THE PARTICLE TECHNOLOGY FORUM (PTF) NEWSLETTER

An American Institute of Chemical Engineers (AIChE) Forum

Message From The Chair

Dear Colleagues:

I hope you and your families have enjoyed the summer. The Summer 2025 issue of the Particle Technology Forum newsletter highlights the career of Dr. Ecevit Biligi, who passed away last year. Ecevit was an AIChE Fellow and founder of PENPAL, the Particle Engineering and Pharmaceutical Nanotechnology Lab at the New Jersey Institute of Technology.

We also have the privilege of making an announcement that AIChE has elected to create its 13th Institute Award in recognition of the career accomplishments of Dr. Liang-Shih (L.S.) Fan. The *L.S. Fan Award for Advancing Fundamentals of Chemical Engineering* will recognize sustained contributions that have advanced the frontiers of chemical engineering, with an emphasis on particle science and technology or related field. This award will be supported by an endowment with fundraising led by Ah-Hyung (Alissa) Park (University of California, Los Angeles) and Bing Du (ExxonMobil), in collaboration with the AIChE Foundation. I also want to congratulate this year's Award winners. You will be able to read more about them in the upcoming Fall Issue of the newsletter.

On behalf of the PTF executive committee, we hope to see you in person at the 2025 AIChE Annual Meeting – November 2-6, which will be hosted at the John B. Hynes Veterans Memorial Convention Center, Marriott Copley Place, and Sheraton Boston | Boston, MA. Online registration with early-bird registration rates is accessible through the AIChE website. I would like to remind you to purchase PTF dinner tickets with your registration. We are working to arrange a tour for members of a Boston-area firm specializing in particle technology. We hope to make an announcement about the opportunity in the next few weeks. I would lastly like to express gratitude to the PTF Leadership committee, our PTF programming leaders, and our numerous volunteers. Without your involvement, our forum would not be as strong as it is today.

Warmest Regards,

Michael Molnar

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Process R&D Fellow, The Dow Chemical Company

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A Peek At The Contents

Welcome to the website for the Particle Technology Forum!

It is your comprehensive resource to keep you informed and stay connected with the Particle Technology Forum community.

FROM THE EDITOR'S DESK

This issue is dedicated to late Dr. Ecevit Bilgili to remember and celebrate his contributions to the field of Particle Technology. I personally came to know him almost two decades ago while evaluating his impressive nomination for the PTF best PhD award. We instantly connected on two fronts - our passion for particle technology and our respective bonding with our families. His passion for the field of comminution remained unabated, and was still evident in his presentation during the Prof. Austin memorial session at the last AIChE annual meeting in San Diego.

We will miss him as a colleague in the PTF community.

Shrikant Dhodapkar
Senior R&D Fellow / AIChE Fellow
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PTF Website

Our website is a resource for our membership, which must be updated and enriched by the contributions from all of us. I encourage you to remain engaged by visiting the website and sharing the links with others.

Some key features...

- PTF leadership, where you can learn about the esteemed professionals guiding our initiatives.
- Stay up-to-date with upcoming events, including conferences, webinars, and workshops that offer opportunities for professional growth and networking.
- Discover our awards section to celebrate the achievements and innovations of our members who are making significant contributions to particle technology.
- Newsletters provide updates, insights, and articles to keep you informed about the latest exciting events, trends, research, and job opportunities.

If you are interested in contributing content to the newsletter or share information about upcoming events, please reach out to me (yfan5@dow.com).

Yi Fan
Senior Research Scientist
The Dow Chemical Company, TX

Past PTF Chairs

Year	PTF Chair
1992 – 1994	L. S. Fan Ohio State University
1994 – 1996	Reg Davies DuPont Company
1996 – 1998	Mike Roco NSF
1998 – 2000	Karl Jacob Dow Chemical Company
2000 – 2002	George Klinzing University of Pittsburgh
2002 – 2004	Ralph Nelson DuPont Company
2004 – 2006	Alan Weimer University of Colorado, Boulder
2006 – 2008	Shrikant Dhodapkar Dow Chemical Company
2008 – 2010	Hugo Caram Lehigh University
2010 – 2012	Raymond Cocco PSRI
2012 – 2014	Jennifer Sinclair Curtis University of Florida
2014 – 2016	Reza Mostofi UOP
2016 – 2018	Raj Dave NJIT
2018 – 2020	Bruce Hook Dow Chemical Company
2020 – 2022	Jim Gilchrist Lehigh University
2022 – 2024	Reddy Karri PSRI
2024- 2026	Maria Silvina Tomassone Rutgers University

In Memoriam - Ecevit Bilgili (1974–2024)

It is with great sadness that I inform you of the untimely death of our colleague and friend, Professor Ecevit Bilgili of New Jersey Institute of Technology (NJIT), who suddenly passed away in his residence in Woodbridge, New Jersey, on the evening of November 25, 2024.

Ecevit Atalay Bilgili was born in Istanbul in 1974 to Mustafa and Mensure Bilgili. He earned a Bachelor of Science in Chemical Engineering with high honors at Bogazici University (1996) and a Doctor of Philosophy degree from the Illinois Institute of Technology (2001). For his doctoral dissertation he was awarded the *George Klinzing Best Ph.D. Award* of the American Institute of Chemical Engineers (AIChE). Between 2002 and 2004 Professor Bilgili was a post-doctoral research fellow at the University of Florida; between 2004 and 2009 he served in R&D positions within Merck Research Laboratories.

Ecevit applied for a faculty position at NJIT in 2008, and he joined our Otto H. York Department of Chemical and Materials Engineering (CME) in 2009 as Assistant Professor, after being selected from a pool of more than 150 candidates. He was chosen for this position not only because of this scholarly excellence but also because he showed a remarkable ability to communicate his scientific findings with notable eloquence, while radiating obvious passion about his professional interests. He was promoted to Associate Professor in 2014, and, at the time of his death, he was a Full Professor of Chemical Engineering, and Associate CME Chair for Undergraduate Study.

Ecevit's research was in Particle Science and Engineering, with a special focus on the development of fundamental understanding of the mechanisms and transformations governing the formation of fine particles; nanoparticles, nanocomposite microparticles, and amorphous solid dispersions (ASDs). At NJIT Ecevit founded and directed PENPAL – the *Particle Engineering and Pharmaceutical Nanotechnology Lab*. Additionally, he was associated for a long time with the *New Jersey Center for Engineered Particulates* and the NJIT National Science Foundation Industry–University Cooperative Research Center (IUCRC). During his years, his work was supported, individually and with others, by the National Science Foundation, the National Institutes of Health, the New Jersey Health Foundation, and a number of private pharmaceutical corporations. Looking at the mere titles and numbers fails to convey the full picture of vibrant research with graduate students and faculty colleagues. Ecevit published in the best journals of his field, including, among others, *Pharmaceutics, Powder Technology, Chemical Engineering Science, European Journal of Pharmaceutics and Biopharmaceutics, and the International Journal of Pharmaceutics*. Overall, he published more than 100 papers in refereed journals. His work was cited in the scientific literature more than 5000 times. Since 2018, he had 400 new citations or more every year. Twelve of his papers were cited more than 100 times each. It is remarkable to notice how his reputation grew in the last few years – between 2022 and early 2025 his citations grew by 75% from 2900 to 5070 while his h-index grew from 30 to 41.

In 2020, during the application process for his promotion to Professor, reviewers provided commentaries praising Ecevit's scholarly activities. One reviewer wrote that *"In my opinion, Professor Bilgili has become one of the leading scholars in the field of particle science and engineering."* Then another, describing a solution provided by Ecevit to a specific problem, indicated that *"only a few years ago…such complete and elegant solution was the dream of many engineers and researchers in this and similar fields."* A third reviewer stated that *"Professor Bilgili's work in modeling of milling, provides very important contributions in the elusive task of describing milling beyond the traditional approaches."* A fourth provided a list of six sub-disciplines, ranging from the formation of drug nanoparticles and nanocomposite microparticles to multi-scale approaches to complex non-linear rate processes, where, in the opinion of this reviewer, Ecevit "*did original, first rate and in some cases pioneering work*."

Other additional recognitions of his original research are his election to *Fellow of the American Institute of Chemical Engineers* (AIChE) (2022) and his serving as Editor of *Pharmaceutics* and *Advanced Powder Technology, as well as* being the founding editor of the *Journal of Pharmaceutical and BioTech Industry.*

Ecevit was not only a first-rate scientist but also an extremely dedicated and beloved educator. He was primary advisor to six doctoral students and co-advisor to additional three. Additionally, he was thesis advisor and project advisor to close to 20 Master of Science students. Ecevit mentored more than 40 undergraduate students under a variety of programs and initiatives, as well as high school students and high school teachers. The list of awards that his mentored students have secured has more than 50 entries. The list of journals and magazines for which he volunteered to provide manuscript reviews has 145 entries.

As an instructor, Ecevit was extremely enthusiastic, effective, and knowledgeable. His success as a teacher came not only from his intimate familiarity with the subject that he taught but also from his meticulous preparation before a course was offered, combined with an intensity, a passion, and an enthusiasm that is not often seen in a faculty member. He was a rigorous and demanding instructor, and he challenged his students to achieve the highest level of proficiency in his class without being overbearing or unreasonably harsh. Ecevit brought his own style to all the courses that he taught and systematically introduced new aspects to the courses to make them more relevant to industrial applications and more appealing to the students. He went beyond the normal duties associated with teaching, and provided extra sessions, even during weekends, to help struggling students progress through the courses. In other words, Ecevit preferred to provide additional resources to the students rather than lower the quality and the standard of the courses he taught.

The best term that comes to mind when I think of Ecevit is "passion." Ecevit was very passionate about everything that he did, and especially about teaching. His passion for teaching was legendary, and it is better expressed through the words of some of the students who took his favorite undergraduate course, CHE 260-Fluid Flow. One student wrote "*Dr. Bilgili is hands down the best professor in the ChemE department. He is incredibly knowledgeable in what he teaches and he shows great passion for it as well. Make sure to pay attention in lectures and write everything down. He also tells you what to expect on exams so make sure to study as well, and you will learn a lot."* Another student in the same class wrote: *"Professor Bilgili is probably the best professor in the whole department. There aren't many professors that put in more time and effort into providing their students with resources to succeed. He teaches a class with hard concepts and you definitely need to put the work in to get a good grade. He is tough but he is fair."*

Because of his excellence as instructor, he received the prestigious title of *NJIT Master Teacher*, as well as and *Fellow of the Institute for Teaching Excellence*. He additionally received *Excellence in Teaching Awards* from both the Newark College of Engineering and NJIT.

Ecevit was also a dedicated family man, a devoted father to his two daughters, Melisa and Kayra, and a caring husband to his wife Melike. He loved spending time with his family and often dreamed of retiring one day in his beloved native country, Türkiye, spending time on the coast of the Mediterranean Sea. Talking to him about politics, history, philosophy, and current events was always very engaging, interesting, and ultimately enjoyable.

Ecevit was very serious when it came to his profession, but he also knew how to be cheerful and lighthearted, and how to have fun. I have many recollections of that, including the last one, spending a whole evening at the AIChE Annual Conference in San Diego, only a few days before his passing, when we visited many of hospitality suites at the event where we chatted, ate, drank, and joked accordingly.

We, friends, colleagues, and students of Professor Bilgili, will remember him not only as an original and diligent researcher, an active and eloquent participant in professional and technical discussions, a generous and dedicated advisor and colleague, an efficient and effective academic administrator, a devoted mentor to students at all stages of their study and development, but also as a considerate, kindhearted, likeable, friendly person that we will be sorely missed.

Piero M. Armenante

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Professor Leonard Austin Memorial Session (AIChE Annual Meeting 2024)

The Human Prof. Austin

- Sofia will always remember Leonard as the sweetest husband, full of life and vitality: witty and with a great sense of humor.
- Leonard loved animals, especially his dogs, enjoyed traveling, was an avid reader and enjoyed listening to music
- Family and friends that meant the most to Len. He remembered his family, students, colleagues and friends around the world with such love.
- Witty and passionate

"Writing a paper with him was a learning experience: he was very particular on how graphs looked like, and did not accept anything but perfection.." (Prof. Luis Marcelo Tavares*)

"...He was short in stature but large in his knowledge and attention to the details. Grad students were pushed to work long hours ... with great satisfaction. I know I learned a lot from him..."

*Credit: https://min-eng.blogspot.com/2022/11/leonard-george-austin-1929-2022.html
https://www.statecollege.com/obituaries/leonard-g-len-austin/

CELEBRATING
THE INTELLECTUAL CONTIBUTIONS OF
LATE PROFESSOR LEONARD (LEN) G. AUSTIN

1929-
2022

To The Field of Comminution Processes

Dr. Bilgili's Introduction to the Particle Technology Area
"Pulverization and Agglomeration Under High Shear and Normal Forces"

Hamid Arastoopour
Linden Professor of Engineering and
Director of Wanger Institute for Sustainable Energy Research (WISER)
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Introductory Remarks

After Ecevit Bilgili received his B.S. degree in chemical engineering from Bogazici University in Istanbul, Turkey, in 1996 (graduating at the top of his class), he enrolled at Illinois Institute of Technology (Illinois Tech) for his graduate studies. I remember that Professor Ali Cinar introduced him to me and said he was an excellent student and was interested in the transport phenomena area. That semester, I was teaching a transport phenomena course and Ecevit agreed to be my teaching assistant. During the first week of classes, Ecevit exhibited exceptional intelligence, an incredible work ethic, and notable energy and motivation for learning, education, and research. I was very impressed by his knowledge and asked him to join my research group. Finally, I also convinced him to work with me on my National Science Foundation grant focused on pulverization of polymeric and elastomeric material for the purpose of recycling them to useful and valuable materials. I also convinced him that particle technology would be more interesting and challenging for him than traditional transport phenomena research dealing with gas and liquid systems. This was Ecevit's introduction to the world of particle technology research and education. I also asked Professor Barry Bernstein, one of the world leaders in polymer rheology research, to become Ecevit's co-advisor.

Ecevit was a very smart and creative researcher with a special interest in education and developing new approaches to teaching and research. As one of my Ph.D. students, he was never afraid to challenge me during our discussions and reviews. Ecevit made such strong and passionate arguments about his hypotheses and suggestions that he forced me at times to reconsider my own position. He did a superb job in his Ph.D. work and evidence of his outstanding research can be seen in his nine referred papers and two patents based on his Ph.D. research in the particle technology area. He received his Ph.D. degree in chemical engineering from Illinois Tech, Chicago, in May 2001. His Ph.D. work was acknowledged in 2004 by the Particle Technology Forum (PTF) of AIChE with the Best Ph.D. Thesis Award. His research shed fundamental light on the mechano-chemical nature of novel noncryogenic pulverization of rubber at high shear and compression, while producing partially devulcanized rubber particles by mechanically breaking the rubber crosslinks. The resulting partially devulcanized particles charted a new direction in the utilization and recycling of elastomeric materials.

Between 2001 and late 2004, Dr. Bilgili worked as a post-doctoral research associate at the Particle Engineering Research Center, University of Florida. His research interests centered on the processing, characterization, and modeling of particulate unit operations and powders. The experimental and theoretical work focused on powder flow in shear testers, nanoparticle production via stirred media milling, and population balance modeling of size reduction operations.

The last time I saw Dr. Bilgili was during a PTF dinner at the 2024 AIChE Meeting in San Diego. I asked him for his CV for the purpose of nominating him for the Illinois Tech Department of Chemical and Biological Engineering Distinguished Alumni Award. Ecevit was selected by the award committee to receive the distinguished award at Illinois Tech. Unfortunately, Dr. Bilgili passed away suddenly, and his daughter Melisa came to Illinois Tech to accept the award on behalf of her father on April 23rd, 2025.

In the following sections, a summary of Professor Ecevit Bilgili's research on "Pulverization and Agglomeration Under High Shear and Normal Forces" during his Ph.D. studies will be discussed.

Objective

Natural and synthetic polymer waste, including rubber, is increasing and environmental concerns regarding its disposition render recycling necessary. However, many reclamation processes to date have been limited to certain types of waste; have been uneconomical, particularly with respect to energy consumption; and have not provided reclaimed material in a form conducive for reusing manufacturing.

Thus, the object of Dr. Bilgili's Ph.D. research was to provide a low-cost, low-energy-consumption pulverization process for recycling of polymeric materials such as rubber, without changing its chemical structure, and thereby enabling reproduction of the rubber with mechanical properties substantially equivalent to the mechanical properties of the original rubber.

To achieve the above objective, a single screw extruder [1] was used to pulverize rubber granulates at high shear and compression without using cryogenic fluid for cooling. This process, solid-state shear extrusion (SSSE) pulverization [1-4] is based on the large compressive shear deformation of rubber granulates that results in the storage of a large amount of strain energy and the formation of cracks. When the stored energy reaches a critical level, the granulates cannot sustain themselves. As a result, the stored elastic energy is converted into surface energy through the formation of new surfaces and, in turn, pulverization occurs. The stored elastic energy is dependent on the viscoelastic response of rubber granulates to the processing condition.

SSSE Pulverization

The schematic geometry of the extruder channel is shown in Figure 1. The use of different pitched screws divides the extruder channel into three different functional zones. In Zone 1, the channel depth is constant and the granulates are dragged by the rotating screw with minimum compression. In Zone 2, the screws are designed to have a uniform taper, and the granulates are slightly compressed due to the decreasing channel depth. In Zone 3, the channel depth remains constant to exert maximum compression on the granulates. Compression ratio (CR) refers to the decrease of the channel depth for a specific screw geometry. The degree of compression of the particles is determined not only by the CR of the screw, but also by the size of the particles and other process variables, such as temperature, feed rate, rotation rate, etc. The relative motion of the screw with respect to the barrel wall results in the development of shear stresses in the rubber. Although Zone 3 is shown to be the place where most of the pulverization takes place, it is expected that some breakage of the particles may occur even in Zone 2, depending on the channel depth, process variables, and the size of the particles.

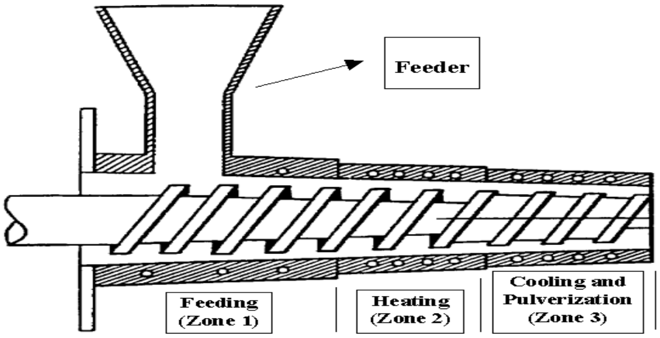


Figure 1. Schematic diagram of the single-screw extruder showing a cross-sectional view of the extruder channel geometry used in the pulverization of rubber.

Particle Size Distribution (PSD)

The cumulative and differential PSDs of the powder produced by the SSSE process are presented in Figure 2(a) and (b), respectively. Figure 2(a) shows a linear cumulative PSD on log-probability coordinates ($r^2=0.99$). This means that the SSSE powder obeys a log-normal PSD law. The mass geometric mean diameter and geometric standard

deviation were, respectively, 424 µm and 2.04. About 60 wt.% of the powder had a size of less than 600 µm. The powder exhibited approximately a unimodal size distribution, as seen in figure 2(b).

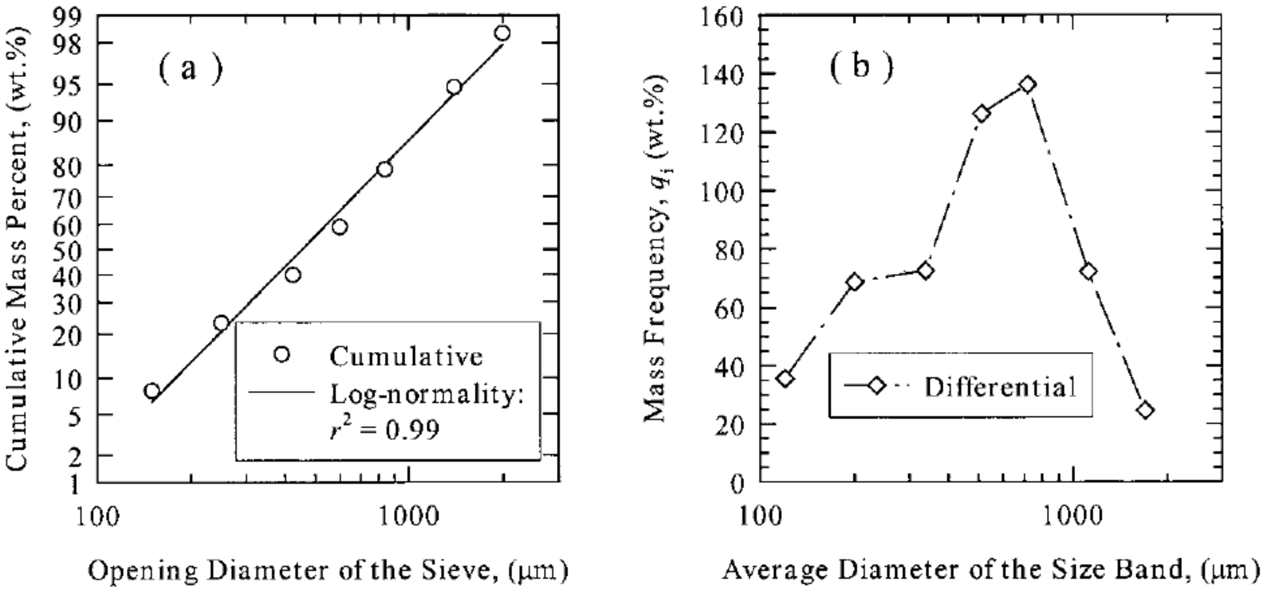
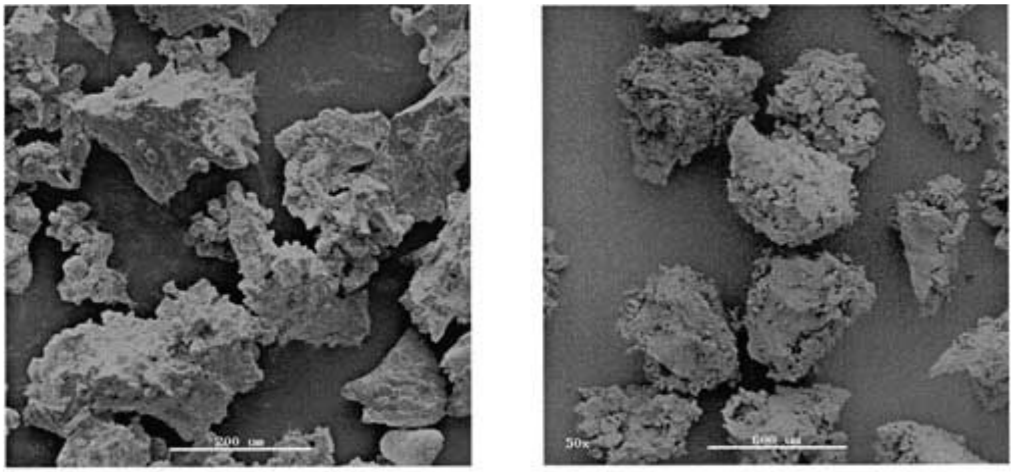


Figure 2. (a) Cumulative particle size (passing) distribution of the rubber powder on log-probability coordinates, and (b) differential particle size distribution of the rubber powder on semi-log coordinates.

Surface Characteristics of the Particles

The SEM images of the particles in the 90–250 and 425–600 µm size ranges of the powder are shown in Figures 3(a) and (b), respectively. The particles in the 90–250 µm size range had irregular shapes with nearly smooth surfaces. On the contrary, those in the 425–600 µm size range had more rounded shapes with convoluted surfaces and external porosity, probably due to the agglomeration of finer particles. Compared with the cryogenically ground rubber with a similar size range [5], the particles produced by the SSSE process had a greater specific surface area [6].



Marker size: 200 µm
(a)

Marker size: 600 µm
(b)

Figure 3. SEM micrographs of the rubber particles in (a) 90–250 mm size range, and (b) 425–600 mm size range

Crosslink Density

The granulates had a crosslink density of 66.9 mol/m^3. The particles in the 90–250 and 425–600 µm size ranges, that were produced via the SSSE process, had crosslink densities of 43.6 and 53.2 mol/m3, respectively. This indicates that some of the sulfur bonds in the network structure were cleaved (partial devulcanization) due to high stresses and large deformation imposed on the rubber. It appears that the SSSE process is mechano-chemical in nature [4,6]. Partial devulcanization significantly assists the particle bonding during the compression molding.

Partially devulcanized rubber particles produced by SSSE enable development of soft and flexible objects (i.e., slabs with no macroscopic defects), which indicates the possibility of producing soft rubber products from the powder without using virgin rubber. This was a significant step forward in recycling rubber and elastomeric materials (for more information, see Bilgili et al. [7]).

Conclusion

The pulverization of rubber granulates under high compressive shear was achieved using a single-screw extruder without using a cryogenic fluid for cooling in the SSSE process. A higher degree of compression of the granulates and significant cooling of the pulverization zone were the most significant factors in the successful pulverization of the granulates. Agglomeration of the produced particles, specifically the fine particles, was found to be competing with the pulverization process. The extent of agglomeration was observed to increase with a higher fraction of the fine particles, a greater degree of compaction, and a higher temperature of the pulverization zone.

The rubber granulates that experience high compressive shear strain can develop tensile stresses and store significant strain energy leading to the formation of new surfaces through the crack opening mechanism. Thus, there is a minimum size of the particles produced by the SSSE process that is determined by the processing conditions and the design of the extruder. In the SSSE process, more mechanical energy should be consumed without allowing the relaxation of stress in order to obtain a rubber powder with a smaller mass geometric mean diameter. This can be achieved by using a screw with a high compression ratio and sufficiently cooling the pulverization zone, making the rubber more solid-like, and resulting in the storage of a larger amount of strain energy.

The produced particles have more irregular shapes with rougher surfaces. The samples have a much larger total surface area than external surface area, which can be attributed mostly to the presence of fine carbon black particles embedded at the rough surfaces of the rubber particles.

References

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Comparison of Particle Breakage Models Accounting for Particle Size–Shape Evolution

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It was my privilege to co-chair this session at the last AIChE meeting with Ecevit. He consistently demonstrated a deep enthusiasm for the comminution and particle breakage sessions at the AIChE Annual Meetings. He was a steadfast advocate for advancing particle breakage research and engaged actively in scholarly discussions within the field. His graduate students were always exceptionally well-prepared, reflecting his commitment to mentorship and high standards in scientific communication.

Introduction

Solution crystallization is widely employed for purification in diverse industries, including pharmaceuticals, food processing, and fine chemical manufacturing. This approach is especially relevant when distillation is not feasible. Although the primary objective is purification, controlling crystal size and shape distribution is also crucial due to its impact on downstream operations. In particular, crystals with high aspect ratios are prone to clog filtration systems used to separate solids from liquids. Such crystal morphologies are frequently encountered in pharmaceutical processes. While breakage can be seen as a drawback—given that it generates fine particles that may obstruct filters—it can also be beneficial. High aspect ratio crystals suffer from poor flowability and filterability. Therefore, breakage of high aspect ratio crystals can improve flowability.

Despite numerous studies on how particle breakage alters particle size distributions (PSD), fewer studies have addressed the evolution of particle size–shape distributions (PSSD), particularly for high aspect ratio particles over time.

Experimental Methodology

Urea crystals were agitated in a slurry at 1250 rpm with 5g crystals per 100mL liquid. Breakage was monitored over multiple time intervals (0, 1, 5, and 15 minutes). Crystal measurements of major and minor axis lengths were taken before and after agitation. This allowed the determination of aspect ratios and particle size distributions (PSDs). Figure 1 below shows unbroken crystals and broken crystals after 5 minutes of agitation at 1250 rpm.

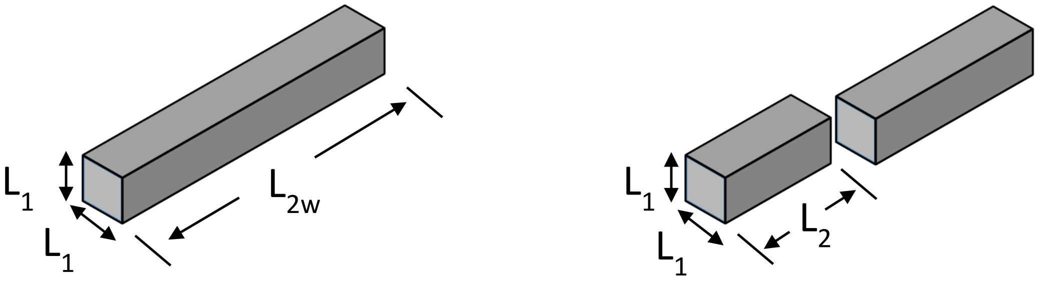
A. Unbroken crystals b. Broken crystals

Figure 1. Urea crystals: A. unbroken crystals, and B. broken crystals after 5 minutes of agitation at 1250 rpm.

Theoretical

In an idealized case, the height and width of an unbroken parent are equal and the length is is greater than the height or width. This is shown in Figure 2a where the parent particle major axis length is L_{2w}, and the height and width are L_1.

We consider the case of binary breakage perpendicular to the major axis as shown in Figure 2b. For this case the height and width of both child particles are L_1, the major axis length of one child particle is L_2, and the major axis of the other child particle is L_{2w} - L_2.



Parent particle

Child particles

Figure 2. Binary breakage of a high aspect ratio particle.

Results and Discussion

The resulting size PSDs from urea crystal breakage is shown in Figure 3. Note that the unbroken crystal PSD at t = 0 minutes is represented by bars for the number fraction in a given size interval where the width of the bar indicates the width of the size interval. For broken crystals at t = 1, 5, and 15 minutes, the number fractions are plotted at the center of each size interval and curves are added to guide the eyes. Data points with fewer than 10 particles were excluded to ensure statistical relevance.

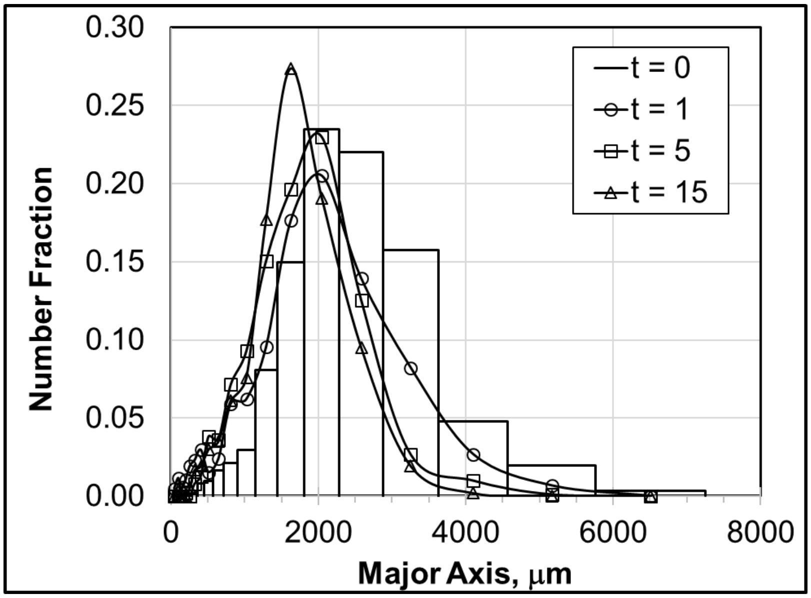


Figure 3. PSD for urea crystal breakage.

In Figure 4, the average aspect ratio of a size interval is plotted as a function of the major axis length. In general, the aspect ratio increases with the major axis length. To develop a general correlation, the average aspect ratios of the broken particles in each size interval were averaged and fit to a linear equation that resulted in

$$AR = 0.0013L + 2.1744 \qquad (1)$$

where AR is the aspect ratio and L is the major axis length. This is a simple empirical model.

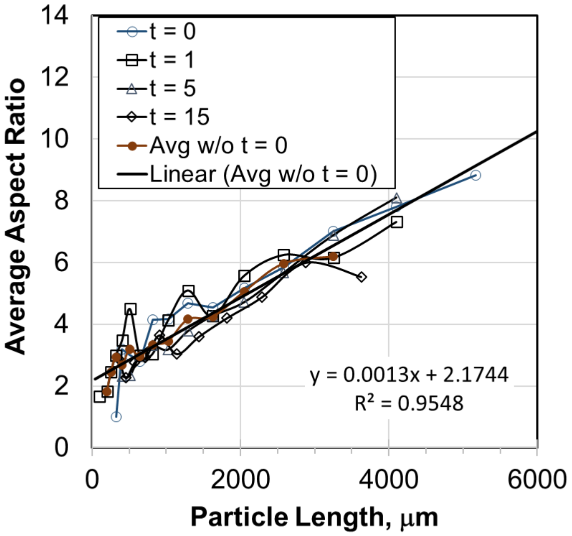


Figure 4. Average aspect ratio of a size interval as a function of major axis length.

Model Comparison

Both the idealized model of breakage perpendicular to the major axis (Model in Figure 5) and the empirical correlation given in Equation (1) (Empirical in Figure 5) are modelled using population balance equations and compared with data. Note that after 1 minute of breakage (t = 1) the results from Model and Empirical are very similar to the experimental data. However, this changes after five minutes of breakage. After 5 minutes of breakage (t = 5), the empirical equation is closer to the experimental results for aspect ratio prediction.

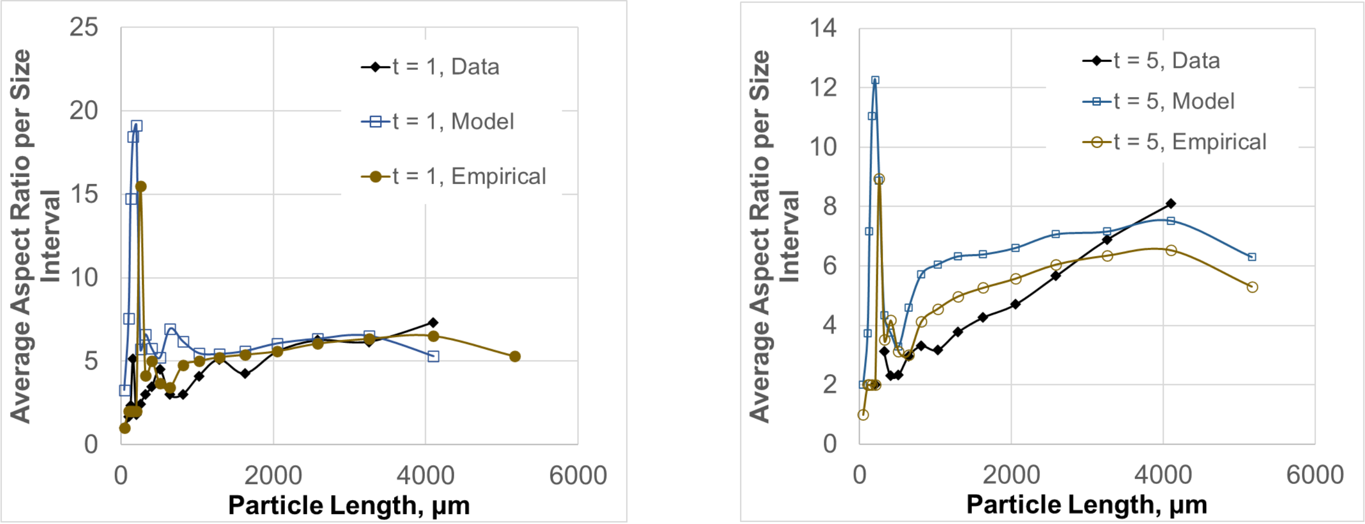


Figure 5. Model comparisons after 1 and 5 minutes of breakage.

Both models captured general trends but were limited by assumptions—such as binary breakage and shape uniformity. Notably, breakage is more complex than simple theoretical models can predict.

Concluding Remarks

This study highlights the nuanced relationship between particle breakage, size, and shape in crystallization systems. While theoretical and empirical models provide useful approximations, experimental data reveal complexities in fracture behavior that warrant further study—particularly for particles with high aspect ratios.

Breakage of Structured Particles Through Crack Propagation Phase-Field Model (PFM) Prediction Coupled with Population Balance Modeling

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The comminution community has lost an incredibly talented ally with the passing of Ecevit Bilgili. First and foremost, Ecevit was a wonderful father and husband. He was extremely proud of all the members of his family. If you spent any time conversing with him, the discussion always included some new achievement of at least one of his family members. They were his world!

The comminution community has often approached breakage from a correlation point of view to find breakage kernels. But Ecevit was never afraid to reach a bit deeper with a goal to discover a method to predict breakage from first principles. He was an avid supporter of using population balance modeling to understand breakage, but extended the population balance modeling concepts to allow for more complex physics-based phenomena. This almost always required the direct solution of the population balance model rather than approximate forms.

Ecevit was dedicated to helping ALL of his students. He was a tough professor as he challenged his students to think and thereby discover. I remember that we discussed one student's educational path and project and he said "He does not get it." But then, with that twinkle in his eye and a smile on his face that we have all seen, he said "but he will." He then proceeded to spend the extra time to help that single student bridge the knowledge gap and, in no time, his student "got it" and excelled. Ecevit could not be prouder of that student's accomplishments.

When he noted a shortfall in recognition of young students studying comminution, he was relentless in suggesting that PTF provide an award highlighting young researchers of excellence in the area of breakage and comminution. Perhaps now is the time that we as a community might consider doing that in his memory. The following is a summary of a talk given in the last session Ecevit chaired with PTF. Ecevit discussed this with me after the conference and said, "This is a good start. You may be onto something here. Let's pursue this." Great praise coming from him. He will be greatly missed.

Introduction

Population balance models are powerful tools that relate the breakage observed to the probability that stress-stain events may induce the breakage of a given sized particle. Population balance models relate the observed breakage to how a particular particle might break. They help determine how, or how much of, a particle of size bin 1 will break into size bin 2, size bin 3, size bin 4, etc. The trouble is that these population balance models are not predictive. They can be used to analyze a set of batch breakage data or even flow data and determine how and at what rate the various-sized particles might break, but they cannot take a look at a set of particles interacting and determine what particle shapes and sizes might result in the most probable breakage scenario. That scenario depends on the fracture pattern that any particle might have when subjected to stress-strain and/or impact conditions. The fracture pattern depends on the shape of the particle that breaks, how the particle interacts with other particles during stress-strain events, the texture of the particle surface, and the internal structure of the particle.

Consider a rod-shaped tablet made from using a compression technique. The fact that the particle is rod-shaped suggests that the particle may self-align with other rod-like particles to minimize the shear during a stress-stain event. In this case rods will slide over each other. However, the rod-shaped nature of the particles also suggests that during a shear event the particle may also orientate where it is supported by interaction with two close neighbors much like a simple beam with two supports (Figure 1). In this case, the shape of the particles determines where a particle will break during shear flows. The maximum bending moment (M_{max}) is the point where a structural component is most stressed in bending. Failure will likely occur at that point if the stress within the particle surpasses the material's failure strength. The actual failure point will be determined by factors such as material type, loading conditions, and the presence of defects. But if breakage does occur, the size of the resulting fragments will be determined primarily by the placement of the forces on the particles. Breakage will occur at or near force Q.

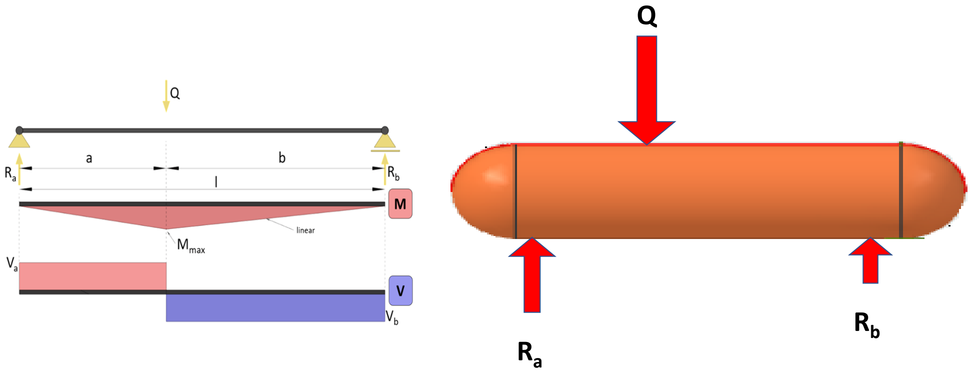


Figure 1. Stresses acting on rod-shaped particle

Next, consider what may happen if the particle shapes were a series of crosses. As the series of cross-shaped particles interact, a single cross-shaped particle could also be supported by two or more particles during a shear event. The resulting stresses could easily induce fracture of one leg of the cross converting a cross-shaped particle to a T-shaped particle. Now suppose the particles are shaped like pretzels or curved potato chips with ridges or other complex particles. During a shear event these more complex particles could be supported by 3 nearest neighboring particles inducing some stress pattern in the particles that would depend on the structure and strength of the particle, as well as the contact points acting on the particle (Figure 2).

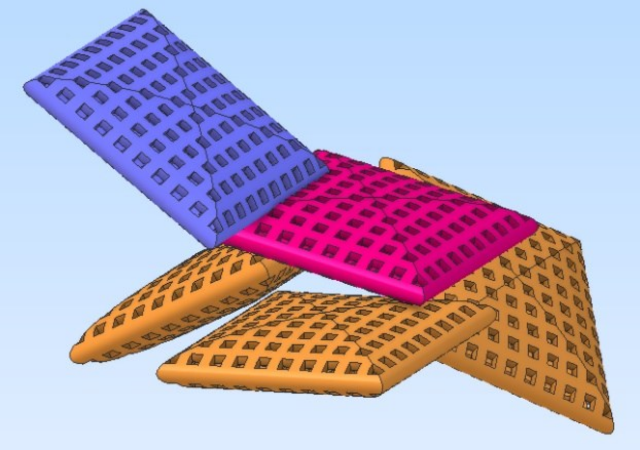


Figure 2. Interaction of more complex particles red particle is acted upon by contact with the blue particle and supported by the three orange particles

Almost all materials subject to breakage have some form of associated structure. It is, therefore, reasonable to suggest that the breakage selectivity in the population balance model could be predicted from first principles if we could define a mathematical structure to capture the shape of the particles of interest and the internal structure of what makes up the particle. Here is where finite element (FEM) and phase-field modeling come into play. The FEM can compute the expected forces and stresses on any complex shape based on simple interactions on the particle surface.

In a very simple example, consider a long slender rod-shaped particle supported between two contact points. As an additional particle presses on the rod it breaks at the prescribed location where stresses are greatest predicted by an FEM analysis or simple solid mechanics computations.

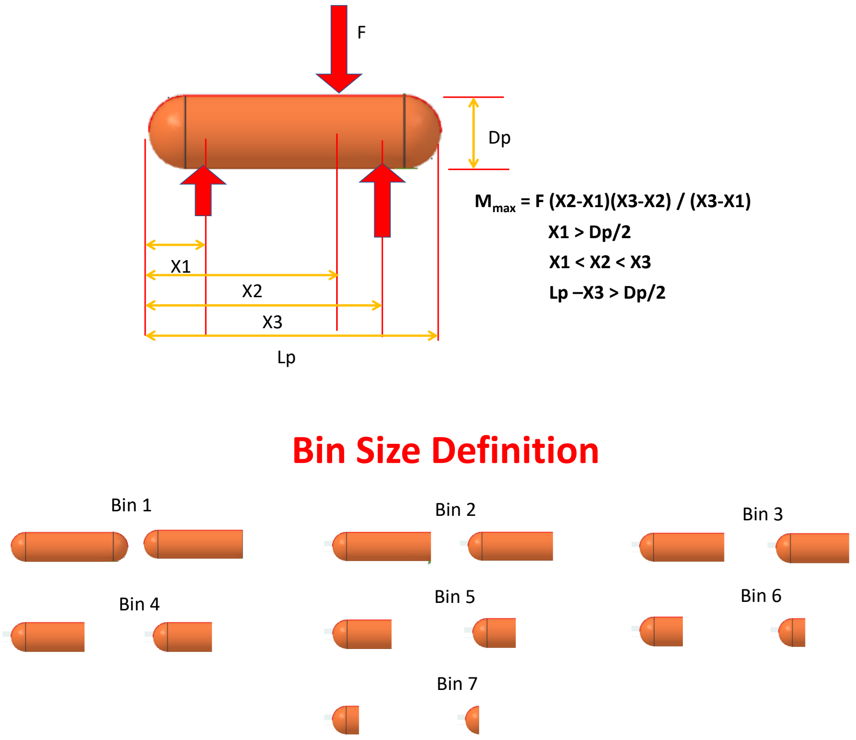


Figure 3. Simple case of rod-like particles

If the above criteria (Figure 3) are met, a particle will always break at X2 – giving rise to particles with length X2 and Lp-X2. One can randomly select the X1, X2, and X3 values and generate breakage points for any length particle Lp with diameter of Dp. Creating a histograph of the lengths generated by this analysis gives a selectivity function for how the original rod-shaped particle breaks. This can be done for any length of partcle. Thus, one can select a set of particles of diffrent lengths (particle size bins) and generate the aproximate breakage selectivity matrix for rod-shaped particles to be used in a population balance model for this particular set of particle size bins. The advantage is that the breakage selectivity function was computed using a simple solid mechanics stress analysis and not inferred by the solution of the poulation balance model. The first step in setting up a population balance model is to define a set of particles of different sizes based on some criteria. This will create a set of particle size bins. A population balance model is a set of differential equations that relate how particles in a given size bin transfer through a process with imposed breakage events. The mass of particles in a given bin size is typically expressed as a funtion of time. But if the breakage events are due to imposed strain events at a constant stress,, and those strain events are continous and increase linearly with time, then the mass balance can be expressed in terms of the total strain on the material without changing the form of the equations. As material is subjected to a given strain at a constant stress some of the particles will break and some will remain unbroken. Of those that break, particles are created in each of the smaller particle size bins. Figure 4 shows a schematic of the particle breakage process. The S-values represent the overall breakage rate of a given size particle. The b-value matrix determines what fraction of the broken particles end up in the various size bins that are smaller than the i^{th} particle size bin. These b-values are the breakage selectivity parameters or matrix.

$$\frac{dm_i(\varepsilon)}{d\varepsilon} = -S_i m_i(\varepsilon) + \sum_{j=1}^{i-1} b_{ij}\, S_j m_j(\varepsilon),$$

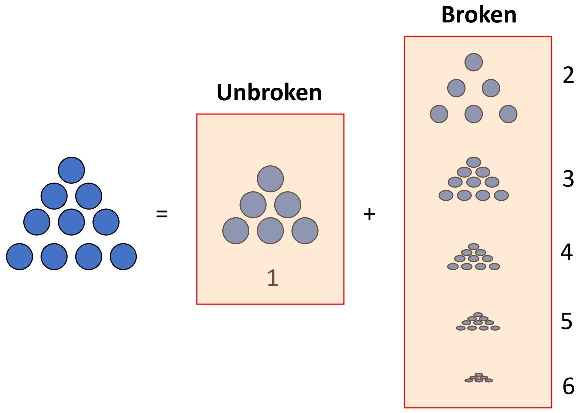


Figure 4. Population balance model

The population balance is a method to track the size of particles as they pass through some process that may induce breakage. The mass of particles in any bin size is given by m(ε). To solve the population balance model one needs to generate a series of particle size data as a function of total strain (ε) where the material is subjected to a continual strain at a prescribed stress value as a function of time using a shear box that induces nearly perfect shear on the particles within the box (Figure 5).

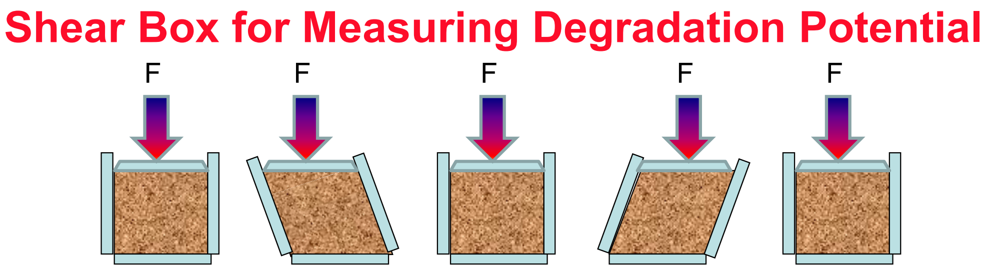


Figure 5. Schematic of Stress Strain Breakage

It was observed that the data showed a dual rate behavior where the initial breakage was quite fast, followed by much slower breakage rates. From this data, it was determined that the initial breakage was due to breakage of structured particles, and the secondary breakage was due to attrition of shards created by the primary breakage. The approximate solution to the cumulative formulation (Kapur, 1970) was used to determine the breakage rate and selectivity parameters for the initial and secondary breakage. Only the initial breakage parameters were considered.

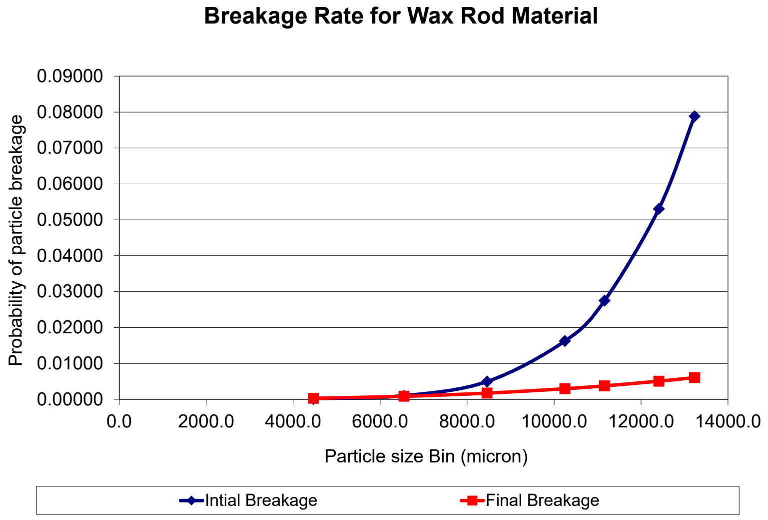


Figure 6. Breakage rates parameters for rods (S-values)

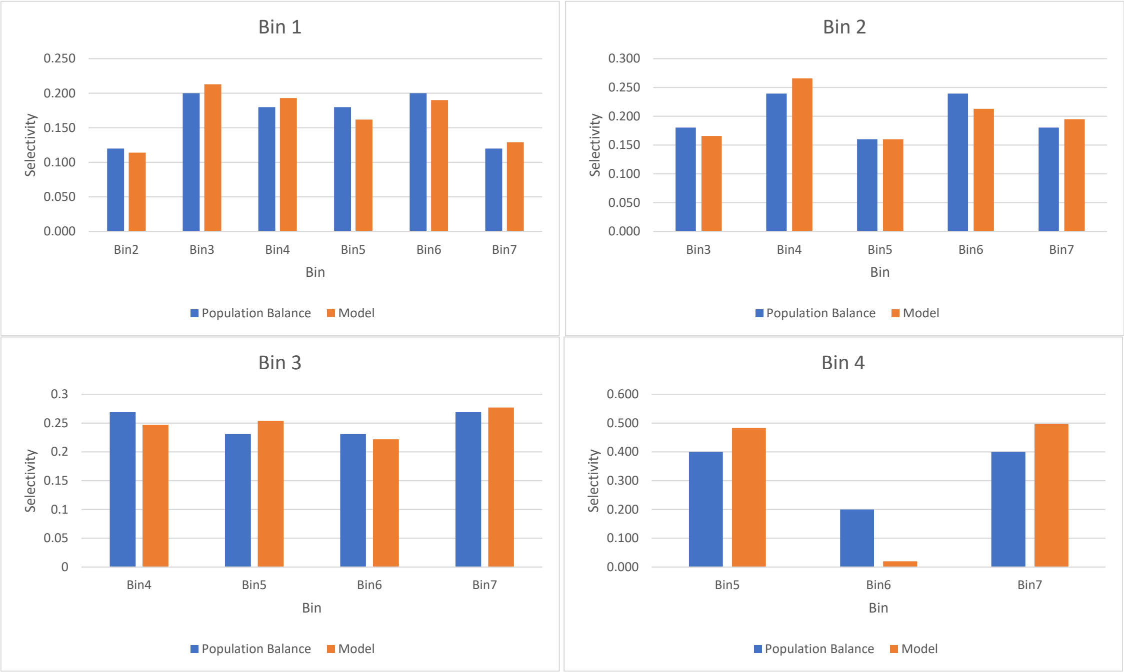
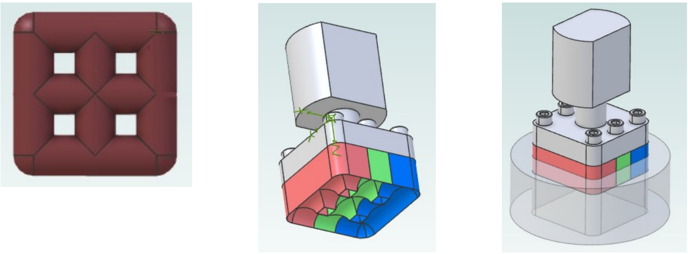


Figure 7. Selectivity parameters for rods (b-values)

There is good agreement between the selectivity matrix for breakage computed from the population balance model and the computed frequency of the breakage of the rods from the solid mechanics model for rods. Now consider more complex systems. More complex structures can be examined using FEM to predict stress on the particles. FEM can be used to estimate the shape of the fractured particles when particles are subjected to stress-strain events. This FEM analysis can then be used to predict selectivity parameters for more complex structured particles. A crack will begin to form when the local stresses (computed by FEM analysis) approaches the yield stress. However, the problem is that FEM analysis requires a continuum to compute the stress and strains. The act of breakage induces gaps in this continuum, thereby making a FEM analysis difficult to accomplish without complex and continual regeneration of meshes as new particles are fractured or as cracks grow in the particle of interest.

This complexity can be modeled using a phase-field model (PFM) to represent a crack geometry in a diffusive way without introducing sharp discontinuities. This enables PFM to model crack propagation using standard FEM. Thus, how a crack will form and propagate in a particle of a given configuration can be predicted. Applying a series of allowable contact points and positions to structured particles, and then using PFM to determine the location of the crack, allows engineers to develop a model that relates shape, powder properties, and potential particle-particle contact patterns to the breakage of structured particles. The powder flow properties help predict the breakage rates in the population balance model while the FEM stress analysis and PFM crack propagation pattern helps predict the breakage selectivity. Thus, this approach allows the population balance model to be predictive from first principles.



Grid Pellet Press

Create the pellets by compressing powder wax to 2100 kPa in a grid mold (pretzel shaped) to give particles with a yield strength of 400 kPa

Figure 8. Creation of pretzel-shaped wax particles

Consider pretzel-shaped wax particles (Figure 8). These wax particles were subjected to several stress-strain conditions using the technique highlighted in Figure 5. This method generated particle size data as a function of total strain on the pretzel-shaped wax particles. It was also observed that the data for pretzel-shaped wax particles showed a dual rate behavior where the initial breakage was quite fast followed by much slower breakage rates. It was determined that the initial breakage was due to breakage of structured particles, and the secondary breakage was due to attrition of shards created by the primary breakage. Similar to the rod-shaped particle work, the approximate solution to the cumulative formulation (Kapur, 1970) was used to determine the breakage rate and selectivity parameters for the initial and secondary breakage. However, only the initial breakage parameters were utilized. Consider the results of an FEM analysis to breakage of pretzel-shaped wax particles. The basic approach used to predict the breakage selectivity parameters begins with a random selection for the placements of the contact load and support load points (Figure 9).

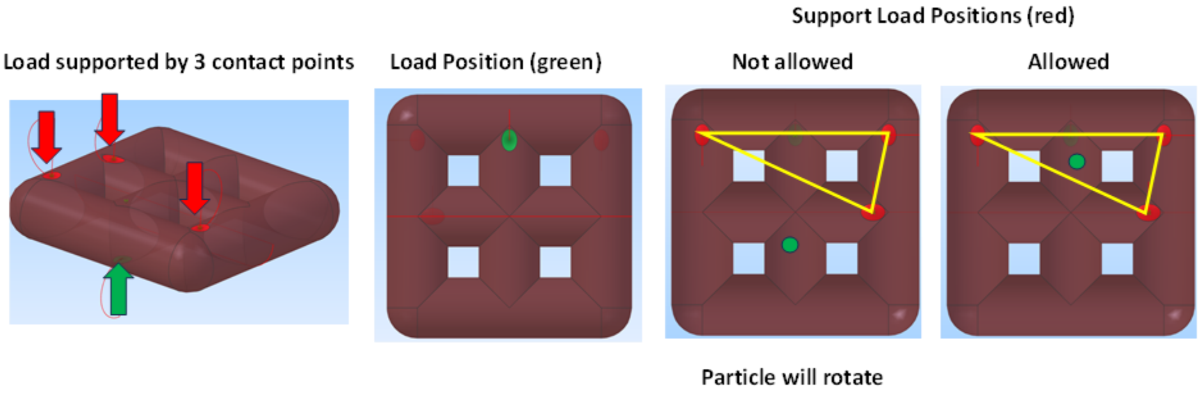


Figure 9. Random selection of the main contact force and the resulting support force for FEM analysis

Some of these random selections would result in unbalanced acceleration on the particles and were excluded from FEM analysis. FEM analysis was then used to compute stress in the particles. This analysis gave contours for the yield stress condition in the particles (Figure 10). Cracks propagated along these contours. Thus, two or more new particles were generated by the single FEM analysis. Repeating this process many times creates a map of how particles might break when subjected to stress-strain events. Each FEM analysis created at least two new particles of different sizes based on the location of the load contact points. A histogram of the data generated from all FEM analyses performed gives a distribution of the breakage fragments obtained from the breakage of the original particles (Figure 11). The selectivity parameters computed from the population balance model were compared to the selectivity parameters computed from FEM analysis. There is good agreement between these selectivity parameters, indicating this may be a reasonable method to estimate the mode of breakage of particles from first principles. It definitely warrants a closer look.

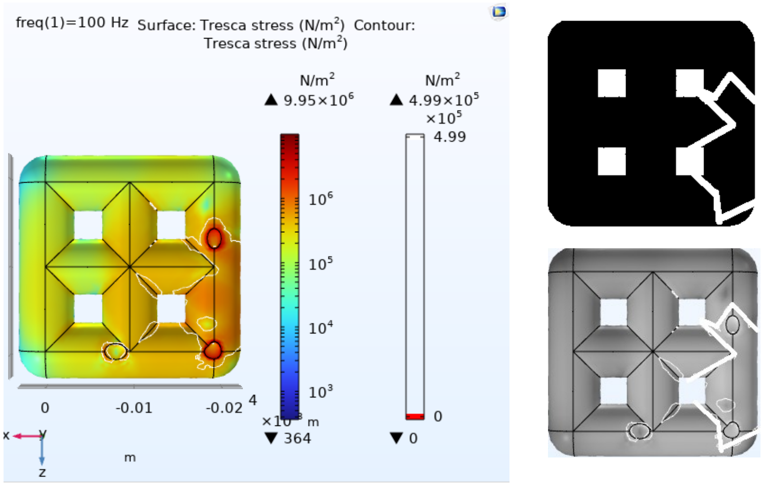


Figure 10 FEM analysis on pretzel-shaped wax particles showing preferred breakage

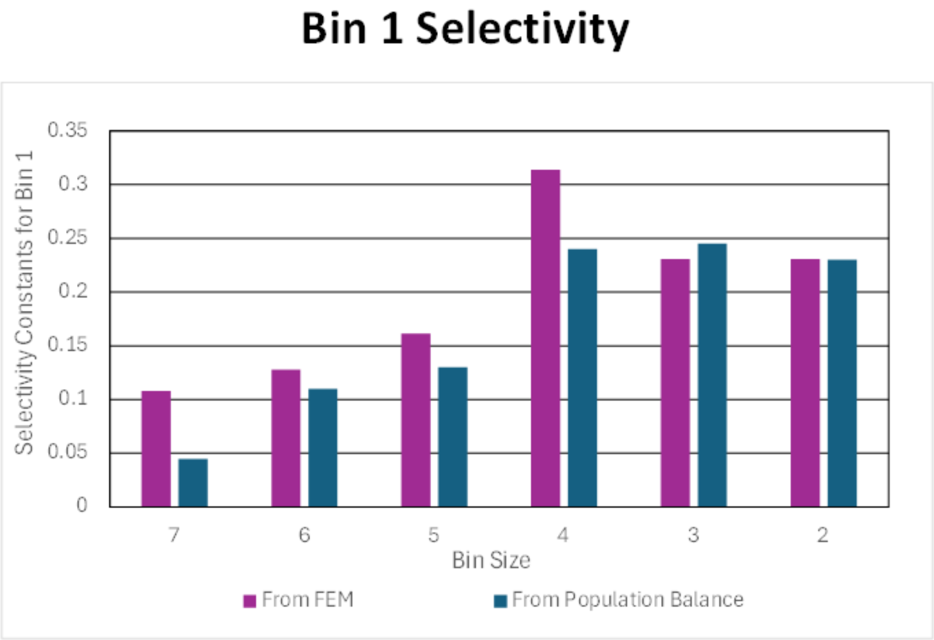


Figure 11. Comparison of breakage selectivity parameters from FEM analysis and population balanced modeling on pretzel-shaped wax particles

Polymeric films embedded with poorly water-soluble drugs for bioavailability enhancement

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Borrowing a word from my colleague Prof. Piero Armenante, the one that comes to mind when thinking of Ecevit is ***"passion."*** *I first witnessed this nearly 20 years ago at various AIChE PTF sessions—his questions, discussions, and presentations were always full of energy and enthusiasm. It was immediately clear that he would become an outstanding teacher—and indeed, he did. His passion, along with his excellent work with Prof. Brian Scarlett, his industry experience, and his clear potential as an educator, all motivated me to recruit him as a junior faculty member at NJIT when a dedicated position in particle technology became available. As outlined below, this decision led to a wealth of productive research, collaborations, and memorable travel experiences over the next 15 years. One that stands out is a trip we took to India in early 2020—just before the pandemic began. Ecevit loved Indian food and thoroughly enjoyed every part of that trip. I'm so glad that he had the chance to make that trip.*

Background

It is rare to find oneself in the right place at the right time. I was fortunate to be in that position 25 years ago, when I had the opportunity to organize and lead a strong team of colleagues from Princeton and Rutgers to secure funding for the New Jersey Center of Excellence in Engineered Particulates (NJCEP). That initiative sparked my involvement in nanotechnology research in the early 2000s, focusing on industrially relevant problems such as fluidization of nanoparticles, coating of nanoparticles with polymeric films, environmentally benign mixing of nanoparticles, and nanoparticle formation via supercritical fluid technology. This foundation led to a leadership role as a founding Co-PI in forming a multi-university National Science Foundation Engineering Research Center (NSF-ERC), launched in the summer of 2006 as a partnership between Rutgers, NJIT, Purdue, and U. Puerto Rico at Mayagüez. As the leader of a Research Thrust and a Technology Testbed, I was responsible for shaping a multidisciplinary team to work on the bioavailability enhancement of poorly water-soluble drugs—a critical area for pharmaceutical innovation at that time. As part of building that team, I recruited Late Dr. Ecevit Bilgili to NJIT in Fall 2009. Naturally, he greatly benefited from the significant resources made available through the ERC; one of the most substantial start-up packages at the time, active projects already in motion within the Engineered Particulates Thrust and Strip Film Testbed, and talented graduate students and postdocs funded by ERC. All of that resulted in a decade of highly productive research, greatly enriched by students and postdocs such as, Dr. Sameer Dalvi, Anagha Bhakay, Mohammad Azad, Scott Krull, Dr. Lucas Sievens, Dr. Ramani Susarla, Dr. Jun Zhang, Dr. Catharina Knieke, Lu Zhang, and Meng Li. What follows is a summary of the collaborative research related to **polymeric films**, developed over years of solution-driven innovation.

Nano and micro drug particles and suspensions: Process intensity and functionalization

The greatest challenge to the delivery of poorly water-soluble drugs, i.e., active pharmaceutical ingredients (APIs), is their inherently limited dissolution and bioavailability. The traditional way to tackle this issue relies on particle size reduction to improve the dissolution rate of poorly water-soluble compounds by increasing the surface area-to-volume ratio of the particles. However, increased surface area to volume leads to increased inter-particle attraction leading to formation of aggerates, bringing in the new challenge of stabilizing and incorporating the fine drug particles in a solid dosage form, from which they can be recovered upon delivery while maintaining their enhanced dissolution during manufacture and release. Our work has shown that regardless of the path, bottom-up or top-down, to fine particle production, process intensification and surface functionalization are key to producing stable fine particle suspensions[1] or non-aggregated dry powders[2]. Preparation of particle suspensions via bottom-up approach of liquid anti-solvent (LAS) precipitation is nicely illustrated in Fig. 1, where fine particles of griseofulvin (GF), a poorly water-soluble drug, were prepared in a T-mixer. Particle size reduced as ultrasound based process intensification was applied, and stabilizers were added. The finest, nano-sized GF particles were produced when highest intensification through increased jet velocity and a combination of two stabilizers were applied providing electro-steric stabilization. This and other papers from our group uncovered nuances of the crystallization process and highlighted the need to judiciously select the type of stabilizers. That topic was further investigated with the intent to find a way for rapid screening of stabilizers via atomistic simulations[3]. This approach allowed identification of the potential synergism of additives, which was evaluated by the simulation of binary mixtures containing the non-ionic additives like polymers with the anionic surfactant, validated by experiments. That work followed several milestone papers[4,5,6], further establishing the role of process intensification and stabilizers in LAS particle formation.

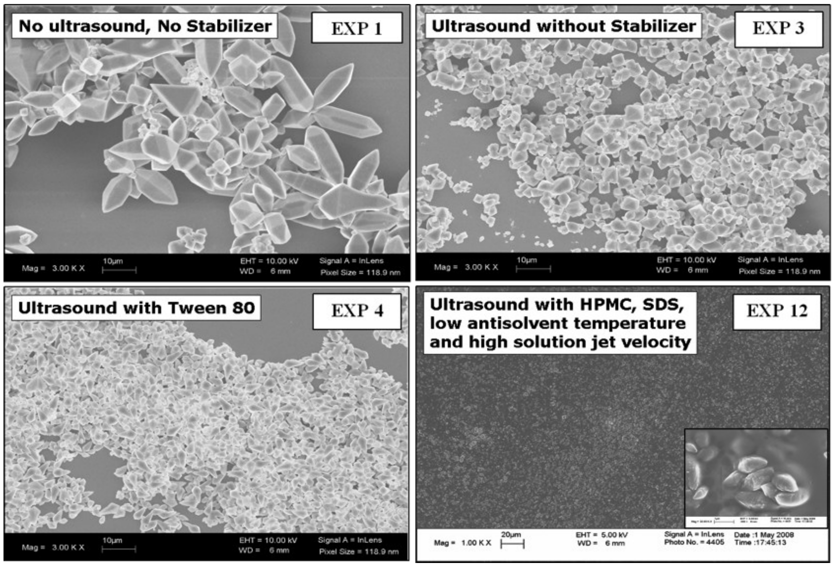

Figure 1. GF micro/nano particles via antisolvent precipitation in a T-mixer.

Both process intensification and stabilizers were also needed in the melt-emulsification process to form very stable fenofibrate (FNB) suspensions. FNB has been reported to be challenging in terms of lack of sustained particle size stability, whereas our work[7] showed that the suspension stayed stable for a week. The time stability data without and with sonication are presented in Fig. 2. Most interestingly, the same principle applies to top-down milling approach, for example in nanomilling through wet-stirred media milling (WSSM). As shown in Fig. 3, for WSSM of GF, the cumulative particle size distribution (PSD) shifts leftwards indicating finer particle sizes as stabilizers are added; the best outcome occurred when a surfactant was combined with a polymeric additive[8] leading to highly stable 200 nm sized GF nanosuspensions. Subsequent work in nanomilling led to a discovery of a new suspension stabilization method which does not require any surfactants. In this innovative, patented approach, a combination of water-soluble adsorbing polymer and a water-insoluble swellable polymer were used and milled together with the drug to form stable suspensions[9]. Please refer to a review paper[10] to learn more about our comprehensive body of work in this area. In the next section, our work in forming polymeric films embedded with such nano and microparticles is presented. As a novelty, we show that while use of stable nanosuspensions is an attractive approach to preparing fast dissolving thin films of poorly water soluble particles, surface modified dry micronized drug particles also provide the same level of dissolution enhancements[2]. Drug particle preparation is greatly simplified in this innovative approach where larger micro-sized pre-blended with silica nanoparticles are simultaneously milled and surface modified[11].

Polymeric films embedded with nano and micro drug particles

Polymeric films embedded with drugs typically use solvent casting technique, which involves dissolving either a water soluble drug and polymer in water or a poorly water-soluble drug in organic solvent. Although that is apparently straightforward, films made through solvent casting pose challenges with respect to API particle size and form control because the API is likely to recrystallize upon drying the prepared film. Solvent casting also imposes a potential limit on the drug loading in the resulting film, as exceeding this limit may exasperate the problems of uncontrollable precipitation of drug particles upon removal of organic solvent during drying. To avoid this issue, our team came up with the slurry casting technique, which was intended to preserve the drug particles in the crystalline state, suspended in an aqueous solution prior to film manufacture. Two early and influential publications[12,13] discussed next, paved the way to numerous significant advances by establishing a novel and practical platform for delivering poorly water-soluble

(BCS Class II) drugs using polymeric strip films incorporating drug nanoparticles. That work addressed the dual challenge of enhancing drug bioavailability while maintaining formulation stability, manufacturability, and patient compliance.

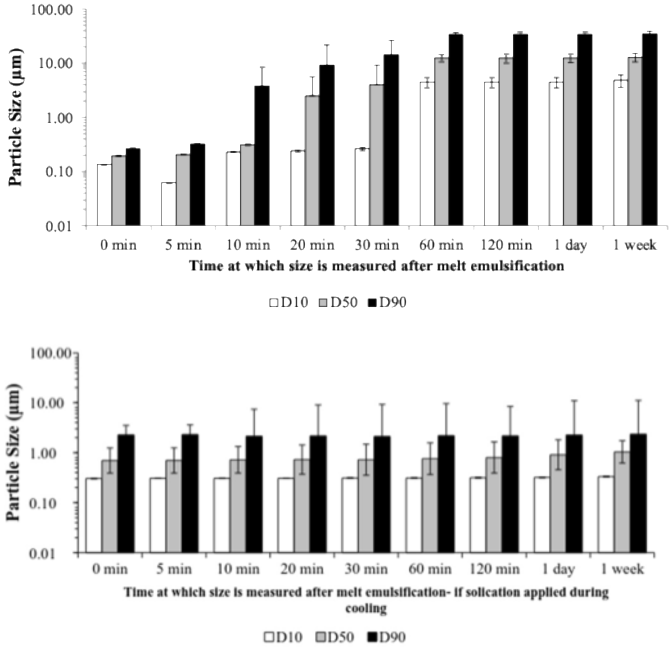

Figure 2. FNB micro particles via melt-emulsification. (Top) No sonication applied; (Bottom) Sonication applied during cooling.

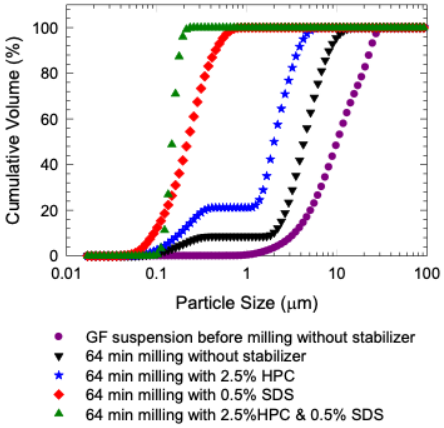

Figure 3. Critical role of stabilizers during wet milling of GF particles.

The first study[12] focused on developing hydroxypropyl methylcellulose (HPMC)-based polymeric films containing nanoparticles of three poorly water soluble drugs; griseofulvin, naproxen (NPX), and fenofibrate (FNB). A top-down WSSM approach was used to produce stable nanosuspensions, which were then incorporated into HPMC matrices through a casting and drying process. The films were uniform, flexible, and showed high drug loading without compromising film integrity. Critically, the embedded nanoparticles remained stable in size and morphology after film formation and storage, confirming that the polymer matrix effectively preserved nanoparticle dispersion, see Fig. 4 depicting 2D near-IR imaging having uniform FNB distribution and lack of any large agglomerates. Uniform drug distribution was also validated thorough excellent drug content uniformity, which is a major regulatory requirement. Dissolution testing confirmed fast, immediate release despite poor water-solubility, indicating that the large nanoparticle surface area was preserved during mixing, casting and drying without formation of any irreversible aggregation.

The second paper[13] extended this concept by demonstrating that polymer strip films could serve as a **surfactant-free** and **robust** oral dosage form for drug nanoparticles, eliminating the need for excipients typically required to stabilize nanosuspensions. The study evaluated two additional BCS Class II drugs and polymers, showing that even in the absence of surfactants, film-based delivery maintained nanoparticle stability and facilitated rapid redispersion upon hydration. This work emphasized the importance of polymer selection, film processing conditions, and mechanical properties in ensuring successful formulation performance. Five drugs utilized in this work had a wide range of solubilities, yet the drug dissolution rate for all five cases was fast and nearly identical (Fig. 5), indicating matrix erosion based drug release, hence a robust platform technology. Together, these two papers highlighted several key innovations:

- **Stable nanoparticle incorporation**: The films preserved the state of drug nanoparticles during processing and storage, avoiding aggregation or crystallization.
- **Improved redispersibility**: Upon exposure to aqueous buffer, the nanoparticles were rapidly released, maintaining their size and dissolution advantages.
- **Surfactant-free Formulation:** Removing surfactants simplified the formulation, reducing regulatory complexity and potential toxicity concerns

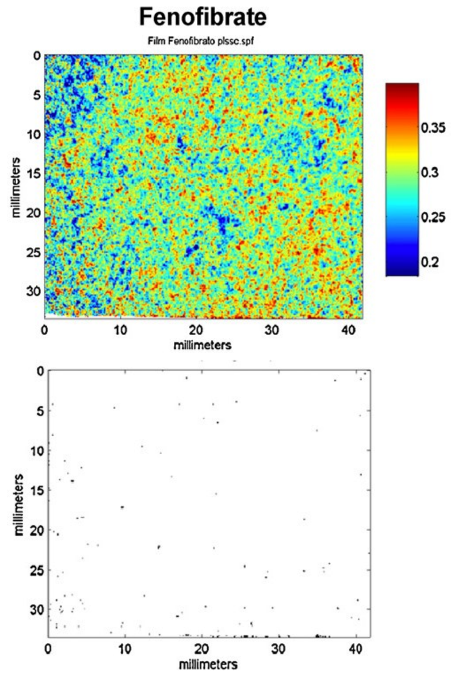

Figure 4. Near-IR imaging (top) of FNB in dried film indicates uniform drug distribution and lack of aggregate formation (bottom).

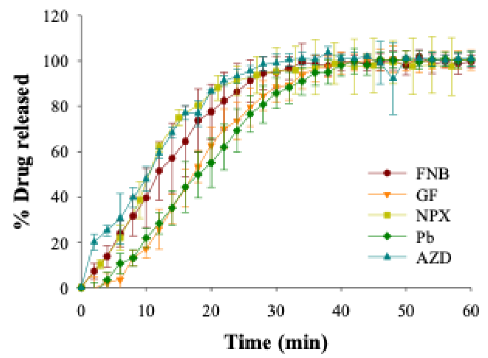

Figure 5. All five APIs exhibited similar, fast, immediate release profiles.

Next, our team tackled the drying process and in-line film product quality sensing relevant to film manufacturing. A schematic diagram of an integrated manufacturing and sensing is depicted in Fig. 6. Fast film drying of nano-drug particle laden stripfilms formed using water-soluble biocompatible polymers via forced convection was studied by building an instrumented drying apparatus[14]. The purpose was to better understand and control the drying process for forming films having uniform drug distribution and fast dissolution. The effects of convective drying parameters, temperature and air velocity, and film-precursor viscosity on film properties were investigated. Two major drying regimes, a constant rate period as a function of the drying conditions, followed by a single slower falling rate period, were observed. Films dried in an hour or less without any wrinkles or irreversible aggregation of GF nanoparticles with low residual water content. Near-infrared chemical imaging and the drug content uniformity analysis indicated a better drug particle distribution when higher viscosity film-precursors were used. Powder X-ray diffraction showed that the GF in the films retained crystallinity and the polymorphic form. USP IV dissolution tests showed immediate release (~20 min) of GF. Overall, the films fabricated from polymer-based suspensions at higher viscosity dried at different conditions exhibited similar mechanical properties, improved drug content uniformity, and achieved fast dissolution. Subsequently, model-based understanding of film drying was developed. First, a rate-based semi-empirical model was formulated, and parameters were estimated by integral method of analysis using a coupled optimizer–ordinary differential equation solver[15]. Next, a detailed transport model[16] was presented to capture moisture diffusion, heat transfer and moving boundary in convective drying of films loaded with GF. It incorporated a solvent diffusivity model based on free-volume theory. The transport model, whose diffusivity parameters were estimated using drying data at a reference condition, was validated at different drying conditions and wet film thicknesses. It delineated underlying mechanisms of drying kinetics, demarcating a smooth transition from constant-rate to falling-rate period.

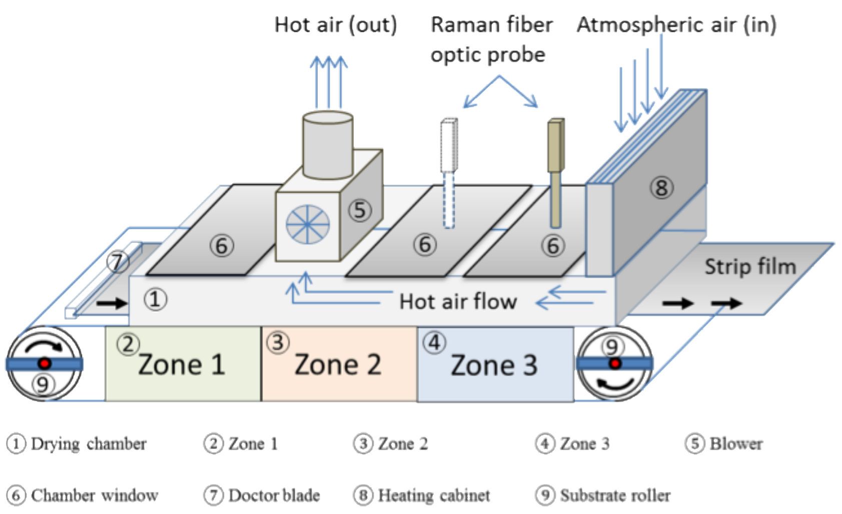

Figure 6. A schematic of film casting, drying, and sensing. This set up can also incorporate near-IR sensing to detect moisture content and film thickness.

Our team continued to build the knowledgebase and regulatory science including process analytical technology (PAT) and sensing for this new class of nanoparticle-based oral film dosage forms, combining the benefits of nanotechnology with a user-friendly delivery vehicle. Examples of work include:

- Better understanding of the role of viscosity enhancers including use of superdisintegrants (SDIs) as viscosity enhancing agents (VEAs)[17]. As a novel outcome, inclusion of SDIs into HPMC–GF films lead to enhanced viscosity without any negative interaction with GF particles, leading to *stable*, well dispersed suspensions, which upon conversion into dry film dosage form resulted both in excellent drug content uniformity and full recovery of GF nanoparticle size upon redispersion. Although the use of SDIs as VEAs led to higher viscosity, it did not have a significant impact on film mechanical properties and resulted in fast drug dissolution. Thus unlike traditionally used VEAs that significantly slow down drug release, SDIs had a positive impact, thereby establishing their novel use as economical VEAs in aqueous low MW polymer based film formulations, exhibiting dual advantage of maintaining drug content uniformity as well as fast and immediate drug release.

- As a novelty, pullulan was established both as a steric stabilizer during the WSMM of GF and as a film-former in the preparation of strip films via casting–drying the drug slurries for dissolution and bioavailability enhancement[18]. The study identified the effects of drug loading and milling time on the particle size and suspension stability, as well as the concentration of xanthan gum as a VEA and casting thickness on film properties and dissolution rate.

- A series of papers establishing the regulatory science behind various critical material attributes (CMAs) of film mechanical properties including dissolution from poorly water soluble drugs: the effects of addition of different types and amounts of plasticizers[19]; the complex and confounding effects of the film forming polymer molecular weight (MW)[20]; and the effect of the drug nanoparticle loading[21] where the most interesting outcome was the retardation of drug dissolution rate as drug loading increased; while establishing that as much as 50 wt % drug can be loaded into thin polymeric films.

- A detailed study[22] demonstrated the integration of Raman spectroscopy as a real-time Process Analytical Technology (PAT) tool in the manufacturing of drug-loaded polymeric strip films. The primary objective was to monitor critical quality attributes (CQAs) such as drug content uniformity, form, distribution, and potentially film thickness during continuous film casting. The research showed that non-destructive, real-time spectral measurements could accurately track drug form and concentration across both the width and length of the film, enabling rapid detection of variability due to edge effects, process transients, or formulation inconsistencies. Spectral data were strongly correlated with off-line HPLC measurements, confirming Raman's suitability for quantitative analysis. Importantly, the study also explored the potential of Raman spectroscopy for estimating film thickness, based on signal attenuation and interaction depth, offering a pathway toward simultaneous monitoring of multiple CQAs from a single sensor platform. Significant separate work was also done using near-IR sensing for the detection of water and film thickness[23]. These advances provide insights such as: Detection of the solid-state characteristics, such as potential crystallinity changes, during drying; support for real-time feedback and control strategies to enhance consistency and reduce waste; and alignment with Quality by Design (QbD) and regulatory expectations for advanced pharmaceutical manufacturing.

- We extended the film format as a potential vehicle for sustained and extended release including future use in patches and implants by exploring sustained release from high poorly water-soluble drug content hydrophilic polymeric films sandwiched between hydrophobic layers[24]. The film thickness ranged from 140-250 μm; thinner films followed Fickian diffusion, and thicker films followed non-Fickian anomalous diffusion. As a novelty, we found that the application of middle layer thickness could be used as a tool to manipulate drug load without the need for altering its formulation or precursor preparation simply by changing its thickness, hence achieving high drug loading yet having sustained drug release due to two outer layers made of an insoluble polymer.
- A major limitation of embedding the LAS precipitated drug particles into film is their limited concentration in the suspensions and hence final drug loading in dry films. This was tackled in our pragmatic work that showed that a step can be added to enhance the suspension concentration and prepare film that have uniform drug particle distribution and excellent drug particle dispersibility. This paper[25] demonstrated fast release of LAS precipitated fenofibrate at high drug loading from biocompatible thin films.

These studies laid the groundwork for a new class of nanoparticle-based oral film dosage forms, combining the benefits of nanotechnology with a user-friendly delivery vehicle. Notwithstanding the potential advantages of incorporating API nanoparticles in polymeric films, there was a critical need for simplifying the processing and formulation of the films while retaining all the advantages offered by incorporating the nanoparticles. Drawing upon the substantial background in dry coating[11,26], and considering potential advantage of not needing any surfactants and cost-intensive nanomilling operations, direct incorporation of 3-5 μm milled and dry coated APIs was tested using the same film formers. In our first breakthrough paper[2], we introduced a simpler, robust method to incorporate dry micronized and surface modified poorly water-soluble drug, fenofibrate (FNB). As a novelty, simultaneous surface modification using hydrophilic silica along with micronization was done using fluid energy mill (FEM) to reduce FNB hydrophobicity and powder agglomeration. Our hypothesis was that silica coating would promote easy, uniform dispersion of micronized and coated FNB (MC-FNB) during direct mixing with aqueous hydroxypropyl methylcellulose (HPMC-E15LV) solutions. That was proven from results demonstrating improved film critical quality attributes (CQAs) such as appearance, drug content uniformity and drug dissolution even when simple impeller mixing was utilized to prepare the slurry prior to film casting with low as well as high viscosity precursors. Fig. 7 presents digital micrography images of small film samples for various cases; demonstrating uniform films when MC-FNB particles were embedded in the film. The visual results were validated by the drug particle redispersibility outcomes for all these cases as well as drug dissolution testing; the results for dissolution are shown in Fig. 8. In a related work, the effect of high drug loading (DL) was investigated for ~20-60 wt% DL. As shown in Fig. 9, higher DL led to faster dissolution rates, which contrasts with what was observed for nanoparticles[20].

Subsequent work demonstrated that dry coated micronized API particles can be used even in thicker films which leads to zero order release without any initial burst or dose dumping[27]. It was demonstrated that as thickness increased from 100 μm to 2000 μm, the release mechanism changed from Fickian diffusion to zero-order release for films ≥ 1000 μm. The diffusional power law exponent, *n*, achieved value of 1, confirming the zero-order release, whereas the percentage drug release varied linearly with the sample surface area, and sample thickness due to fixed sample diameter. Two approaches to manufacture thick films (≥ 1000 μm), monolithic thick film and stacked film, were evaluated, and compared. This novel approach is expected to have practical value by providing a new method for sustained release dosage form development where the hydrophilic matrix can have near zero porosity and zero-order release beyond certain critical thickness. Another important paper discussed different methods of measuring film disintegration time (DT) while exploring the effect of using SDIs to enhance DT[28]. Three different methods were used to measure film DT. Two SDIs, sodium starch glycolate (SSG) and croscarmellose sodium (CCS), were selected, acting also as VEAs for hydroxypropyl methylcellulose (HPMC-E15LV) and glycerin solutions. Significant decreases in film DT were observed due to the addition of SDIs. Percentage reduction in DT was a strong function of SDI amount while thinner films disintegrated faster as expected. Films with either higher SDI concentrations (>9%) or films under 80 μm, exhibited fast DT (<180s, Ph. Eur.). All thin films (50−60 μm) exhibited immediate release (>80% in 10 min). All such films achieved good content uniformity. These and other advances in incorporating micro-sized drug particles are discussed in detail in this comprehensive patent[29].

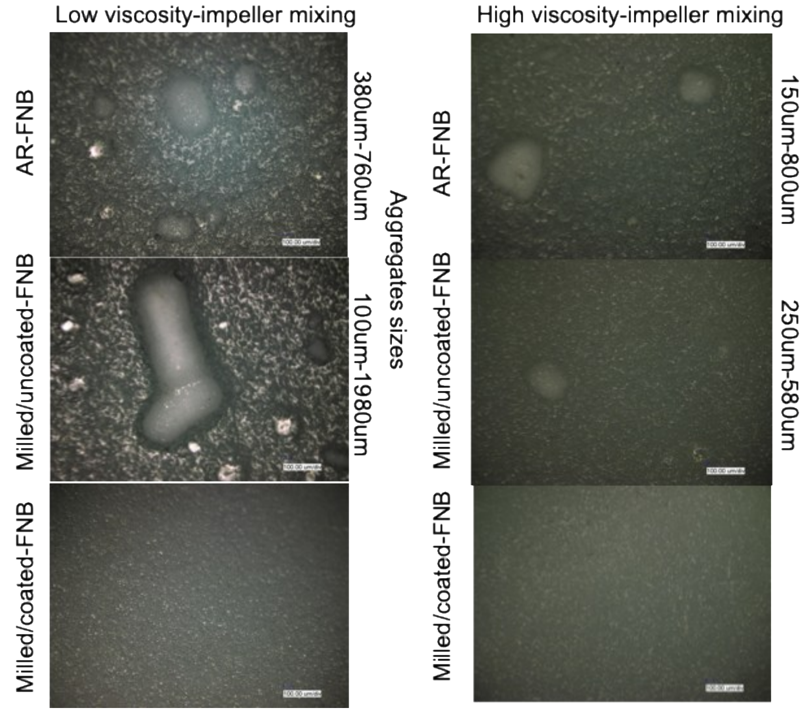

Figure 7. Digital microscopy images of films (employing impeller mixing).

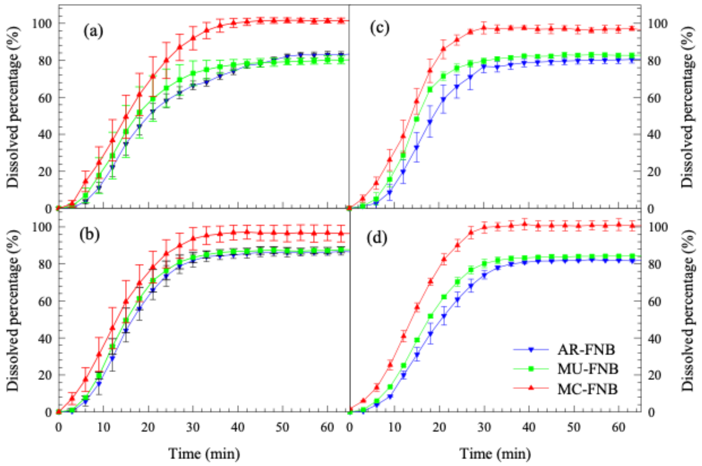

Figure 8. Film dissolution profiles of films loaded with AR-FNB, MU-FNB and MC-FNB in 100 ml dissolution medium. Low viscosity: (a) impeller mixer; (b) planetary mixer; High viscosity: (c) impeller mixer; (d) planetary mixer.

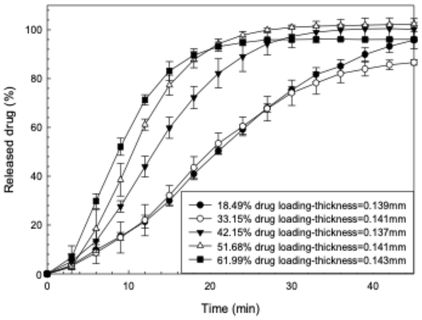

Figure 9. Dissolution from high drug loaded films containing micronized and dry coated FNB particles.

More recently, we have also explored solution casting of poorly water-soluble drugs, knowing well the challenges it poses. This important work[30] demonstrated that the choice of the polymer and the solvent system affected the film quality attributes, such as the drug recrystallization, drug content uniformity, film mechanical properties, drug dissolution rate including supersaturation, and storage stability. In addition to the solvent affecting the film structure, it had a significant impact on drug recrystallization, which in turn impacted many other film attributes, including the content uniformity and film mechanical properties which were found to be adversely affected by uncontrolled recrystallization leading to the occurrence of large and uneven crystals in some of the films. The most important finding was that the drug recrystallization was affected strongly by drug solubility in the solvent, and to a lesser extent by the polymer type, which also affected the film structure and related properties. Consequently, the recrystallization of drug also affected its dissolution rates as well as supersaturation under non-sink conditions, again largely attributed to uncontrolled crystal sizes. These results demonstrated that for solution-cast films, uncontrolled recrystallization and poor time-stability would be unavoidable, and over time, amorphous drug content would diminish significantly. Thus while an attractive method for commercial usage, there are many pitfalls and failure modes in solvent casting that a practioners must be aware of. All our work to date indicates that using milled and dry coated micro-sized APIs in slurry based film casting approach appears to be the most robust and practical method for commercial applications.

In summary, the results presented here open avenues for further development of regulatory science, hopefully forming the technology knowhow for clinical translation of strip film technologies in drug delivery[31].

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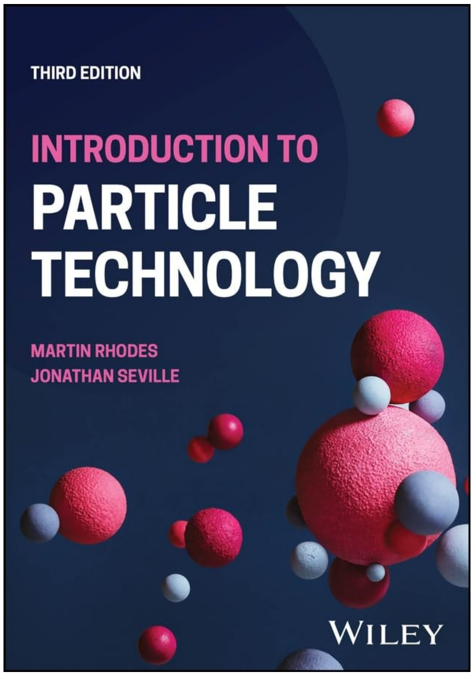
Book Review

Introduction To Particle Technology, 3rd Edition

Authors: Martin Rhodes and Jonathan Seville



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The latest edition of this authoritative textbook on Particle Technology represents a collaborative endeavor by Professor Martin Rhodes and Professor Jonathan Seville. Since its initial publication in 1990, this book has been widely recognized for its clear exposition, comprehensive treatment of key concepts, and concise explanations of fundamental principles. The authors have further expanded the second edition by incorporating new chapters on mechanical properties, Discrete Element Modeling (DEM), colloids and aerosols, as well as product applications. Although primarily written for undergraduate and postgraduate instruction, it also serves as a valuable desk reference for industry professionals. Its effective combination of clarity and brevity provides readers with the foundation to engage in critical inquiry and address complex industrial challenges.

- This survey text covers essential topics in Particle Technology, offering a solid foundation and encouraging further study in the field.
- The authors have interspersed many practical recommendations (knowledge nuggets) within the text. The practicing engineers will find them most useful.
- The new chapter on DEM is an excellent addition for the benefit of future particle technologists.
- Each chapter has worked examples, test questions and exercises to engage and challenge the readers. The test questions are thought provoking and well-written.
- The choice of worked examples with their step-by-step approach provides the guidance needed to master the subject. The equations and relevant nomenclature are easy to understand and consistent across the text.
- The companion website, once available, would be an excellent avenue to facilitate the learning process and keep the content updated.

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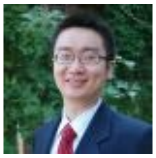

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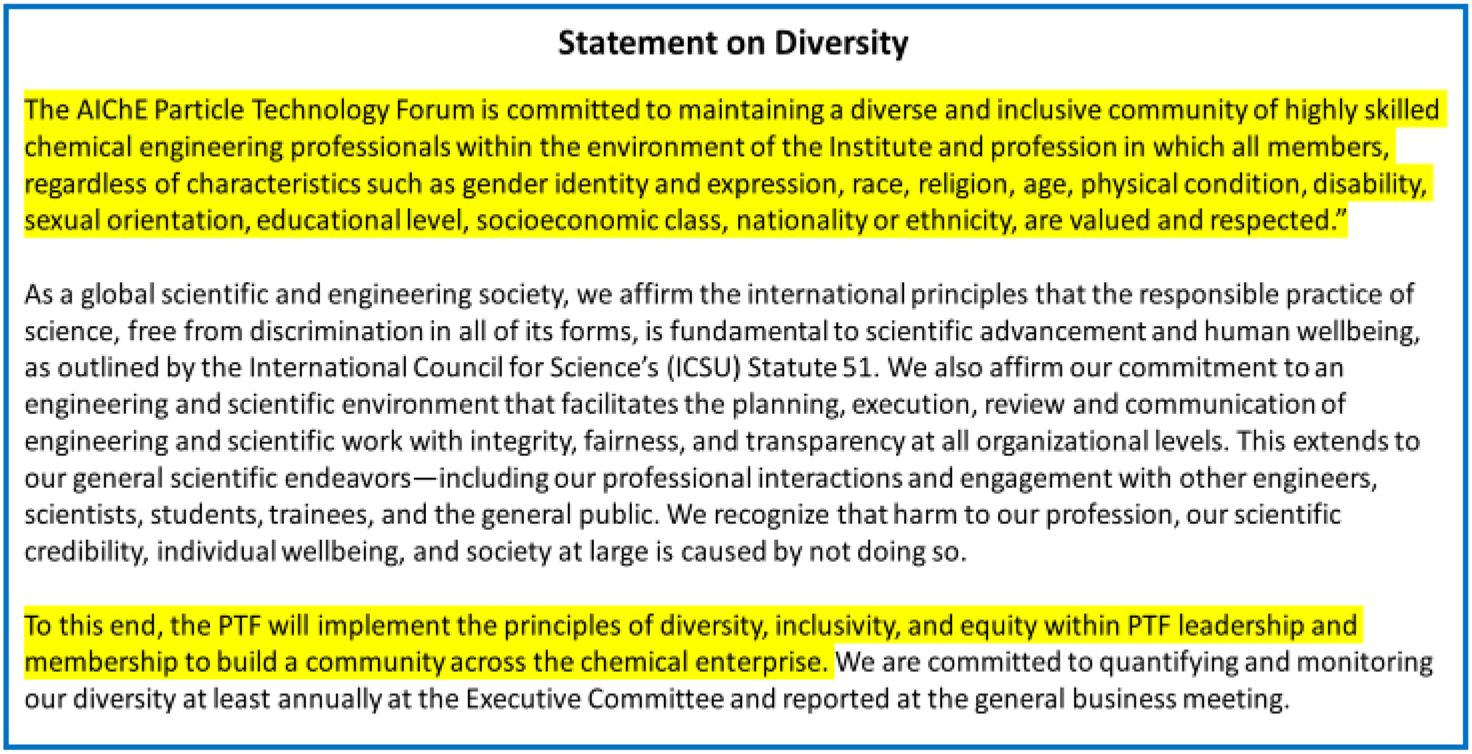

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