#### RFI Category 2 – LIGNIN

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#### Introduction:

American Science and Technology Corporation (AST) is an R&D service provider with pilot plant capabilities that can process various lignocellulosic biomass and produce lignin from grams to several tons per week. Over the years, AST has developed two patented Organosolv-based fractionation technologies that can extract lignin from lignocellulosic materials and produce pulp and bio based chemicals such as furfural, butyl acetate, etc.

Question 1: To which types of research entities are you willing and able to sell or otherwise provide your lignin? (e.g., university researchers, national laboratories, industry/private sector)? Are there any types of research entities to whom you are not willing and able to sell your lignin?

AST sells its lignin worldwide to any entity that may need it for any purpose. So far, AST's lignin samples have been shipped to various universities (domestic and international), national labs and government entities, as well as public and private companies (domestic and international). We impose no restrictions and no limitations unless it is prohibited by US laws.

### Question 2: What are the maximum and minimum quantities of lignin that you are willing and able to sell (kg)?

We can supply from a few grams to two ton per week. AST's current capabilities allow us to produce lignin from a few grams per batch (in our laboratories) to about 125 Kg per batch using our 2000 gallon pilot plant. If necessary, AST can operate its pre-commercialization pilot plant to run four batches per day to produce about half ton of lignin per day.

### Question 3: In what units do you sell your lignin and is it packaged (e.g., super sacks), or sold in bulk?

Any unit and any packaging as defined by customers. So far, AST has shipped lignin to its customers from a few grams inside Ziploc plastic bags to a few hundred kilo grams in heavy duty super sacks inside cardboard boxes and on wooden pallets.

#### Question 4: How do you ship lignin?

We can ship our lignin as requested by our customers. So far small samples were shipped via overnight delivery services or ground transportations, and large samples were shipped via trucks and ground transportations.

#### Question 5: What is the lignin concentration in your product?

AST's lignin purity is 85-95% (Acid soluble Lignin + Acid insoluble Lignin, measured by TAPPI method T222-om02).

#### Question 6: What type(s) of biomass do you use in your process?

AST can use any lignocellulosic biomass to produce lignin. We have processes set for various hardwood, softwood, and agricultural wastes such as corn straw, wheat straw, tobacco stalk, etc.

### Question 7: What process do you use that produces lignin (dilute acid, ammonium fiber expansion (AFEX), hot water, Organosolv, etc.)?

AST uses its proprietary and patented Organosolv process to fractionate lignocellulosic biomass and separate lignin from pulp. AST solvent includes organic solvent and water at a pH of less than 4. During AST's fractionation process, lignin is dissolved in organic solvent and all water soluble materials are dissolved in aqueous solvent. At the end, because of the difference in their specific gravities, the mixture of organic solvent and lignin separate themselves from aqueous solvent and everything else is dissolved in water. After separating the organic layer from the solvent, it goes through the evaporation process to separate the lignin in a solid form and recover the organic solvent and recycle it for the next batch.

### Question 8: What details of the scale of your process are you willing to share (e.g. batch and/or continuous or volumetric productivity)?

AST's current capabilities are all batch processors and include the following:

- a) Two small 250 ml laboratory PARR reactors that can process about 20 grams of biomass per batch, and each batch from start to end will take about 4 hours. At the end of each batch, we cool down the reactor, open it up, remove the solvent and the remaining biomass, filter the pulp out of the solvent, let the solvent stay for a few minutes to allow most of the organic materials to move upward while the aqueous moves downward, separate the organic and aqueous layers from each other and recover lignin from the organic layer.
- b) One 40 gallon pilot reactor with about 15 to 20 kg biomass processing capability per batch. Inside this reactor, we have a perforated basket that holds the biomass. During the process, the biomass is continuously showered with hot solvent from the top of the basket. While traveling from the top of the basket toward the bottom, the solvent washes the biomass and therefore removes the lignin. After exiting from bottom of the basket, the solvent is the collected in the bottom of the reactor where it is directed toward a heater to heat up the solvent and maintain process temperature for the duration of our process. At the end, the solvent is removed, passed through the heat exchanger, and directed toward a separation tank where the organic and aqueous layers

- are separated from each other in a short period of time. The organic layer then goes through the evaporation process to separate the solid lignin from the recycled organic solvent.
- c) One 2000 gallon pilot reactor with about half ton biomass processing capability per batch. In this reactor, first the biomass in loaded via a bucket elevator. After that, hot solvent at process temperature is injected into the reactor at a high pressure and a heating jacket tries to maintain the process temperature during fractionation period. A high shear mixer continuously mixes the materials inside the reactor. At the end, the materials inside the reactor are directed to a blow tank to further open up the pulps and at the same time cool them down. From the blow tank, the materials are pumped into a screw press to separate the pulp from the solvent. The solvent is either directed toward a stack centrifuge to separate the organic and aqueous layers from each other or directed to a separation tank where time and specific gravity separate them. After the organic layer is separated, it goes through the evaporation process to separate lignin from the recycled organic solvent.

### Question 9: Do you measure the typical composition of your lignin? If so, what method do you use? How consistent is the composition of your lignin?

AST lacks the capabilities to measure its lignin composition. However, some of the AST's lignin compositions have been measured by universities and customers. Typically, lignin produced from agricultural wastes presents the following qualities:

- over 80% purity (Klason lignin method),
- 2% to 3% phenolic content
- 1% to 1.5% ash.
- The S:G:H ratios are 10% to 40% of S, 50% to 80% of G, and 0% to 20% of H lignin.
- Elemental analysis of one sample shows availability of:
  - o 59.3% of Carbon
  - o 6.1% of Hydrogen
  - 32.2% of Oxygen
  - 1.5% of Nitrogen, and
  - o 0.9% of Sulfur

## Question 10: Do you routinely test your lignin for consistency within and between lots? AST performs routine tests to measure lignin purity, melting point, ash content, and solubility in various solvents between the fractionation runs.

### Question 11: What impurities are present in your lignin and what testing do you perform to determine the presence of impurities?

AST measures lignin purity (Klason Lignin, TAPPI method T222-om02) and ash content (using TAPPI method T211-om02). Depending on the biomass source, AST lignin can have anywhere between 1% to 6% impurities. The impurities are mainly ash 1% to 2%, and carbohydrates (various left over sugars) 1% to 5%.

#### Question 12: Does your process include a purification or filtration step?

AST process does include centrifuge and filtration. Any further purification is done per customer's request. After the organic layer is separated from the aqueous layer, it is sent to the centrifuge to separate any leftover solids from liquid. The remaining liquid is then passed through half micron filter to remove any other fine particles.

#### Question 13: What is the typical concentration in g/L you can provide?

Typically AST lignin is in solid form with over 90% solid content. It is hydrophobic so it does not attract any moisture. Since this lignin is originally separated from organic solvent, there are some remaining organic solvent in the solid that can be removed. We can produce lignin with up to a 99% solid content. Our past experiences show that the availability of organic solvent in lignin helps it to better blend with polymers to produce a transparent film.

### Question 14: Have you examined the impacts of transport and storage on lignin? If so, can you please provide any relevant (non-proprietary) details of these impacts?

AST lignin is in the form of dry granulated solid form with about 0.5% to 8% organic solvent in it. Depending on the biomass sources, and depending on the availability of the solvent in it, AST lignin may stay granular solid (at low percentage of solvent) or may clump together at higher temp (at a high percentage of solvent). AST lignin has been shipped overseas by ships and planes, domestic destinations by trucks and planes, and we have not received any reports regarding any problems.

# Question 15: What additional information are you willing and able to provide to the research community about the lignin? Please provide any non-proprietary cost information you are willing to share.

AST lignin is extracted from any lignocellulosic biomass using organic solvents. The type of lignocellulosic biomass is one the determining factor for the quality of lignin. Although to a lesser extent, the other contributing factors in AST's lignin are the Organosolv process parameters such as solvent composition, solvent to solid ratio, process time, and process temperature. And depending on the final application of the pulp (paper or sugar), and depending on the type of biomass, AST's Organosolv process parameters and solvents may change slightly. For example, the application of more oxidants in solvent may reduce the melting points for some of the lignin and can potentially change the balance of S lignin in favor of G lignin.

As of now AST has provided its lignin to research institutes and manufacturers for R&D purposes. For larger applications, AST product's price would be based on the reasonable costs of labor and the use rate of the facilities and equipment required to perform one batch digestions as listed on AST's web site.

### Question 16: Into what markets do you typically sell your lignin? What is a typical application for your lignin?

Since Organosolv lignin is a relatively new product its application has not been fully developed yet. However, because of its unique qualities such as high purity, hydrophobic, etc., a lot of

research and development efforts are being carried out by universities. As one of the main suppliers of Organosolv lignin during the last 4 years, AST has shipped its lignin to many universities, research institutes, private and public companies, both domestically and internationally. Although most of the Organosolv lignin-related product development R&D are being conducted behind closed doors, published reports indicate that AST lignin is being used for experimental production of Biofuel, bio-based chemicals, paint, polymeric parts, composite materials, carbon fiber, etc. The largest use of AST lignin is reported by the University of Wisconsin Platteville where they mixed AST lignin with various polymers to make a mixture resin, inject the new resin into various molds and produce polymeric products. When mixed with polymers, AST lignin produce a transparent product that at low percentage of lignin to polymer, it looks tan in color and as the percentage of lignin in the mixture increases, the color changes from tan to brown, dark brown, and finally black. Typically, the melting point of AST's lignin is from 100 to about 200 C (depending on the processes used to make them).

