

# Rheological Properties and Processing Characteristics of Soluplus<sup>®</sup>, Kollidon<sup>®</sup> VA 64 and HPMCAS

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## Abstract

Twin screw extruders have historically been used to melt mix filled polymers, polymer blends, formulations and enable consistent feeding into downstream polymer processing operations. Recently, twin screw extrusion is being used widely in the hot melt extrusion process to melt blend active pharmaceutical ingredients (API) and polymers for various pharmaceutical and drug delivery applications. The aim of this study was to examine the rheological and processing behavior of three pharmaceutical grade polymers under extrusion at different temperatures and shear rate.

**KEYWORDS:** Soluplus<sup>®</sup>, Kollidon<sup>®</sup> VA 64, HPMCAS, Melt Extrusion, Rheology

## Experimental Methods

Three pharmaceutical grade polymers were used in this experiment: Soluplus, Kollidon VA 64 and HPMCAS. Soluplus, Kollidon VA 64 were obtained from BASF Corporation (Florham Park, NJ). HPMCAS (MF grade) was obtained from Shin-Etsu Aquot (Tokyo, Japan). Soluplus is a graft copolymer comprised of polyethylene glycol, polyvinyl caprolactam and polyvinyl acetate designed for use in the hot melt extrusion process for poorly soluble drugs. Kollidon VA 64 is a vinyl pyrrolidone-vinyl acetate copolymer traditionally used in the pharmaceutical industries as a dry binder, granulating agents, and as a film forming agents. Hydroxypropyl methylcellulose- acetate succinate (HPMCAS) is being used in pharmaceutical, nutritional and personal care industries.

### (a) Twin Screw Extrusion:

An 18mm twin screw extruder (Leistritz) was used to extrude the polymers. The polymers were extruded at 170°C with a constant feed rate of 2 lbs/hr and screw speed of 100 RPM. The effect of Soluplus on the rheological and processing characteristics was tested by making a three batch mixes each of 500g. The batch mixes are a) 20% Soluplus with 80% Kollidon VA 64; b) 20% Soluplus with 80% HPMCAS; and c) 20% Kollidon VA 64 with 80% HPMCAS. Batch mixing of these polymers were done manually and extruded in a twin screw extruder at 180°C with a constant feed rate of 2 lbs/hr at screw speed of 100 RPM. The extruded strands were drawn through a conveyor belt, air cooled and pelletized using a CON-AIR strand pelletizer.

A constant feeding rate and screw speed throughout the process is important as the combination of these two factors establishes the level of fill in extruder. A constant feed rate and screw speed, helps to deliver a constant amount of material into the extruder thereby the shear stress and residence time subjected on to the material remains constant. During each run of the experiment the percentage torque & head pressure required to extrude the polymers was recorded.

### (b) Capillary Rheology

A Capillary Rheometer (Dynisco, Model: LCR 7000) was used to measure the dynamic viscosity of the polymers. A 10 point shear rate test was performed for all three polymers at 160°C, 180°C and 200°C. Attempts to measure the dynamic viscosity of the polymers using powder samples failed due to difficulty in filling the barrel. The extruded polymer pellets were easily filled and closely packed into the barrel. The piston was operated from low to high shear rates (0 to 6000 (1/s)). Attempts to measure shear viscosity at 120°C and 140°C did not yield results due to insufficient melting and poor flowability of the pellets inside the capillary. Dynamic viscosity of the 3 batch mixes were tested at 180°C, 200°C and 210°C. Attempts to measure the viscosity below 180°C failed due to difficulty in filling the barrel.



## (c) Parallel Plate Rheometry

The dynamic viscosity of all polymers also was measured on an AR-2000Ex parallel plate geometry Rheometer with ETC (Environmental Test Chamber) Oven temperature control system. The frequency sweep test gives the Newtonian plateau of the polymer i.e.; the frequency range at which the fluid is independent of the shear condition. The operating parameters were:

Frequency Sweep: 1 to 100 rad/s, 0.5 % strain, 5 points per decade at 200°C

Strain Sweep: 0.1% to 10% Strain, 10rad/s Frequency, 5 points per decade at 200°C

## Results and Discussion

Soluplus, Kollidon VA 64 and HPMCAS each weighing 400g were extruded separately in a twin screw extruder at 170°C and at a constant feed rate of 2 lbs/hr. Figure 1a and 1b shows the effect of the constant feed rate and screw speed on the percentage torque on each polymer. The data suggests that Soluplus has the least amount of the % torque and HPMCAS has the highest amount of % torque required to extrude the polymer. The three batch mixes were extruded at 180°C at a feed rate of 2 lbs/hr. The attempt to extrude the batch mixes at 170°C failed due to insufficient melt temperature and die swell. Processing of the batch mixes were improved by increasing the melt temperature to 180°C.

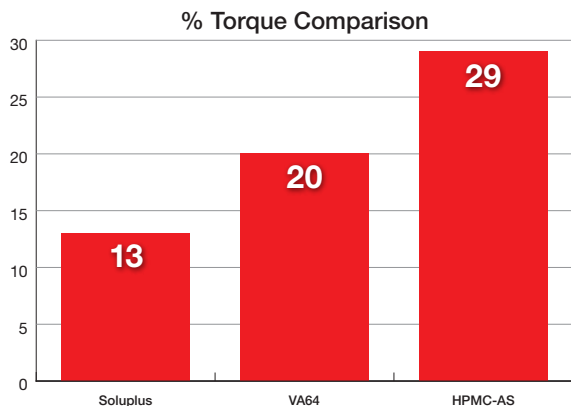


Figure 1a. % torque comparison of the polymers extruded at 170°C.

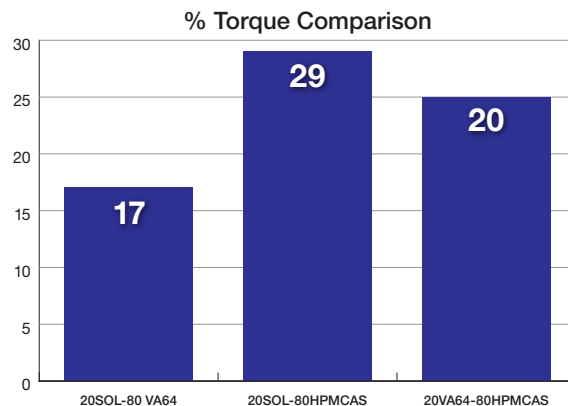


Figure 1b. % torque comparison of the polymers extruded at 180°C.

The melt pressure of a polymer is dependent on the feed rate and screw speed. With a constant screw speed and feed rate it will depend on the molecular weight and miscibility of the polymer blend ratios. Figure 2 shows the effect of the constant feed rate and screw speed on the melt pressure. Soluplus and Kollidon VA 64 has minimum melt pressure. The data suggests that Soluplus and Kollidon VA 64 are easy to melt process. It can be noted that HPMCAS has the highest melt pressure because of its high T<sub>g</sub> (130°C). The batch mix of 20% Soluplus/80% Kollidon VA 64 showed no melt pressure which indicates good miscibility of the polymers and better processibility. The addition of Soluplus to HPMCAS, whereas 20% addition of Kollidon VA 64 increased the ease of processing as indicated by the reduction in melt pressure.

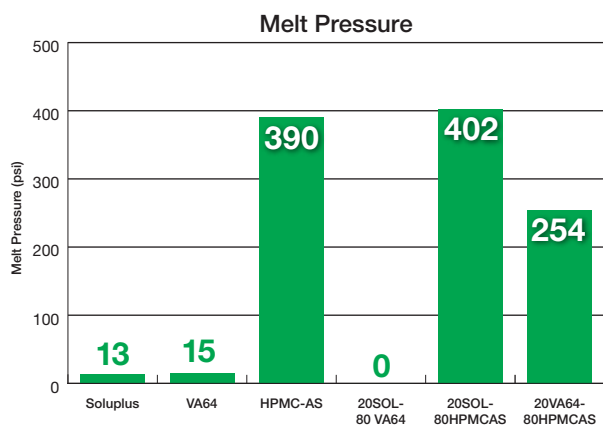


Figure 2. Plot of melt pressure comparison of the polymers used.

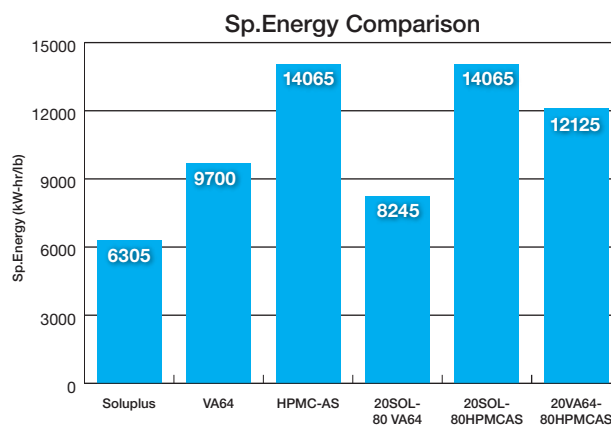


Figure 3. Comparison of Specific energy of polymers.

Specific energy<sup>2</sup> is the amount of mechanical energy that is being input by the extruder per unit mass of material being processed. The specific energy required to extrude the polymer was calculated in two steps as follows

- 1) Applied power kW = kW(motor rating) x % torque x rpm x .97(gearbox efficiency)
- 2) Specific energy =Applied power/ pounds per hr = kW-hr/lb

A plot of specific energy was calculated with the above mathematical expression for each of the polymers processed using a TSE. Figure 3 shows the amount of specific energy that each polymer requires for its processing at a constant feed rate of 2 lbs/hr and at a constant screw speed of 100 rpm. The data suggests that the specific energy was reduced for the batch mix of Soluplus/Kollidon VA 64 (20:80) compared to the Kollidon VA 64 specific energy. It can be inferred from the data that the work required to process HPMCAS is more than two times that of Soluplus. Addition of Soluplus in small quantities did not make any change to the specific energy, whereas, 20% addition of Kollidon VA 64 increased the ease of processing of HPMCAS and decreased the specific energy by 13.8%.

Viscosity measurements of the three polymers investigated was performed at 3 different temperatures 160°C, 180°C & 200°C. The viscosity of Soluplus was found to be higher at 160°C in low shear rate region, and decreased gradually at higher shear rates >500 (1/s). As the temperature increases, the viscosity of Soluplus decreases which is attributed to its material property. The pure Soluplus can be extruded up to 180°C depending on the screw configuration but after that the polymer starts to show chemical degradation. Figure 4 shows the effect of shear rate on the viscosity of Soluplus at different temperatures. The decreased viscosity of the polymer is attributed to the polymer chain movement when subjected to high shear and temperature. The same pattern was observed with Kollidon VA 64 polymer. Figure 5 shows the effect of shear rate on the viscosity of Kollidon VA 64 at different temperatures. It is to be noted that the viscosity of Kollidon VA 64 is comparatively lower than Soluplus and also decreases with increase in temperature, shear rate.

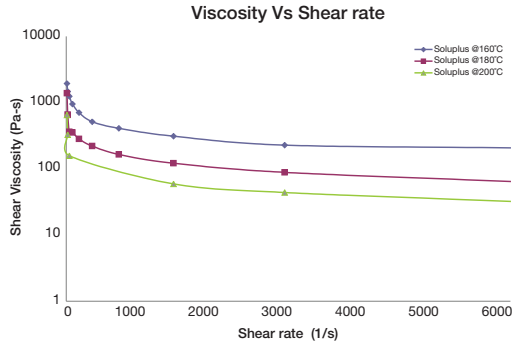


Figure 4. Shear viscosity of Soluplus at 160°, 180° and 200°C.

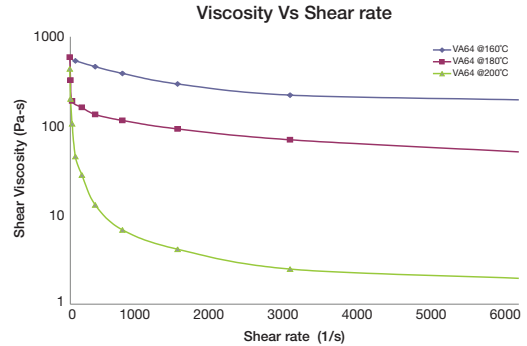


Figure 5. Shear viscosity of Kollidon VA 64 at 160°, 180° and 200°C.

Figure 6 shows the effect of shear rate on the viscosity of HPMCAS polymer at three different temperatures. A different pattern was observed in the viscosity of HPMCAS at 200°C unlike the other two polymers. At lower temperatures the viscosity of HPMCAS was found to be following the same pattern of the other two polymers, but an increased viscosity at 200°C even at lower shear rate of the polymer can be attributed to polymer degradation and/or cross-linkage.

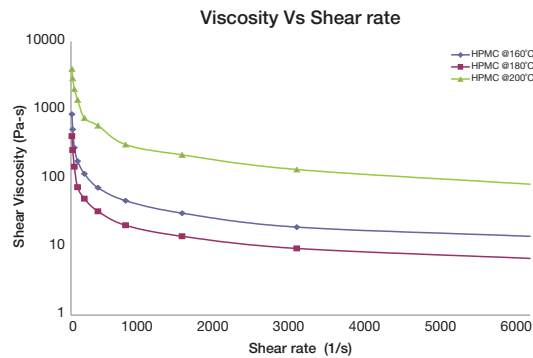


Figure 6. Shear viscosity of HPMCAS at 160°, 180° and 200°C.

Figure 7 illustrates the viscosity as a function of stress for HPMCAS containing 20% Soluplus (A) and 20% Kollidon VA 64 (B) at 180°C, 200°C and 210°C. The data suggests that the addition of Soluplus or Kollidon VA 64 helped reduce the melt viscosity at a relatively lower shear stress. In contrast, increasing temperatures to 200°C and 210°C increased the melt viscosity of HPMCAS with either blend of Soluplus or Kollidon VA 64, presumably due to polymer degradation/cross-linkages at higher temperatures.

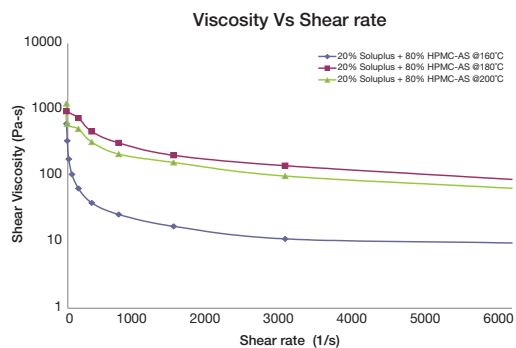


Figure 7a. Shear viscosity comparison of batch mix 20% Soluplus/80% HPMCAS.

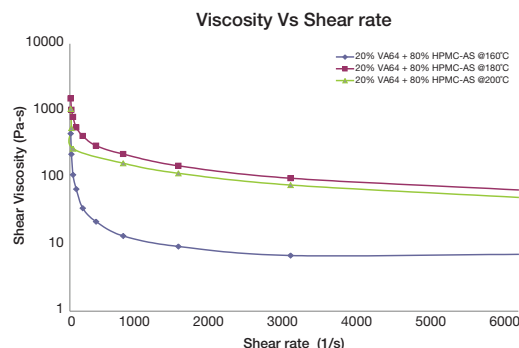


Figure 7b. Shear viscosity comparison of batch mix 20% Kollidon VA 64/80% HPMCAS.

The complex viscosity of three polymers was compared using a parallel plate viscometer. Figure 8 shows the effect of angular frequency on the viscosity of the polymers. A plot of viscosity of the three polymers in both the capillary and parallel plate was observed to be more or less same which confirms the rheological behavior of the polymers.

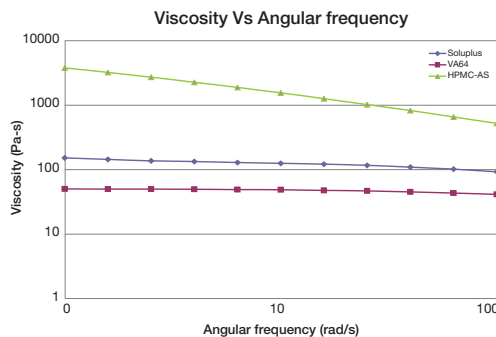


Figure 8. Shear viscosity comparison of polymers at 200°C using parallel plate viscometer

## Conclusion

Twin screw extrusion data suggests that, Soluplus and Kollidon VA 64 can be easily processed at lower temperatures compared to HPMCAS. The torque, melt pressure and specific energy observed with Soluplus were much lower than Kollidon VA 64 and HPMCAS and hence exhibits better processibility. A higher shear viscosity of HPMCAS at 200°C and 210°C indicates material degradation under high shear rates and temperature. Capillary and parallel plate data confirms and compliments the rheological behavior of the three polymers.

## Acknowledgements

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