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# Controlled separation of low temperature coal tar based on solvent extraction–column chromatography



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#### ABSTRACT

The heavy tar (H-tar) of low temperature coal tar was collected from a low temperature carbonization plant in Shanbei. H-tar was separated into eight fractions by silica-gel column chromatography (SGCC) with a binary eluent of *n*-hexane and ethyl acetate (EA). A series of alkanes (C14–C28), phenolic compounds and aromatic nitrogen species were fractionated from H-tar by SGCC and analyzed by gas chromatography–mass spectrometry (GC–MS). Most of the GC–MS detectable phenolic compounds are phenol, cresol, xylenol, C3–C4 alkyl phenols, indanols, naphthalenols, methyl naphthalenols, benzenediols, fluorenols, phenanthrols and pyrenol, among which cresol is the most abundant one and pyrenol is the largest molecular weight one. According to the change rule of phenolic compounds with the polarity of eluting solvents, the solvent extraction–column chromatography (SECC) device and crude phenol recovery processing have been designed and developed, which could realize the separation of neutral oil, pitch and crude phenol from low temperature coal tar.

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#### 1. Introduction

Low temperature coal tar is an important byproduct of low temperature carbonization industry and its yield is up to ten thousand tons every day in Shanbei (Northern Shaanxi Province in China) [1]. About one third of low temperature coal tar is phenols [2]. Phenolic compounds are important chemical materials, which have been widely used in industry, but phenols are harmful substances in the process of oil hydrogenation, and they will cause some negative influences on the hydrogenated oil [3]. Therefore, it is necessary to extract phenols from low temperature coal tar. What is important is that the separation of phenols could be a key in achieving the separation and utilization of low temperature coal tar. Up to now, the separation of phenols from coal tar has been studied for decades and a great deal of work has been done by a number of researchers [4-7], e.g., extraction with selective solvents [8,9] and precipitation using ions of soluble metal salts [10]. So far, the well-known method of acid-base extraction is the only way to recover phenolic compounds from coal tar in industry [11, 12]. Compared with the above methods, acid-base extraction is the simplest and most mature way. However, all of them have some disadvantages: the low yield of crude phenol (especially the lower extraction

\* Corresponding author. *E-mail address:* maxym@nwu.edu.cn (X. Ma). yield of senior phenols), complicated technological process, disposal of waste water and waste residue, etc. [13,14].

It is very important to develop new methods or processes to extract phenolic compounds from coal tar. Column chromatography is an important technique for separating soluble organic matter with complex composition, including coal extracts [15], petroleum asphaltenes [16] and crop-derived bio-oils [17], which are similar to coal tar. The samples were eluted by changing the polarity of eluting solvents, and then the eluted products such as aliphatic hydrocarbons, nitrogen compounds were obtained [18–21]. In this work, a new method of solvent extraction–column chromatography (SECC) equipment and crude phenol recovery processing were designed and developed. Low temperature coal tar was separated into three parts: crude phenol, neutral oil and pitch.

 Table 1

 Some characteristics of H-tar.

Property	Value
Density (20 °C/g·mL <sup><math>-1</math></sup> )	1.05
Viscosity (80 °C/mPa·s)	14.10
Moisture (%)	1.66
Ash (%)	0.15
Toluene insolubles (%)	0.46

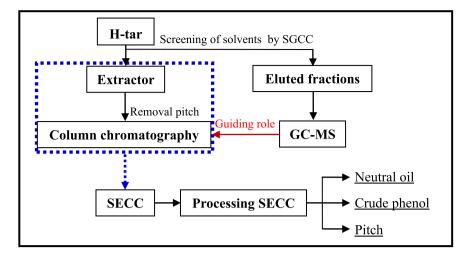


Fig. 1. The separation process of H-tar.

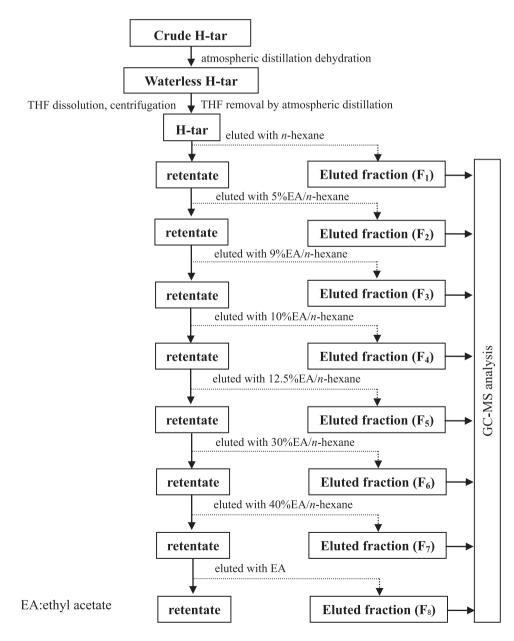


Fig. 2. The process of screening eluting solvents by SGCC.

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