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HUMATE IN THE UPPER CRETACEOUS FRUITLAND FORMATION IN NORTHWESTERN NEW MEXICO

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ABSTRACT—Humate is a naturally occurring, high humic acid content material. It is associated with weathered coal and humate-rich mudstones and shales in the Upper Cretaceous Fruitland Formation in northwestern New Mexico. These humate-bearing deposits have been mined primarily to produce humic substances used as soil amendments. Mining of humate in New Mexico started in the 1970s, and nearly 60,000 metric tons were mined by six operators in 2016. Humic substances are water soluble and are beneficial to soils and plants. They are high molecular weight molecules (generally between 5,000 to 50,000 g/mol), with complex structures, large surface areas and cation-exchange capacities, and multiple functional groups. These properties facilitate molecular binding to salts, metals, and hydrophobic compounds in soils and water. Humic substances may play a key role in controlling the aqueous concentrations, mobility, bioavailability and toxicity of contaminants in the environment.

INTRODUCTION

Mining and the production of humate resources in northwestern New Mexico has been growing steadily since the 1970s. Mineable humate deposits are associated with weathered coal and organic-rich Upper Cretaceous sedimentary rocks in the San Juan Basin. This paper focuses on the humate associated with the Upper Cretaceous Fruitland Formation in the Star Lake Coal Field, about 48 km west of Cuba, New Mexico (Fig. 1). Humate and humic substances are formed from the decay of plants and the weathering of the coal deposits. Humic substances are complex compounds of polymeric organics with molecular weights ranging from a few hundred to several hundred thousand or even a few million grams per mole. Humic substances are used primarily as soil amendments but also have or have had applications in pharmaceuticals, wood finishing, water treatment, hydrocarbon remediation, and as drilling fluid additives. There may also be future applications in mine-waste remediation.

Shomaker and Hiss (1974) provided an overview of humate mining in northwestern New Mexico in the 1970s. At that time, humate and humic substances were recognized largely as soil amendments with agricultural applications. Roybal and Barker (1987) described the state of the industry in 1980s and the growing use of humic materials in other applications. This paper provides an update and outlines the growing humate mining and production industry in New Mexico.

WHAT IS HUMATE?

Humate terminology can be confusing, particularly as it relates to its use in scientific, mining and business terms (Hoffman et al., 1994; Table 1). The weathering and decay of organic matter and coal produces dark-colored humate. Humate contains humic substances, which are naturally occurring het-

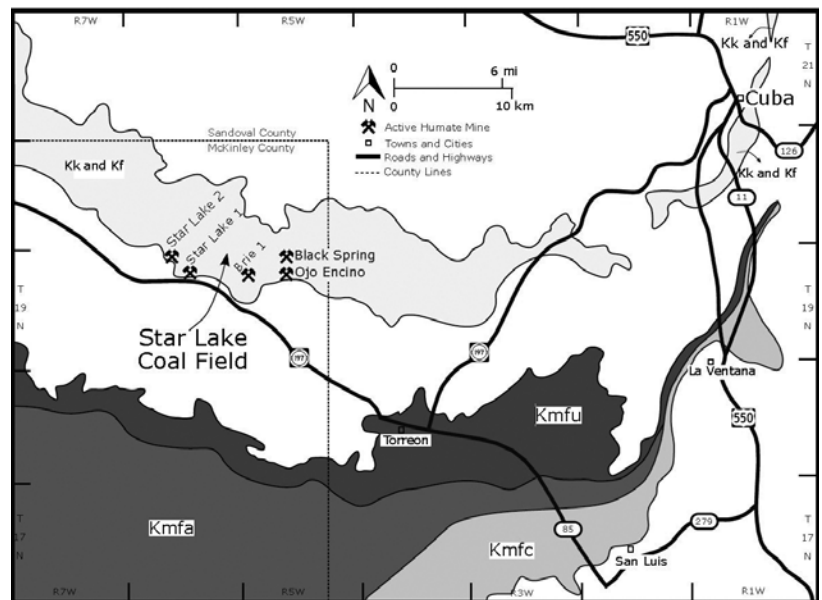


FIGURE 1. Map of Star Lake Coal Field area showing active humate mines in northwest New Mexico. Kk = Kirtland Formation; Kf = Fruitland Formation; Kmfu = Menefee Formation, upper coal bed; Kmfa = Menefee Formation, Allison Member; Kmfc = Menefee Formation, Cleary Coal Member. Geology modified after Hoffman et al., 1992. Locations of active humate mines as listed with the New Mexico Mining and Minerals Division as of 2019.

TABLE 1. Terms and definitions for humic acid-rich and associated materials (modified after Roybal and Barker, 1987).

Terminology	Definition
Carbonaceous Mudstone/Shale	Called humate by geologists if it contains base-soluble humic acids
Fulvate	Salt of fulvic acid
Fulvic Acid	Base-soluble and acid-soluble fraction of humate; fraction of humic substances soluble in water under all pH conditions
Humalite	A soft, brown coal-like material, which has many similarities to leonardite (an informal term, used in Alberta, Canada) that occurs as a weathering product of sub-bituminous coal and carbonaceous shales
Humate (singular)	Geologically a weathered coal (brown coal), carbonaceous claystone/mudstone/shale rich in humic matter
Humates (plural)	Chemically, salts of humic acids
Humic Acid (singular)	Base-soluble organic material; fraction of humic substances not soluble in water under acidic conditions (below pH of 2), but soluble at higher pH
Humic Acids (plural)	Humic, fulvic and ulmic acids; hydrogen ion in exchange sites; Base soluble organic material insoluble in acid
Humic Substances (Matter, Substances, Deposits)	A general category of naturally occurring, biogenic, heterogeneous organic substances that have a high molecular weight and are refractory; completely decomposed organic matter containing humic acid
Humins(s)	Chemically an alkali-insoluble fraction of humate; fraction of humic substances not soluble in water at any pH value
Humus	Chemically an alkali-soluble fraction of humate; lithologically a carbonaceous mudstone or shale; oxidized (weathered) lignite or coal, rich in organic matter; dark, organic, well decomposed soil material consisting of plant, and animal residues and various inorganic elements
Leonardite	Oxidized (weathered lignite); sometimes includes weathered sub-bituminous coal, but this is not the preferred term for weathered coal
Ulmate	Salt of ulmic acid
Ulmic Acid	Base-soluble organic material that is acid insoluble then alcohol soluble
Weathered Coal	Oxidized coal that contains humic acids due to the weathering process

erogeneous mixtures of organic materials. Humification is the process of forming these substances as a product of the decomposition of organic material during weathering and degradation of plant material (Tan, 2014). Humus is the alkali-soluble fraction of humate. Even though humic acid was first extracted from humus in 1786, it wasn't until the 1970s that the technology was available to assess the chemical structure of the molecule in some detail (Susic, 2001).

The humic substance molecule has a high molecular weight (generally between 5,000 to 50,000 g/mol). These molecules are partly colloidal and weakly acidic due to their humic acid and fulvic acid contents (Roybal and Barker, 1987). Humic substances are not pure materials, which has resulted in ambiguous use by geologists, chemists, soil scientists, agronomists and producers. For instance, the weathered coal that is mined for its humic acid content is an extremely variable mixture of base-soluble humic, fulvic and ulmic acids, as well other organics and their salts.

The use of the term "humic acid" varies between geology and chemistry with geological humic acid including additional smaller molecules and having a greater acidity than chemical humic acid derived in the laboratory (Roybal and Barker, 1987). Humic acids are structurally complex molecules and are amorphous with poorly defined x-ray patterns. Molecular structural models for humic substances can be broadly divided into two main types, the "macromolecular" and "supramolecular mixture" (Billingham, 2012). Billingham (2012) provides a summary of the various molecular structural theories and the history of research associated with them.

Humic substances have large cation-exchange capacities (CEC) ranging from 200 to 500 milliequivalents per 100 grams at a pH of 7. The wide range in CEC can be explained by the variations in acidity between humic acid and humate (chemical), because a molecule with hydrogen-filled exchange sites yields humic acid with lower pH than a molecule with sites filled with other cations that yield chemical humate. Therefore, the determination of various forms of humate in the laboratory and for classification purposes is generally operationally defined (Hoffman et al., 1992).

Soil humic substances are known to be beneficial for soils and plants (Billingham, 2012; Tan, 2014). As a conditioner of soils, humate generally increases soil aggregation, water-holding capacity and improves soil aeration and permeability. Chemically, it increases the CEC and provides a stronger buffering capacity to resist chemical changes in the soils. Although some of the literature regarding humate benefits is promotional and the effectiveness and value of humic substances is not always clear, there are many published studies showing the direct and indirect benefits to plant growth when tested under controlled conditions (Tan, 2014; Wright and Lenssen, 2013; Billingham, 2012; Hartwigsen, and Evans, 2000; Lee and Bartlett, 1976; Lyons and Genc, 2016).

Chemists consider humate to be a salt of humic acid, a comparatively restricted usage. The term humate is used lithologically, in geology and mining, for mud rocks rich in organic acids, for weathered coal and humic-acid containing organics found in the pore spaces in sandstones. The later usage is applied throughout the rest of this paper.

GEOLOGY AND COAL ASSOCIATION

This paper focuses on one author's experience with mining operations and the development of humate resources at Menefee Mining Company's Black Spring Mine. At the Black Spring Mine, humate is mined from open cuts in weathered coal beds in the uppermost part of the Cretaceous Fruitland Formation (Dames and Moore, Inc., 1979; Schneider and Kirschbaum, 1981; Fig. 2). The Black Spring Mine is in the eastern part of the Star Lake Coal Field, which is described in Shomaker et al. (1971). Humate deposits also occur in some of the older Cretaceous sedimentary rocks that underlie the Fruitland Formation in the San Juan Basin. These humate deposits include the weathered coal and organic-rich shales in the Menefee and Crevasse Canyon Formations (Mesaverde Group) east and south of the Black Spring Mine (Roybal and Barker, 1987).

The Star Lake Coal Field is in the southeasternmost part of the San Juan Basin. The San Juan Basin is an asymmetric structural depression in northwestern New Mexico that also extends into a small part of northeastern Arizona, southern Utah and southern Colorado. According to Fassett and Hinds (1971), the depression contains sedimentary rocks of Cambrian, Devonian, Mississippian, Pennsylvanian, Permian, Triassic, Jurassic, Late Cretaceous, Tertiary and Quaternary age. The maximum known thickness of sedimentary rocks is at least 4300 m in the deepest part of the basin. Late Cretaceous rocks, of which the Fruitland Formation is part, are more than 1800 m thick. These rocks, which contain coal and humate deposits, consist largely of intertonguing marine and non-marine units that represent three basin-wide transgressive-regressive cycles of deposition (Fassett and Hinds, 1971).

The final regression of the Cretaceous seaway resulted in deposition of the marine Pictured Cliffs Sandstone (a point-bar sand deposit, with the sea on the northeast side and swampy

areas on the south side between the bar and the land). The Pictured Cliffs Sandstone is overlain by and intertongues with the Fruitland Formation. The Fruitland Formation is overlain by the Kirtland Formation. With the withdrawal of the seaway, uplift within the southern Rocky Mountains, and structural deformation to the San Juan Basin, terrestrial sediments were deposited over the Kirtland strata. These later units included the Upper Cretaceous to Paleogene Ojo Alamo Sandstone and Paleogene to early Eocene Nacimiento Formation and San Jose Formation. In the Star Lake Coal Field, this deposition occurred episodically adjacent to the active Nacimiento uplift (Smith, 1992). Late Cretaceous and Paleogene terrestrial strata record the local disruption and partitioning of the Cretaceous foreland basin and retreat of the seaway by Laramide time (~65 million years ago).

The Fruitland Formation thickness in the San Juan Basin ranges between 61 and 91 m. In the Star Lake field area, the Fruitland Formation is thinner, ranging from 15 to 30 m due to depositional thinning in the eastern part of the San Juan Basin and erosion prior to deposition of the Ojo Alamo Sandstone.

Since the Fruitland Formation grades upwards into the Kirtland Formation, it is somewhat arbitrarily mapped as it varies spatially. Smith (1992) provided a summary of the stratigraphy of these rocks in the southeastern part of the San Juan Basin and indicated that the top of the Fruitland Formation should be mapped at the top of the highest coal bed or carbonaceous shale bed (or a correlative thin shale) above the last coal (Ayers et al., 1990; Hoffman et al., 1992).

The Fruitland Formation, in the Star Lake Coal Field, dips generally from one to five degrees to the north-northwest (Hoffman et al., 1992). In the Black Spring Mine area, the Fruitland and associated coal/humate beds occur approximately parallel to the depositional strike (essentially parallel to the shoreline of the Cretaceous seaway). The terrestrial direction was south-southwest, and the sea was to the north-northeast.

The Fruitland Formation contains an abundant record of biota that lived along part of the western shore of North America in the Late Cretaceous (Lucas and Mather, 1983). Fruitland strata are also the major coal-bearing unit in Upper Cretaceous rocks, and it reflects depositional environments associated with extensive marshy habitats (Hunt and Lucas, 1992). Detailed sedimentology indicates the Fruitland Formation was deposited as mixed terrestrial-marine facies along a shoreline locally influenced by deltaic complexes that developed along river systems and extended into the sea. Surface drainages associated with these river systems flowed to the northeast at approximate right angles to the Cretaceous seaway coastline. The overlying Kirtland Formation sediments are largely fluvial in nature and formed as the land advanced to the northeast and the seaway retreated in that direction. These fluvial systems were believed to be low-sinuosity meandering and braided streams with well-drained floodplains. With the migration of the seaway north-eastward, no more coal was formed. Fruitland Formation depositional environments represent a transitional phase between completely marine and completely continental deposits during shoreline regression. Periods when the rate of retreat of the shoreline was slow, relatively stable swamp areas developed,



FIGURE 2. Humate deposit in Cretaceous Fruitland Formation at the Black Spring Mine. Note pen in right center of photograph for scale (see arrow).

which resulted in thicker coal deposits in the lower part of the Fruitland (e.g., the Carbonero bed, with a thickness of up to 24 m of coal and partings).

HUMATE MINING IN NEW MEXICO

The mining of humate appears to have started in New Mexico in the early 1970s. There were two operators mining humate, and one deposit in development in 1974 (Shomaker and Hiss, 1974). Roybal and Barker (1987) reported a production of 12,293 m³ (an estimated 12,270 metric tons, assuming a dry bulk density of 1 g/cm³) of humate in 1983. The growth of humate production since the early 1980s has been relatively steady, and the totals since 1989 are shown in Table 2. Production from 1989 to 2016 has reflected a nearly 10-fold increase, and in 2018 there were five operators working various properties within New Mexico and four operators in the Star Lake Coal Field (Fig. 1).

Weathered coals of the Upper Cretaceous of northwestern New Mexico contain many millions, and possibly billions, of tons of humate (Shomaker and Hiss, 1974). Most of the mining of humate has focused on the thicker, near-surface, deposits that are close to processing facilities in Cuba, New Mexico.

The Black Spring Mine is permitted as a Minimal Impact Mine Project under the New Mexico Mining Act and began operations in 2011. The humate is mined from shallow cuts, and, as mining is advanced into new areas, the old cuts are concurrently reclaimed. Using loaders, the run-of-mine material is transported by truck to a production plant in Cuba, New Mexico. The material is stockpiled at the plant, crushed and screened to uniform particle-size fractions, and bagged for sale (granular products; Earthgreen Products Inc., 2020). The finest size fraction is dissolved in water and then dried to form a concentrated water-soluble powder (powdered products). This material is packaged in drums for sale.

CURRENT USES OF HUMIC SUBSTANCES

There are generally two types of products produced from mined humate in New Mexico, powdered and granular forms. Today, the primary applications from these products are in agriculture, with additional uses in industrial, animal feed and pharmaceuticals (Billingham, 2012). Hoffman and Austin (2006) described the use of humic substances primarily as soil amendments and discussed other end uses, such as dispersants to control viscosity in drilling fluids, as stabilizers for ion-exchange resins in water treatment, as stains in wood finishing, and as binders for briquets made from lignite char. Humate and humic substances have also been examined to determine their suitability in the remediation of hydrocarbon-, metal- and salt-contaminated soils and groundwater (Mosley, 1998; Bezuglova and Shestopalov, 2005).

HUMATE GEOCHEMISTRY

Essington (2004) defines humate as very complex, amorphous mixtures of highly heterogeneous chemically reactive,

TABLE 2. Humate production in New Mexico between 1989 and 2018 (from New Mexico Energy, Minerals and Natural Resources Department records; this does not include production from tribal lands and reservations, as one operator produced from trust lands, and there has been additional production from the Navajo Coal Mine).

Year	Number of Operating Mines	Production (metric tons)
1989	2	6418
1990	0-1	not reported
1991	3	9084
1992	3	7737
1993	2	1401
1994	2	6897
1995	0-1	not reported
1996	0-1	not reported
1997	0-1	not reported
1998	2	10,891
1999	3	9575
2000	2	10,254
2001	4	17,536
2002	3	18,397
2003	5	15,645
2004	5	17,848
2005	5	21,534
2006	4	25,093
2007	5	25,307
2008	5	31,405
2009	5	28,420
2010	4	30,654
2011	3	36,928
2012	5	52,768
2013	6	52,234
2014	5	50,402
2015	5	45,691
2016	6	59,074
2017	6	51,569
2018	5	53,914

yet refractory molecules. They are produced by the early diagenesis and decay of biomatter and form ubiquitously in the environment via processes involving chemical reactions. During decomposition and humification, plant material or other biomatter undergoes diverse modifications (Tan, 2014; Rashid, 1985). These modifications depend primarily on the nature, amount and types of organic matter. The number and nature of microbial populations and the physiochemical conditions associated with the humification (amount of oxygen, rate

of burial, effects of clay minerals) are believed to influence the formation of the humic substances (Susic 2001; Rashid, 1985).

The amount of oxygen in the system is important as it affects the rate of degradation of plant organics. Rashid (1985) notes that the chemical characteristics of humic compounds vary with the degree of oxidation. The humic acid contents tend to be higher in sediments where oxidation is occurring. The total acidity of the humic compounds is also noted to be considerably lower in anoxic sediments than in oxic sediments.

The higher quantities of humic and other organic compounds are more often found in sediments enriched in clay minerals, relative to those with poor clay content (Rashid, 1985). Clays appear to have a catalytic effect on the decomposition of organic matter and on the process of humification. The clays and other aluminosilicate minerals may also accelerate the variety of organic reactions and transformations that occur as part of the humification process. Humic substances can also attach themselves to clay minerals and can solubilize nearly 10 times their own molecular weight in clay particles (Susic, 2001). This can create some of the very large humic particles with very large apparent molecular weights.

According to Essington (2004), several mechanisms and two categories of pathways have been used to describe the genesis of humic substances: 1) those that are purely biological and involve enzymatic decomposition of biopolymers and the enzymatic recombination of the microbiological byproducts; and 2) those that involve the biotic decomposition of biopolymers and the abiotic assemblage of macromolecular structures or aggregates. Much of the understanding of the specific mechanisms and pathways for humic substance formation are theoretical (Billingham, 2012; Susic, 2001). It is likely that the formation of humic substances involves elements of both pathways in the formation of a given humate deposit.

Ultimately, the humic substances become exceedingly complex macromolecules and/or supramolecular mixtures held together by weak chemical bonding forces, hydrogen bonding, or both. Unfortunately, irrespective of the specific mechanism(s) of formation or structural characteristics, humate is usually studied in detail only after the humic substances have been isolated and purified chemically (Susic, 2001). The focus of the many studies of humic substances has been on their elemental composition, their functional groups, and their interactions with other ions and chemicals in the environment. Essington (2004) notes that humic substances are formed through random polymerization or aggregation of a diverse array of compounds from a pool comprised of the microbial degradates of biopolymers. Therefore, the probability of finding two humic molecules that are exactly alike is small, particularly among the larger humic molecules. He also notes that even though the humic substances are refractory (resistant to change), they can evolve and degrade, further enhancing their random character. The macrostructural and supramolecular characteristics of humic substances, especially *in situ*, remain poorly understood.

If humic substances are produced with early diagenesis of buried organic matter, the process of humate formation could begin shortly after (in geologic time), provided there is enough oxygen available. Diagenesis typically involves changes to the

buried sediments due to interactions with pore waters (connate and groundwater), minerals, organic material and gases as the sediments experience increases in temperature and pressure with burial. In the case of the Fruitland coals, another period of humate formation would have been expected when the coals were uplifted to the surface, reacted with oxygenated groundwater and were exposed to weathering processes.

HUMATE AND URANIUM

There is a natural association of uranium with humate in Cretaceous and Jurassic sedimentary rocks in the Grants Mining District in New Mexico (Leventhal, 1980; Turner-Peterson, 1985). Some of the humate ores consist of trace uranium minerals and dark humate in organic-rich sandstones (Fig. 3). The relationship between uranium and plant detritus as well as humic substances has been known on a worldwide scale (Leventhal, 1980). Humic substances are effective cation exchangers and are capable of concentrating uranium and other metals from dilute solutions. They appear to be important in the formation of the uranium deposits in the Grants mining district.

In addition to the interest in the relationship between humate and uranium in the formation of the deposits, there has been recent interest in their interrelationships relative to the fate and transport of uranium and other metals released from the uranium mine waste (Velasco et al., 2019). Recent work with uranium-bearing humic substances (described as natural organic material [NOM]) has been conducted to better understand the occurrence of and chemical reactions between the organic matter and the uranium. Organic matter and the associated humate strongly affect the redox, complexation and precipitation chemistry of uranium. Organic matter also appears to influence the mobility of the dissolved and particulate forms of uranium (+4) and uranium (+6) (Velasco et al., 2019). The unique characteristics of humic substances (i.e., their solubility



FIGURE 3. Uranium minerals with dark humate in Jurassic sandstone from Grants Mining District (1 cm cube for scale).

in water, large molecular size, surface areas and CEC) may be effective in mitigating uranium and other metals released from mine wastes. Additional work is needed to better understand these relationships.

CONCLUSIONS

The mining and production of humate and the processing to recover humic substances is expected to be a growth industry in New Mexico well into the future. This is largely due to demand for agricultural amendments to increase crop yield and improve soil conditions. Ongoing research for other uses of humate and humic substances is moving forward on many fronts, and there are likely to be other beneficial uses developed that will further increase demand for the humate resources in the marketplace.

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