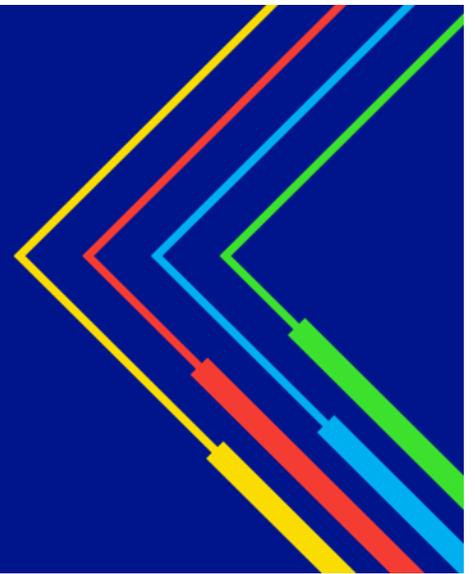


ACE: D2.2 Pole Extraction Test Methodology



Testing Methodology

The following information sets out the proposed methodologies for determining the feasibility of the use of supercritical fluid carbon dioxide for the reduction in concentration of creosote in impregnated end of life wooden poles.

Feasibility of the use of Supercritical fluid carbon dioxide for the extraction of creosote in impregnated end of life wooden utility posts.

This work is based on 2 parts,

1. Supercritical Fluid Carbon Dioxide (SF CO₂) solubilisation and removal of creosote from end of life wooden utility poles.
2. If point one is proven to be achievable, the second part of the project is to identify the most favourable conditions for extraction.

During extraction the concentration of creosote being extracted will be followed by Infrared spectroscopy using a custom-made high-pressure observation cell.

After each extraction, samples of wood will be taken for analysis from different depths. These will be ground up to aid extraction and homogeneity, and split into 2 equal portions, 1 portion being analysed in-house with the other going for external analysis.

An accurately weighed portion is then solvent extracted and analysed in house by High Pressure Liquid Chromatography (HPLC). These samples are compared to pre-extraction samples to determine extraction efficiency.

1. This work is a continuation of work completed in 2015 where the extraction of creosote was shown to be possible in chipped end of life wooden utility poles, (with chips less than 1 cm in thickness). Two main observations were made from that study,
 - a. It was shown to be possible to extract creosote from the chipped wood under supercritical conditions, and
 - b. All 16 major components of the creosote, 16 different Poly Aromatic Hydrocarbons (PAHs) were equally extracted, this is important because the creosote concentration is the sum of the 16 individual PAHs concentrations.

The current work is based on whole (unchipped) wooden utility poles of approximately 25 to 30 cm in diameter, which means that the SF CO₂ has to penetrate much further into the wood.

Initial analysis of the untreated poles showed that the creosote impregnated the wood to a depth of approximately 8 cm. Analysis of the poles indicated, as expected, the outer 4 cm had a highest concentration level of approximately 5000 mg/kg, while the next 4 cm band had a concentration level of approximately 3000 mg/kg, with the remaining core generally had a concentration level of < 1000 mg/kg.

The wood is deemed to be hazardous if it has a creosote concentration > 1000mg/kg, where it is deemed non-hazardous, and this level remains our minimum goal, therefore it is required that we permeate the SF CO₂ at least 8 cm into the poles.

Analysis of poles with a substantial amount of cracking showed the second inner band (4 to 8 cm in depth) had much higher creosote concentrations than uncracked poles, this anomaly was attributed to creosote migration into the pole via these cracks.

2. Once it has been established that SF CO₂ can penetrate the poles, to the required depth, and solubilise the creosote, we have to establish the most favourable conditions for this to be achieved. To this end we have 4 methods to be tried.
 - i. Pressure and flow dependance
 - ii. Pulsed extraction
 - iii. Two different modifier chemicals, Propanone (Acetone) and Methanol.

- i. Our extraction vessel has a maximum working pressure of 1400 psi, and has been pressure tested to 2000 psi. Therefore extraction can be attempted at pressures up to 1400 psi. Although the critical conditions for carbon dioxide are 1072 psi and 32 oC, extraction can sometimes be achieved using a dense gas (where the pressure is slightly below the SF CO₂ conditions), and acts as a dense gas rather than a supercritical fluid. We also have control of the carbon dioxide flow from the pump, and so this allows a further parameter to be varied.

- ii. As already noted, the SFCO₂ has to ingress into the wood to a depth of approximately 8 cm, this results in the internal pressure of the wood being equal to the external pressure within the extraction vessel but outside the wood, therefore because the pressures are equalized, what is the driving force for mass transfer of the creosote out of the wood. To assist the mass transfer if we dropped the external pressure in the extraction vessel we then create a pressure differential between the internal pressure in the wood, and the external pressure in the extraction vessel.

The easiest way of dropping the external pressure is to stop the pump and increase the volume within the extraction vessel. This will be achieved by the addition of a pressure accumulator (a 60 litre hydraulically driven high pressure syringe), see below.

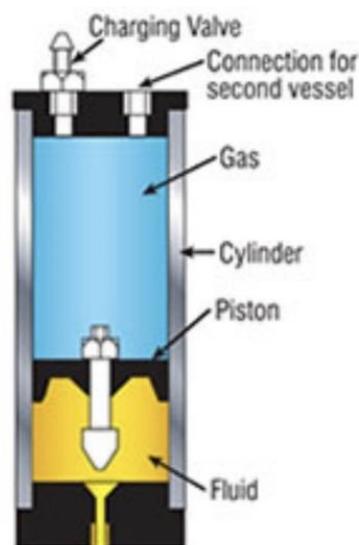


Figure 1 Pressure accumulator

Initially the piston will be fully extended (minimum volume) the pump is stopped and the piston retracted to add a further 60 litres to the volume. As the internal volume of the extraction vessel has been increased, we have decreased the pressure and so introduce a pressure difference increasing the mass transfer from within the wood.

We have 2 parameters we can now control:

- a. The number of pulses
 - b. The period of each pulse
- iii. Addition of a modifier chemical.
- As well as using pure carbon dioxide as our extractant, it can be modified with chemicals which can alter the solubility and surface tension, and therefore potentially increase the mass transfer of the creosote out of the wood. Modifiers, used will be Propanone and Methanol, and are added at a level of approximately 1% and are introduced via a high pressure chromatography pump.

Table 1 Extraction methodology summary

Procedure	Description	Variables	Outcome	Proposed number of extractions
1	Extraction using pure carbon dioxide under dense gas and supercritical conditions	Carbon dioxide pressure, temperature and flow rate	Determination of most appropriate conditions of pressure temperature and flow rate	6 – 10 depending on outcomes
2	Pulsed extraction	Pulse number and duration	Determine whether pulsed procedure improves extraction efficiency	4-6 depending on outcomes
3	Modification with Methanol	Modifier concentration	Determine whether methanol has an effect on extraction efficiency or duration	2-4 depending on outcome
4	Modification with Propanone	Modifier concentration	Determine whether Propanone has an effect on extraction efficiency or duration	2-4 depending on outcome

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