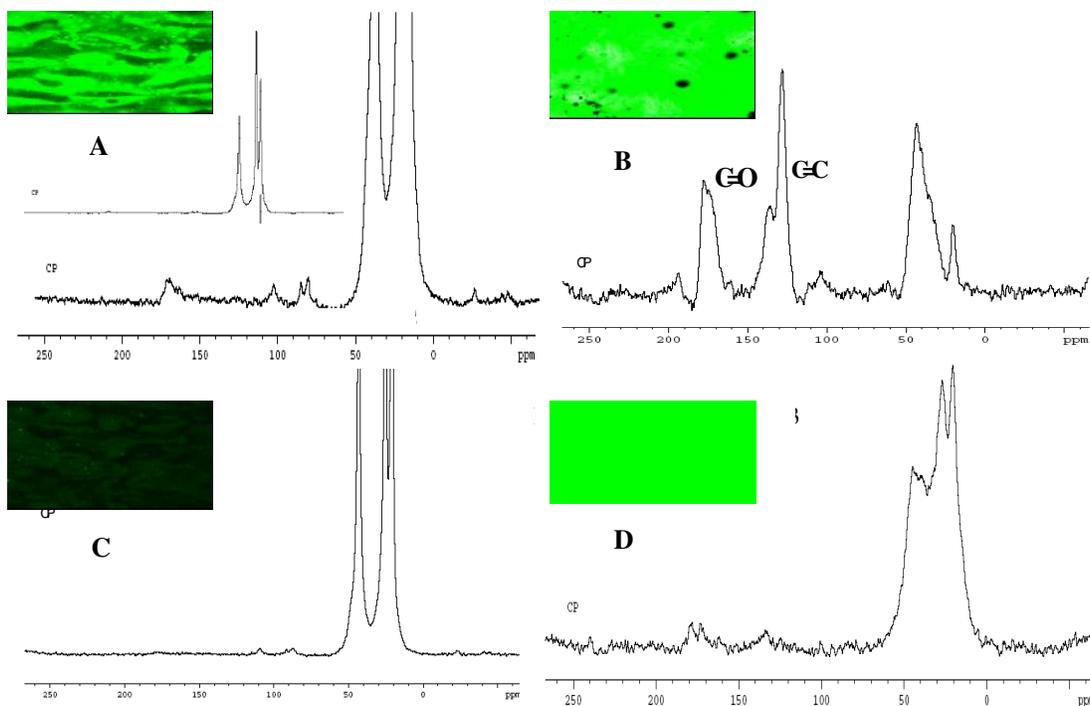


Figure 1. Solid-state <sup>13</sup>C NMR and fluorescence characteristics of Maleated polypropylene (MP-1) (A), Pure poly(maleic anhydride) (B), Acetone insoluble portion of mPP (C), and Acetone soluble portion of mPP (D)

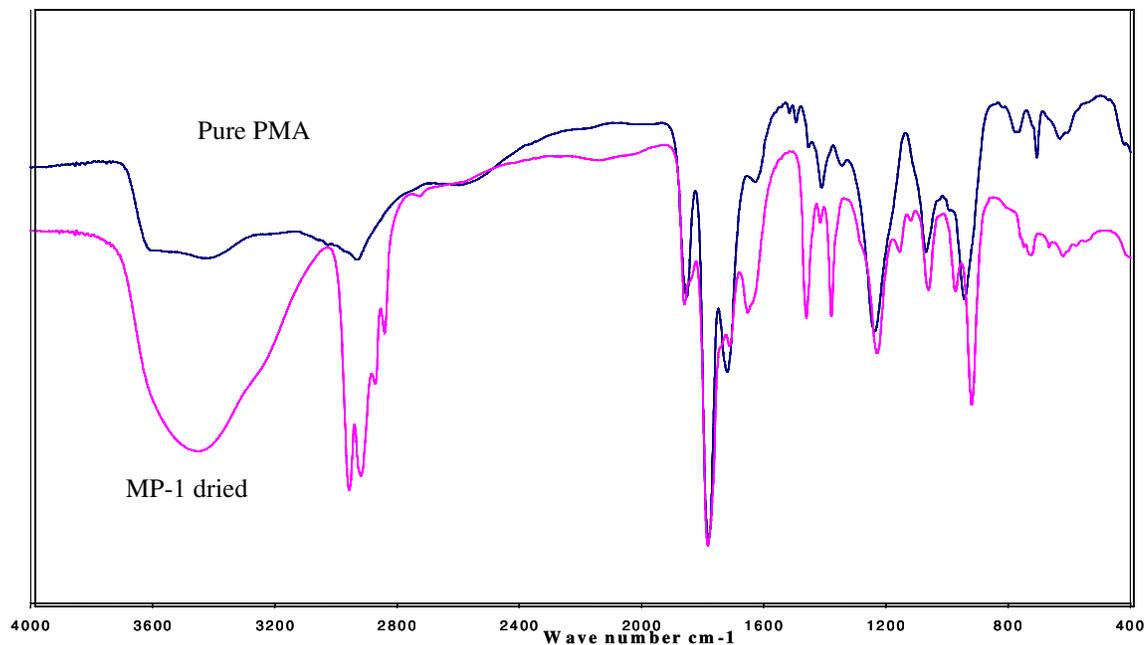


Figure 2. FTIR spectra of pure poly(maleic anhydride) (PMA) and acetone soluble portion of maleated polypropylene (MP-1) dried at 120°C for 24 hrs.



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