

## DEVELOPMENT OF PETROLEUM ENHANCED COAL TAR PITCH IN EUROPE

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### Abstract

Petroleum enhanced coal tar pitches are proven products in the USA. In Europe, they are a preferred response to market analysis predictions for future pitch demand. The paper will discuss laboratory scale product development based on similar technology to Koppers in the USA, but based predominantly around more local materials for economic and strategic reasons. Successful development has called for the identification of suitable petroleum raw materials and the methodology to combine petroleum and coal tar components to make homogeneous, performance products.

Pitch properties and particularly bench scale anode information are the backbone of the paper. Traditional, standard pitch test results fail to do full justice to the enhanced binder materials. Bench scale anode results indicate unanticipated, positive synergies between coal tar and petroleum derived molecules in an anode or other electrode environment where filler carbon is the largest constituent.

### Introduction

Petroleum enhanced coal tar pitches (PECTP's) are an opportunity to build jointly and separately on the positive characteristics of coal tar materials and the feedstocks from certain petroleum cracking processes. From the beginning they were regarded as separate pitches in their own right and this ensured that the study checked for the unexpected, as a precaution against adverse processing or usage characteristics.

Currently there are adequate supplies of coal tar to satisfy current world-wide pitch requirements but demand and supply imbalances already exist in some areas of the World. North America is presently a net importer of coal tar feedstock and pitch (1). Europe is not yet a net importer at the start of the new Millennium, but far-sighted producers have already recognised the mutual benefits of improving and expanding the raw materials base in Europe and elsewhere

It is sound strategy to develop local implementations of global technical and process developments. This paper unveils the first part of Koppers Europe's intentions for Petroleum Enhanced Coal Tar Pitches (PECTP's).

PECTP's have been marketed industrially for several years in North America (1, 2). High integrity PECTP's are not 'stretched' coal tar pitches and they certainly do not include unattractive streams from the refinery. In fact the reverse is true, sourcing quality petroleum derived material has proved to be essential in Europe, just as it is in North America.

This paper covers laboratory evaluations, however PECTP's have been made on a bigger scale to gain more experience.

### Experimental

#### PECTP Identifiers.

Samples were prepared containing two levels of petroleum content, at two target softening point ranges. See Table 1.

**Table 1 Pitch Identifiers**

| Target S. Pt. Range °C Mettler                         | Petroleum material %    | Identifier |
|--|-------------------------|------------|
| <b>Petroleum Enhanced Coal Tar Pitches</b>             |                         |            |
| 110/115  | ≤ 20                    | PECTP 1    |
| 110/115  | ≤ 50                    | PECTP 2    |
| 120/125  | ≤ 20                    | PECTP 3    |
| 120/125  | ≤ 50                    | PECTP 4    |
| <b>100% Coal tar and Petroleum Pitches as Controls</b> |                         |            |
| 110/115  | CTP control for PECTP 1 | CTP 1      |
| 110/115  | PP control for PECTP 1  | PP 1       |
| 120/125  | CTP control for PECTP 2 | CTP 2      |
| 120/125  | PP control for PECTP 2  | PP 2       |

Standard pitch properties were measured by either ISO or British Standard 6043 derived methods except softening point which used ASTM D 3104 and trace element impurities which were determined by in-house atomic absorption spectrophotometry procedures.

#### Pitch Storage tests

Empirical molten pitch storage tests can reveal information about product behaviour, performance and handling. It is already known that the storage stability performance of coal tar pitch is industrially proven; for new products such as PECTP's it is reassuring to have data from a laboratory test to make comparisons. Laboratory test conditions are more severe than industrial storage conditions because of an unfavourable surface area to volume ratio, so data has to be comparative and not absolute.

"Tall form" 250 ml Pyrex beakers were each loaded with 125 gram of pitch. Each beaker was covered with a fitted aluminium foil cap to limit but not prevent exchange with the external atmosphere. Beakers were placed in an oven controlled so that

the pitch was at  $225 \pm 2^\circ\text{C}$  for the required periods of time. After re-weighing, beaker walls and contents were inspected and the pitch homogenised prior to test.

#### Bench-scale Anode Tests

Bench scale anodes at four binder levels were formed and baked in the laboratory using a programme, based on industry practice, lasting some 72 hours (3). The three-fraction aggregate recipe included 20% by weight of butt material cleaned to current industry standards. This material was restricted to the coarsest fraction.

For each binder pitch, only the two sets of anodes with the optimum and next-to-optimum binder levels, based on anode green dry density, were tested in full.

Air reactivity tests were carried out for one hour at  $525^\circ\text{C}$ . Carbon dioxide reactivity tests were carried out at  $975^\circ\text{C}$  for 5 hours.

### Results and Discussion

#### Pitch Properties

The standard pitch properties in Table 2 show that PECTP 1 and 3 with up to 20% petroleum content are similar to those for anode binder coal tar pitch such as CTP 1 and 2. Petroleum pitches PP 1 and PP 2 had distinctly lower standard coking value (CV) test results, see Table 2 below. They were made using process conditions skewed towards promoting desirable electrode binder properties. PECTP 2 and 4, with up to 50% petroleum content, also reflected this in their CV test results.

The viscosity measurements for all four PECTP's agree with expectation based on softening point. The test results for PECTP 3 and 4 would meet most existing anode binder pitch specifications. PECTP 2 and 4, based on their constitution, offer the prospect of lower PAH content particularly for smelters operating Söderberg cells. The degree of improvement depends on the judgement criteria. These are the subject of intense deliberation at the present time, but there is no clear consensus yet, so no attempt will be made at numerical comparisons.

#### Pitch Storage tests

Behaviour in empirical liquid pitch storage tests, as already stated, gives information about processes taking place over prolonged periods at relatively low temperature ( $225^\circ\text{C}$ ). According to Figure 1 for weight loss, PECTP 1 and 2 behave similarly to pure coal tar pitch CTP 1, which is industrially proven. The petroleum enhanced coal tar pitches show no evidence of the high volatile weight loss exhibited by petroleum pitch control PP 1. The coal tar present in PECTP 1 and 2 appears to dominate weight loss behaviour in a sufficiently beneficial manner to make it a positive point for PECTP's.

Volatile loss or polymerisation, or both, could explain the increases in softening point illustrated in Figure 2. Where softening point increase is concerned, PECTP 1 and 2 behave like the 'pure' coal tar pitches in the study.

Formation of toluene insolubles is another indicator of polymerisation processes, which in excessive amounts at

**Table II Standard Pitch Properties**

| Property                      | PECTP 1 | PECTP 2 | PECTP 3 | PECTP 4 | CTP 1<br>Control for<br>PECTP 1<br>and 2 | PP 1<br>Control for<br>PECTP 1<br>and 2 | CTP 2<br>Control for<br>PECTP 3<br>and 4 | PP<br>2Control<br>for PECTP<br>3 and 4 |
|-------------------------------|---------|---------|---------|---------|--|---|--|--|
| Mettler S. Pt °C              | 112.5   | 113.7   | 120.0   | 123.2   | 113.9                                    | 112.1                                   | 121.7                                    | 119.5                                  |
| Quinoline<br>Insoluble %      | 3.2     | 5.0     | 4.8     | 7.0     | 5.9                                      | 0.1                                     | 7.0                                      | 0.6                                    |
| Toluene<br>Insoluble %        | 22.8    | 25.4    | 23.2    | 27.9    | 27.0                                     | 15.0                                    | 30.6                                     | 9.9                                    |
| Alcan (ISO)<br>Coking Value % | 52.7    | 55.6    | 55.4    | 58.9    | 57.2                                     | 47.3                                    | 59.3                                     | 46.4                                   |
| Density @20°C<br>gcm-3        | 1.266   | 1.293   | 1.271   | 1.301   | 1.308                                    | 1.209                                   | 1.319                                    | 1.208                                  |
| Sulphur %                     | 0.48    | 0.50    | 0.56    | 0.53    | 0.52                                     | 0.32                                    | 0.51                                     | 0.29                                   |
| Pitch at. C/H ratio           | 1.62    | 1.73    | 1.58    | 1.68    | 1.82                                     | 1.35                                    | 1.87                                     | 1.28                                   |
| Volatiles                     | 1.4     | 1.3     | 1.1     | 1.2     | 1.3                                      | 1.6                                     | 1.0                                      | 1.2                                    |
| Ash content %                 | 0.14    | 0.21    | 0.175   | 0.235   | 0.27                                     | 0.02                                    | 0.33                                     | 0.02                                   |
| Sodium                        | 130     | 145     | 145     | 185     | 145                                      | 8                                       | 185                                      | 25                                     |
| Iron                          | 80      | 110     | 85      | 110     | 125                                      | 14                                      | 150                                      | 10                                     |
| Silicon                       | 130     | 195     | 85      | 115     | 230                                      | 15                                      | 190                                      | 10                                     |
| Vanadium                      | <10     | <10     | <10     | <10     | <10                                      | <10                                     | <10                                      | <10                                    |
| Nickel                        | 1       | 1       | 2       | 2       | 1  | 1                                       | 2  | 1                                      |
| Lead                          | 70      | 90      | 75      | 90      | 120                                      | 2                                       | 135                                      | 5                                      |
| Viscosity mPa.s<br>@ 140°C    | 12815   | 12285   | 46000   | 64500   | 13100                                    | 15950                                   | 38800                                    | 51500                                  |
| @ 160°C                       | 2020    | 1980    | 5630    | 6900    | 1940                                     | 2725                                    | 440                                      | 7000                                   |
| @ 180°C                       | 515     | 490     | 1310    |         | 445                                      | 695                                     | 975                                      | 1530                                   |
| @ 200°C                       | 190     | 183     | 375     | 390     | 170                                      | 255                                     | 315                                      | 500                                    |
| @ 220°C                       | 100     | 97      | 158     | 165     | 92                                       | 115                                     | 135                                      | 185                                    |

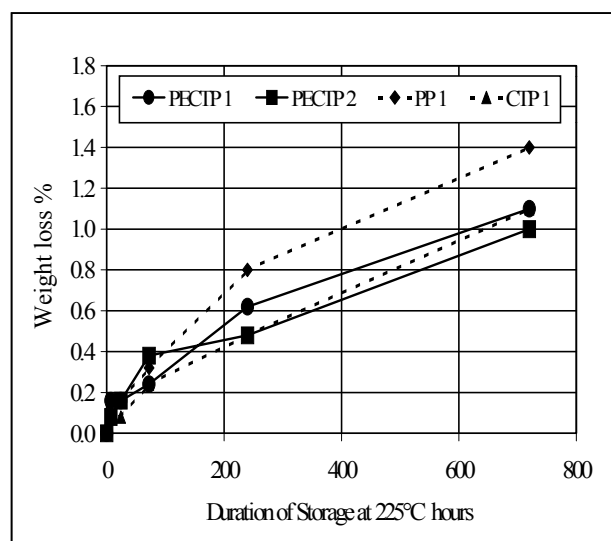


Figure 1: Weight Loss vs Storage Duration – PECTP 1 and PECTP 2 compared with CTP and PP Controls

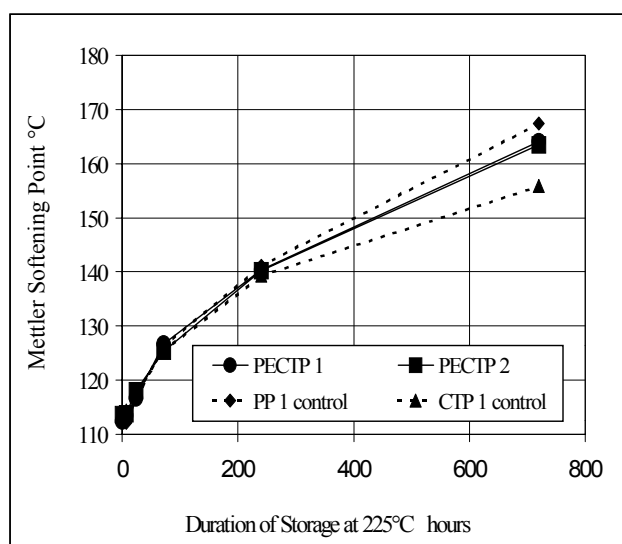


Figure 2: Mettler S.Pt. vs Storage Duration – PECTP 1 and PECTP 2 compared with CTP and PP Controls

relatively low temperatures is considered undesirable in an anode binder pitch. It makes no difference whether the petroleum material is present at up to 50% (PECTP 2) or only 20% (PECTP 1), the enhanced pitches follow closely the behaviour of pure coal tar pitch control CTP 1. See Figure 3.

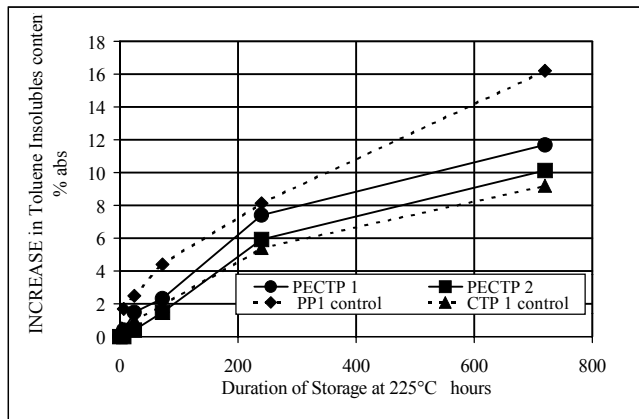


Figure 3: Increase in Toluene Insolubles vs Storage Duration – PECTP 1 and 2 compared with CTP and PP Controls

Figures 4, 5 and 6, which illustrate the same properties for 120/125 Mettler counterparts, convey very similar information to the Figures discussed above. PECTP 3 and 4 again behave like the well proven 'pure' coal tar pitch control, CTP 2. They show even lower weight losses (Figure 3) than PECTP 1 and 2 (Figure 1) because the softening points are some 10°C higher. The increase in Mettler softening point and the rise in toluene insolubles content for PECTP 3 and 4, again follow their coal tar control, CTP 2., rather than petroleum control PP 2, see Figure 6.

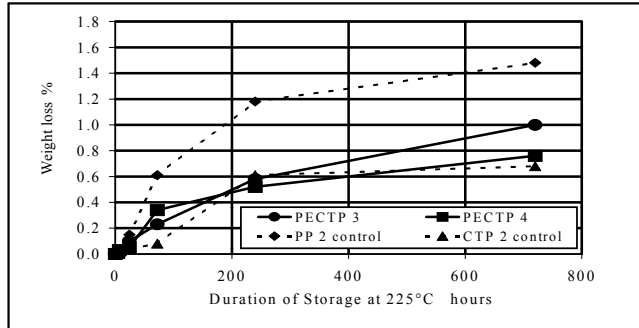


Figure 4: Weight Loss – PECTP 3 and 4 with CTP & PP Controls

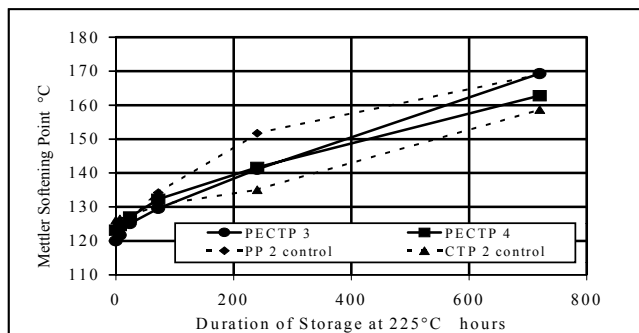


Figure 5: S.Pt. Increase – PECTP 3 and 4 with CTP & PP Controls

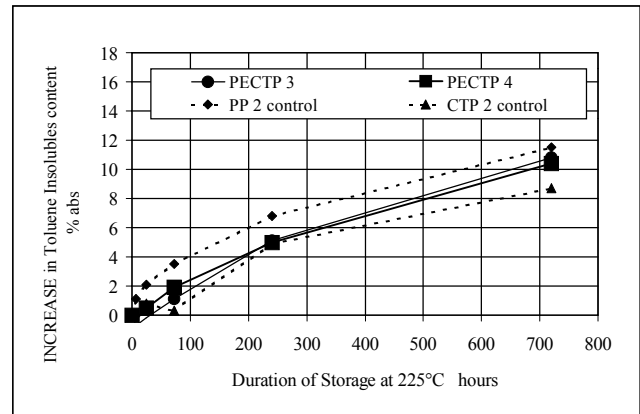


Figure 6: Increase in Toluene Insolubles – PECTP 3 and 4 with CTP & PP Controls

### Bench-scale Anode Tests

The bench scale anode results are good indicators for the likely behaviour of Enhanced pitches in industrial anode production. All the basic results are in Table 3. Key basic data and dry density values derived from them are illustrated in Figures 7 to 16.

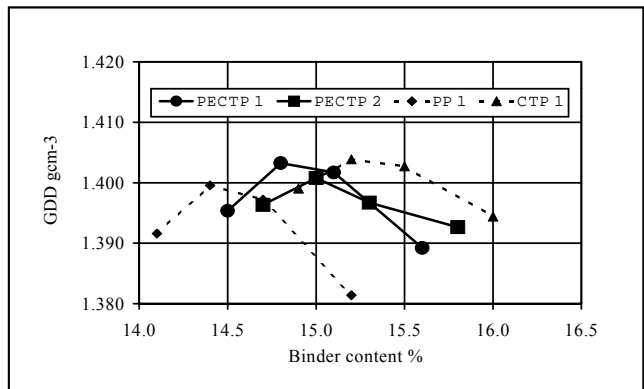


Figure 7: Anode Green Dry Density using PECTP 1 and 2 with CTP and PP Controls

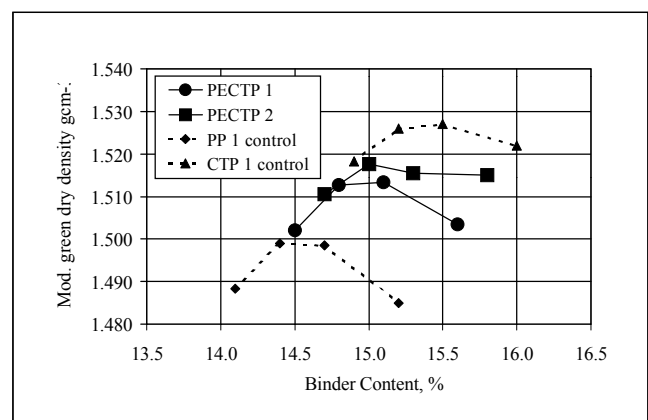


Figure 8: Modified Anode Green Dry Density using PECTP 1 and 2 with CTP & PP Controls

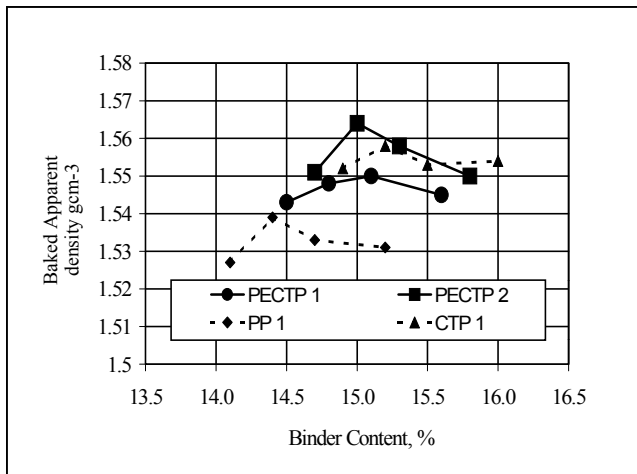


Figure 9: Anode Baked Apparent Density using PECTP 1 and 2 with CTP and PP Controls

The optimum binder content for each pitch, estimated from the peak in the dome-shaped curves for green dry density (Figures 7 and 12), lie within the range of binder contents used to fabricate the anodes.

While the curves for the PECTP's are sufficiently dome-shaped to identify preferred binder levels, they also show a sufficiently flat response to increased pitching rate, so they are unlikely to be over-sensitive to process weight variations when used on an industrial scale. In this behaviour they are similar to proven 'pure' coal tar pitches. With 'pure' petroleum pitches, green dry density was much more sensitive to small changes in pitching level; such sensitivity would make anodes more vulnerable to process variation factors in an anode carbon plant, see Figures 7 and 12. Whether this higher level of sensitivity is acceptable cannot be established from the laboratory data.

The optimum binder contents for PECTP are one-half to one percent less those for CTP 1 and CTP 2, but higher than PP 1 and PP 2. Optimum binder content has increased in pitch QI order although other factors may well be important in determining the best pitching level with PECTP's

Modified green dry density data, (Figures 8 and 13), where the pitch coking value test results are included, give expectation values for the baked apparent density. Comparing these projections with the actual baked density data in Figures 9 and 14 shows that PECTP's 1,2,3 and 4 all exceed expectation because the anodes are just about the equal of the appropriate coal tar control in this respect.

It is binder carbon yield in an anode environment that is vitally important for successful industrial application. This important finding cannot be over-emphasised because it is characteristic of the PECTP's discussed in this paper.

Baked apparent density is relatively insensitive across a 1.1% absolute binder content range for all the PECTP's where the graph lines are only very slightly curved, see Figures 9 and 14. Such behaviour is often seen as an indicator of tolerance to inevitable process variation in industrial usage

A measure of in-situ carbon yield can be extracted from the bake weight loss and original binder content. This data is illustrated in Figures 10 and 15, which shows that all four PECTP's out-perform their simple, pitch only coking value test results. In contrast, both of the petroleum pitch controls show a sudden and sharp decline in carbon yield, even when the degree of over-pitching is very small, such as 0.3% 'too much'.

The data for 115/120 Mettler pitch (Figures 14 and 15) show that densities and carbon yields are generally higher compared with anodes containing 110/115 softening point binders, (Figures 9 and 10). Higher softening point pitch brings with it higher costs, which would have to be recovered by the producer even if the impact on yield and quality of other tar products can be accommodated.

The anode porosity data (Figures 11 and 16) independently show that all four PECTP's contribute to anodes with porosities as low as those achieved with the coal tar control pitches. This finding must be attributed in large part to the quantity and structure of the carbon binder matrix produced in these sets of anodes.

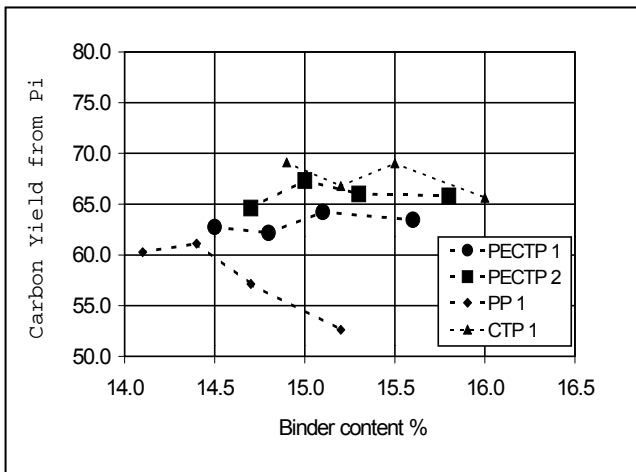


Figure 10: Carbon Yield from Binder in the Anode (approx.)

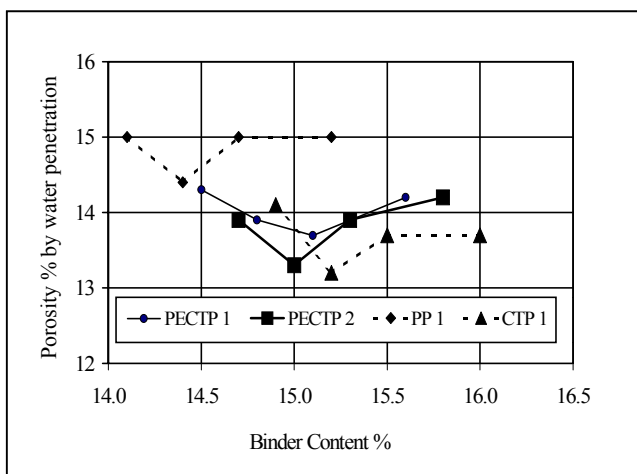


Figure 11 Anode Porosities with PECTP 1 and 2 Compared with CTP and PP controls

**Table 3 Laboratory Prebake Anodes made with Petroleum Enhanced Coal Tar Pitch (PECTP)**

|                       | PECTP 1                                     |         |         |         | PECTP 2' |         |         |         | PECTP 3                                     |         |         |         | PECTP 4 |         |         |         |
|-----------------------|---|---------|---------|---------|----------|---------|---------|---------|---|---------|---------|---------|---------|---------|---------|---------|
| Pitch content %w/w    | 14.5  | 14.8    | 15.1    | 15.6    | 14.7     | 15.0    | 15.3    | 15.8    | 14.7  | 15.0    | 15.3    | 15.8    | 15.0    | 15.3    | 15.6    | 16.1    |
| Green density gcm-3   | 1.632                                       | 1.647   | 1.651   | 1.646   | 1.637    | 1.648   | 1.649   | 1.654   | 1.643                                       | 1.653   | 1.655   | 1.653   | 1.649   | 1.66    | 1.662   | 1.666   |
| Bake loss to 1080°C   | 5.4   | 5.6     | 5.4     | 5.7     | 5.2      | 4.9     | 5.2     | 5.4     | 4.8   | 4.7     | 4.6     | 5       | 4.1     | 4.0     | 4.2     | 4.5     |
| Baked density gcm-3   | 1.543                                       | 1.548   | 1.550   | 1.545   | 1.551    | 1.564   | 1.558   | 1.550   | 1.552                                       | 1.561   | 1.556   | 1.554   | 1.562   | 1.573   | 1.572   | 1.570   |
| Elec. Resistiv. ohm.m | 9.3E-05                                     | 8.3E-05 | 8.8E-05 | 9.5E-05 | 9.4E-05  | 8.4E-05 | 9.2E-05 | 9.6E-05 | 7.7E-05                                     | 7.5E-05 | 7.2E-05 | 7.7E-05 | 7.9E-05 | 7.3E-05 | 7.2E-05 | 6.9E-05 |
|                       | <b>Reactivity to Carbon dioxide @ 975°C</b> |         |         |         |          |         |         |         | <b>Reactivity to Carbon dioxide @ 975°C</b> |         |         |         |         |         |         |         |
| Residue %             |   | 79.9    | 79.5    |         |          | 79.0    | 77.2    |         |   | 80.2    | 80.0    |         |         | 83.5    | 80.8    |         |
| Gas loss %            |   | 9.2     | 9.3     |         |          | 9.7     | 10.7    |         |   | 8.7     | 8.3     |         |         | 7.6     | 8.5     |         |
| Dust loss %           |   | 10.9    | 11.2    |         |          | 11.3    | 12.1    |         |   | 11.1    | 11.7    |         |         | 8.9     | 10.7    |         |
|                       | <b>Reactivity to Air @ 525°C</b>            |         |         |         |          |         |         |         | <b>Reactivity to Air @ 525°C</b>            |         |         |         |         |         |         |         |
| Residue %             |   | 79.4    | 78.9    |         |          | 78.7    | 76.8    |         |   | 82.9    | 85.1    |         |         | 82.0    | 83.1    |         |
| Gas loss %            |   | 10.1    | 10.9    |         |          | 10.5    | 11.3    |         |   | 8.3     | 7.7     |         |         | 8.9     | 8.9     |         |
| Dust loss %           |   | 10.5    | 10.2    |         |          | 10.8    | 11.9    |         |   | 8.8     | 7.2     |         |         | 9.1     | 8.0     |         |

**Table 4 Laboratory Prebake Anodes made with Coal tar and Petroleum Control Pitches**

|                       | PP 1 control                                |         |         |         | CTP 1 control |         |         |         | PP2 control                                 |         |         |         | CTP 2 control |         |         |         |
|-----------------------|---|---------|---------|---------|---------------|---------|---------|---------|---|---------|---------|---------|---------------|---------|---------|---------|
| Pitch content %w/w    | 14.1  | 14.4    | 14.7    | 15.2    | 14.9          | 15.2    | 15.5    | 16.0    | 14.2  | 14.5    | 14.8    | 15.3    | 15.1          | 15.4    | 15.7    | 16.2    |
| Green density gcm-3   | 1.623                                       | 1.635   | 1.638   | 1.640   | 1.644         | 1.655   | 1.660   | 1.660   | 1.629                                       | 1.642   | 1.646   | 1.636   | 1.648         | 1.655   | 1.660   | 1.661   |
| Bake loss to 1080°C   | 5.6   | 5.6     | 6.3     | 7.2     | 4.6           | 5.1     | 4.8     | 5.5     | 5.9   | 6.3     | 6.3     | 6.6     | 3.9           | 3.9     | 4.1     | 4.5     |
| Baked density gcm-3   | 1.527                                       | 1.539   | 1.533   | 1.531   | 1.552         | 1.559   | 1.553   | 1.554   | 1.536                                       | 1.545   | 1.536   | 1.531   | 1.57          | 1.578   | 1.568   | 1.564   |
| Elec. Resistiv. ohm.m | 8.3E-05                                     | 8.4E-05 | 8.1E-05 | 8.7E-05 | 9.2E-05       | 8.4E-05 | 9.1E-05 | 9.1E-05 | 9.0E-05                                     | 8.0E-05 | 8.1E-05 | 8.4E-05 | 7.7E-05       | 7.3E-05 | 7.2E-05 | 7.0E-05 |
|                       | <b>Reactivity to Carbon dioxide @ 975°C</b> |         |         |         |               |         |         |         | <b>Reactivity to Carbon dioxide @ 975°C</b> |         |         |         |               |         |         |         |
| Residue %             | 74.3  | 78.0    |         |         |               | 79.5    | 76.5    |         |   | 75.9    | 76.2    |         |               | 77.2    | 78.1    |         |
| Gas loss %            | 11.9  | 9.7     |         |         |               | 10.1    | 11.2    |         |   | 8.5     | 10.7    |         |               | 11.2    | 10.9    |         |
| Dust loss %           | 13.8  | 12.3    |         |         |               | 10.4    | 12.3    |         |   | 15.6    | 13.1    |         |               | 11.6    | 11.0    |         |
|                       | <b>Reactivity to Air @ 525°C</b>            |         |         |         |               |         |         |         | <b>Reactivity to Air @ 525°C</b>            |         |         |         |               |         |         |         |
| Residue %             | 79.4  | 79.7    |         |         |               | 75.0    | 75.1    |         |   | 81.8    | 82.9    |         |               | 76.3    | 81.5    |         |
| Gas loss %            | 10.4  | 10.1    |         |         |               | 12.2    | 12.4    |         |   | 7.7     | 8.2     |         |               | 13.6    | 9.7     |         |
| Dust loss %           | 10.2  | 10.2    |         |         |               | 12.8    | 12.5    |         |   | 10.5    | 8.9     |         |               | 10.1    | 8.8     |         |

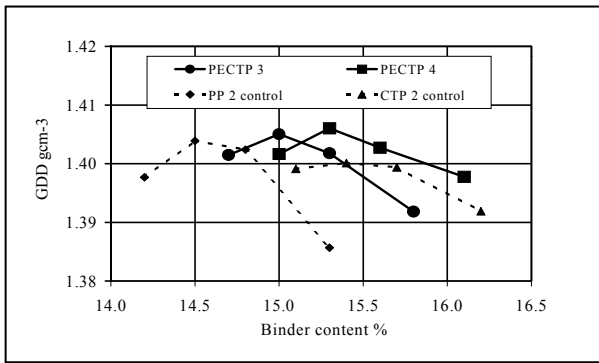


Figure 12: Anode Green Dry Density using PECTP 3 and 4 with CTP and PP Controls

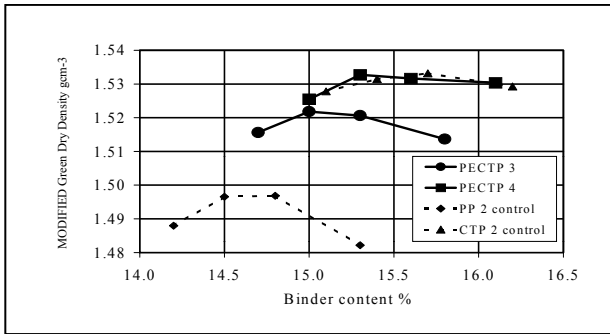


Figure 13: Modified Anode Green Dry Density using PECTP 3 and 4 with CTP & PP Controls

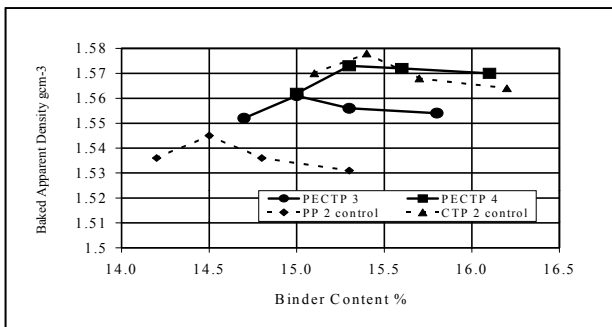


Figure 14: Anode Baked Apparent Density using PECTP 3 and 4 with CTP and PP Controls

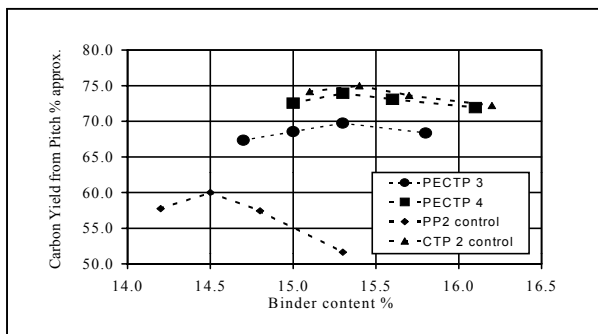


Figure 15: Carbon Yield from Binder in the Anode (approx.)

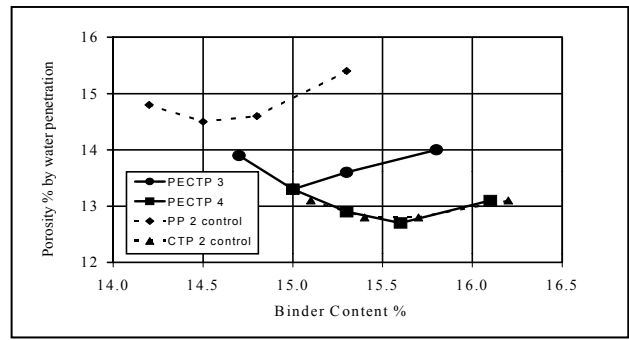


Figure 16: Porosities for PECTP 3 & 4 vs CTP and PP controls

Electrical resistivities reported in Tables 3 and 4 are higher than industry standards but reflect the modest baking temperature of 1080°C maximum, as well as other factors associated with the physical size of the bench scale anodes. PECTP 1 and 2 were about equal at optimum binder level with their controls, (Table 3).

Anodes made with the higher softening point PECTP 3 and 4 were lower in resistivity than their controls, CTP 2 and PP 2 (Table 4). Electrical resistivities that are very similar to those achieved with industrial coal tar pitches are another positive point for petroleum enhanced pitches.

Carbon dioxide and air reactivity results (Tables 3 and 4) have to be considered in broader terms because the methods are not sufficiently sensitive to compare small differences in the data. We have found generally that anodes made with petroleum pitch give better gas loss figures whereas coal tar pitches return lower dust losses. The results for PECTP 1 and 2 (Table 3), made with 110/115 Mettler pitch, generally equalled the gas loss for PP1 anodes and the dust loss for CTP 1. (See Table 4 for control pitch anodes). In other words, PECTP 1 and 2 equalled whichever of the controls had the better performance. The verdict is the same for the 120/125 softening point PECTP 3 and 4 compared with controls CTP 2 and PP 2.

The air and carbon dioxide reactivity results are very encouraging for petroleum enhanced coal tar pitches.

### Conclusions

Results for PECTP's with up to 20% or up to 50% petroleum material are as good as, and possibly better, than similar softening point controls made separately from 'pure' coal tar and 'pure' petroleum materials. This conclusion applies equally to pitches targeted at the 110/115 and 120/125 Mettler softening point ranges.

The carbon yield in bench scale anodes, baked anode densities, porosities and resistivities all indicate that lower pitch-only coking value test results for PECTP's are balanced by higher conversion to carbon in an anode environment.

Petroleum enhanced coal tar pitches must be judged on their performance and not simply on their standard test properties.

PECTP's in the molten state are more similar to less thermally active coal tar pitch rather than petroleum pitch, which can change properties more quickly and is therefore less easy to store and use.

PECTP's offer advantages besides lower PAH levels to the anode carbon industry. These can best be exploited by co-operative trials with progressive anode producers and smelters in the European area just as they have been on the American continent.

### **References**

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