Development of PVA/Alumina(Al_2O_3) Composite via Gelation Encapsulation

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Abstract

In this work, polyvinyl alcohol (PVA)/alumina composite films are fabricated using a gelation encapsulation approach followed by melt processing of final films. Due to the high density of alumina, in order to prevent aggregation and sedimentation, a gradual flow approach is used to steadily encapsulate alumina particles in a PVA gel matrix. The robust nature of the gel is expected to prevent alumina sedimentation. PVA gels are explored at different concentrations ranging from 3 wt% to 8 wt%. The alumina concentration is kept constant. Subsequent characterization of the encapsulated gels and final films are performed to understand how gel concentration can improve the alumina dispersion in the final film. Optical microscopy, scanning electron microscopy, and tensile testing are all performed. Based on the results, as the gel concentration is increased larger particles can be dispersed and prevented from sedimentation. However, as the gel concentration increases, aggregation is also more common. Aggregation is found to compromise mechanical behavior of the films. It is observed that at 5 wt% the gel and subsequent films shows the most promising results, where the morphology is most uniform and the mechanical behavior is good. This work is an introductory study to understanding the use of gelation as a means of particulate encapsulation to improve dispersion, and will be followed by more in depth studies to further understand the results presented in this summer study.

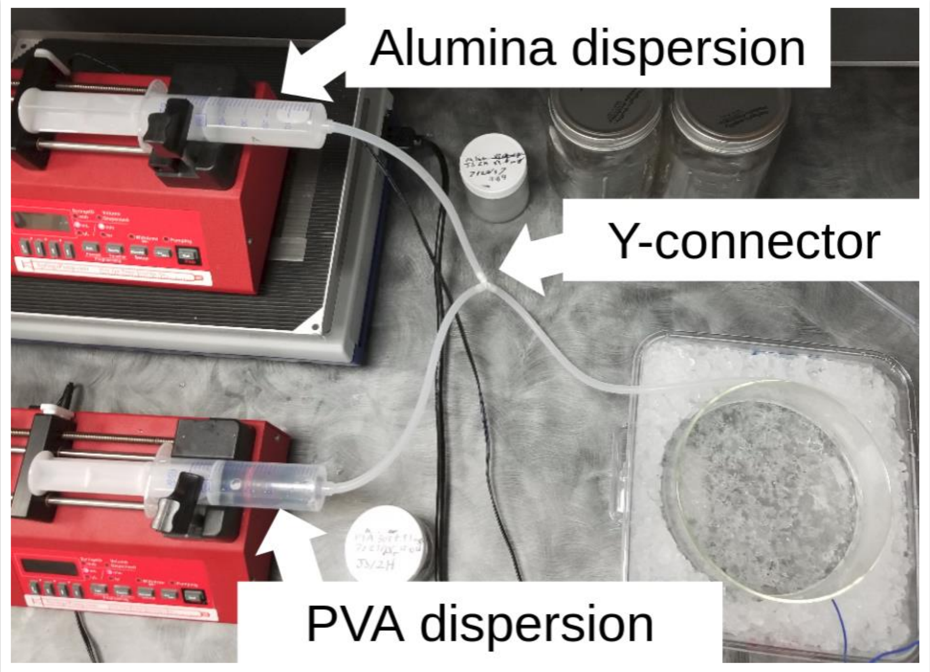
Materials and Methods

Materials: Polyvinyl alcohol(PVA), Alumina(Al_2O_3), Methanol, Water

Methods:

1. PVA solution preparation
-Measure PVA to target weight percentage: 3%, 4%, 5%, 6%, 7%, 8%.
-Dissolve PVA in water at 90°C for 2 hours.

2. Alumina dispersion preparation
-Mix 85.4mg of Alumina into 100mL water, and sonicate to disperse the particles.

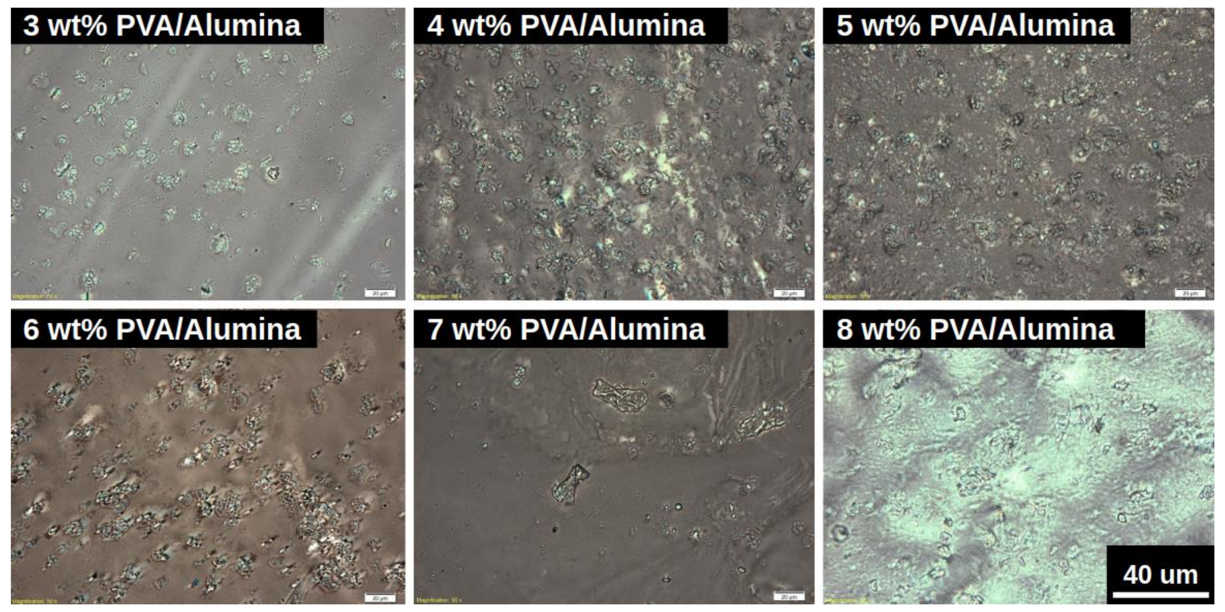


3. Gelation encapsulation
-Load PVA solution and Alumina dispersion into syringes. To prevent particle sedimentation, shaker table is used.
-Connect tubing's to both syringes through the Y connector. Once methanol reaches at ~3°C, inject PVA and Alumina simultaneously into the bath for gelation encapsulation. Collect precipitated PVA/Alumina gel using strainer.

4. Sample film melt processing
-Place sufficient amount of PVA/Alumina gel in between Kapton-covered glass slides, and leave on 60°C hotplate for 1.5 hrs.
-Peel of the film and samples are ready for characterization.

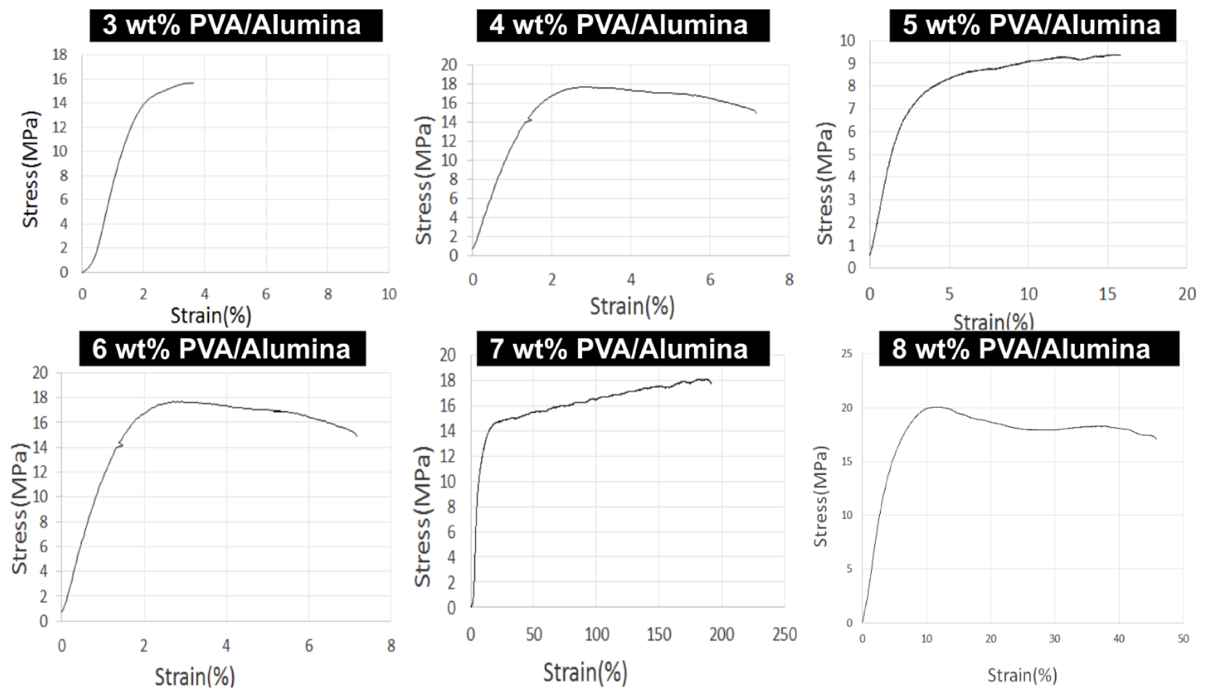
Composite Film Property Testing

Optical Microscopy



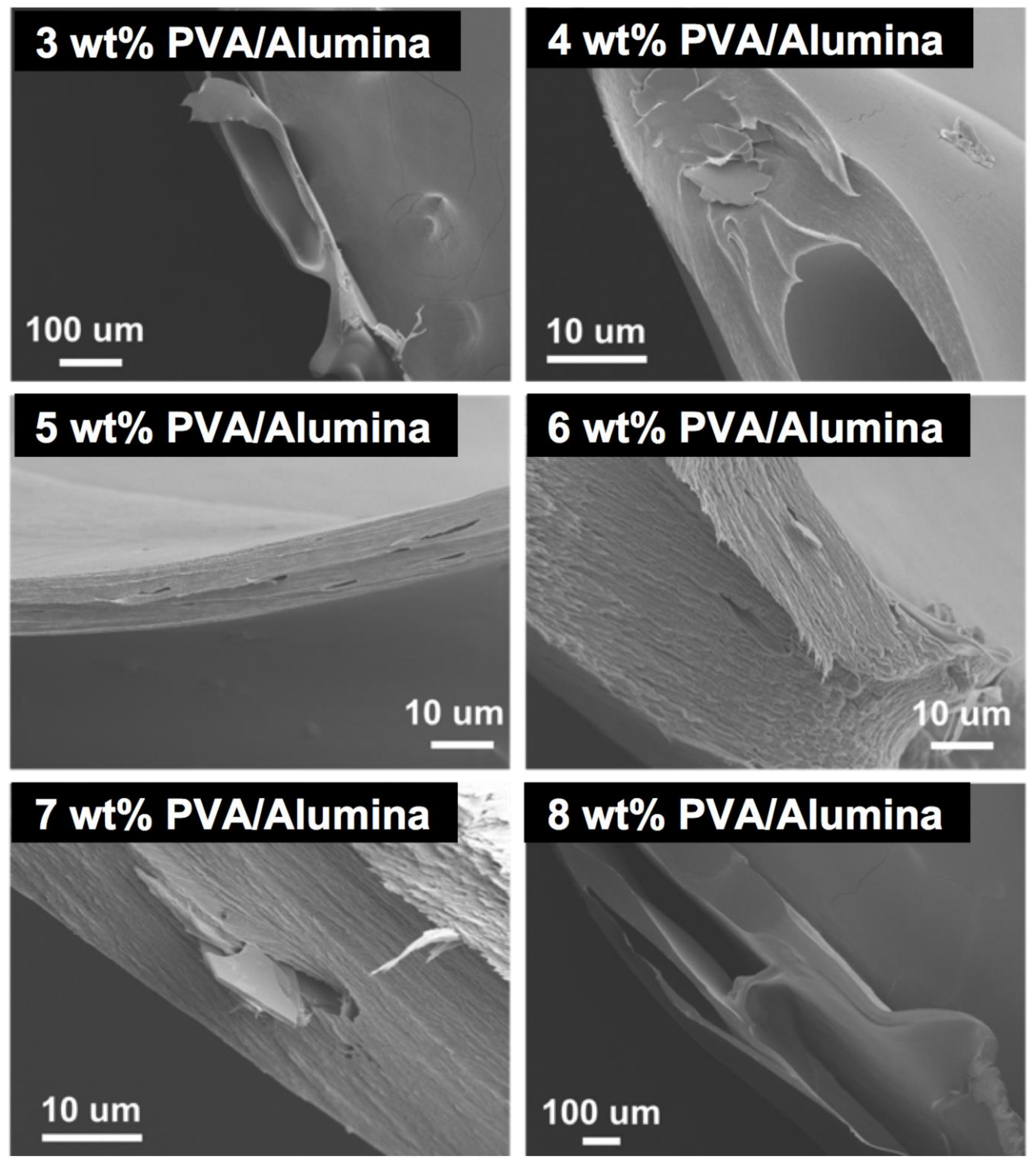
Sample	Average area (um²)	Average diameter (um)
3 wt%	65.05	9.10
4 wt%	54.16	8.30
5 wt%	130.27	12.88
6 wt%	176.14	14.98
7 wt%	430.05	23.40
8 wt%	336.08	20.69

Mechanical Test



Sample	Strength (MPa)	Modulus (MPa)	Strain (%)
3 wt%	11.81 ± 4.39	628.27 ± 124.35	23.94 ± 23.42
4 wt%	14.11 ± 4.55	796.41 ± 312.02	27.75 ± 23.88
5 wt%	34.59 ± 18.38	1130.95 ± 561.07	157.61 ± 145.09
6 wt%	10.37 ± 11.00	450.98 ± 350.37	34.00 ± 7.93
7 wt%	16.63 ± 5.03	642.00 ± 257.68	77.91 ± 77.42
8 wt%	21.43 ± 4.25	552.40 ± 158.26	19.79 ± 6.02

SEM on Fractured Surface



Results

- From the optical microscope images, the macroscopic dispersion of Alumina particles inside the PVA matrix is good. The gelation encapsulation process assisted to main the Alumina dispersion. As the gel concentration is increased larger particles can be dispersed and prevented from sedimentation. However, as the gel concentration increases, aggregation is also more common.
- Aggregation is found to compromise mechanical behavior of the films. It is observed that at 5 wt% the gel and subsequent films shows the most promising results, where the morphology is most uniform and the mechanical behavior is good.
- The SEM micrographs show the fracture during tensile test occurred due to the air pockets and Alumina aggregates.

Challenges

- Filtration time to remove methanol is extremely long by using regular filter paper. A metal strainer is used instead to retain precipitated PVA/Alumina gel.

- Evaporation of water in dissolving PVA caused the solution to change in weight percentage altering the overall dispersion of PVA and structure of the sample.

- Another difficulty we encounter was proper sample production, as our original methods of producing samples would cause them to be too thick and accessible for proper testing.

Conclusions

A gelation encapsulation process is successfully developed to produce PVA/Alumina composite. The resultant film at 5 wt% shows the most uniform distribution of particles and promising mechanical properties.

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