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# Review Paper on Black Powder and Benite Igniter Compositions with Emphasis on Charcoal Performance

by JR Schmidt and RA Pesce-Rodriguez

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# **Review Paper on Black Powder and Benite Igniter Compositions with Emphasis on Charcoal Performance**

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<b>13. SUPPLEMENTARY NOTES</b>					
<b>14. ABSTRACT</b> This report offers a brief history (1950s to present) of black powder (BP), its constituents, formulation, and the functionality of each component, highlighting research from the US Army and Navy. Included is a 1) history of its manufacture and why BP is not just a mixture of its components; 2) review of what makes BP so unreliable (moisture absorbance, batch-to-batch variation, and packed bed limitations on flamespreading); 3) discussion of the development of nitrocellulose (NC) and its propensity to self-ignite (necessitating thoroughly washing NC and adding an alkaline material for stability); 4) review of the development of Benite, a so-called “non-hydrophilic” form of BP combining BP with NC; and 5) review of the advantages of Benite over BP, specifically, its extrudability (e.g., more uniform propellant bed ignition produces better performance); and review of short-term stability tests and improvement efforts. Recent investigations on substandard Benite lots suggest charcoal is the root cause of the performance variability. Analysis was done to determine the make-up of “good charcoal”. While no consensus was reached, several indicators were widely accepted. Possible replacements for charcoal were suggested, with the most promising being phenolphthalein, anthraflavic acid, and carbon black. Recent developments in BP substitutes are also discussed.					
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## **Contents**

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<b>1. Background/Introduction</b>	<b>1</b>
<b>2. Development of BP</b>	<b>1</b>
<b>3. Attempts at BP Replacements</b>	<b>5</b>
<b>4. Development of Guncotton, or Nitrocellulose</b>	<b>6</b>
<b>5. Development of Benite</b>	<b>7</b>
<b>6. Summary and Conclusion</b>	<b>8</b>
<b>7. References</b>	<b>10</b>
<b>List of Symbols, Abbreviations, and Acronyms</b>	<b>13</b>
<b>Distribution List</b>	<b>14</b>

## **1. Background/Introduction**

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Black powder (BP), the very first chemical explosive, has substantially changed the history of mankind: the conversion of chemical energy to do useful work in a way not available previously by the burning of wood or such materials.

The following offers a briefing history of BP:

- The ingredients of BP were first formulated as a compound in China between approximately 600 and 900 AD. At the time, the compound was intended for medicinal purposes. Many historians believe the first BP formulations were prepared in attempts to restore virility or as a “fountain of youth”.
- As such, the story is that after mixing the ingredients and then putting them in a broth in a hearth, some of the mixture fell into the flames and sparks started to fly. This is assumed to be the start of BP as the first chemical explosive.
- While some historians believe BP originated in the Middle East in 1300 AD, most attribute it to originating in China.
- Historians cite the first nonmedicinal use of this mixture in fireworks in ancient China. An alchemist would put BP-like (BPL) flakes into bamboo sticks to produce these first fireworks. The fireworks were brought into Europe in the 13th century AD and their popularity immediately “took off”.
- BPL mixtures were used in a number of types of weaponry, but this weaponry was often more show than lethal. The major problems with BPL formulations are the same as those for BP (i.e., that the mixture is highly hydrophilic and the mixing process is very intricate). As such, the chances of a misfire were always high. However, when they did ignite, there was a load bang, fire, and hot embers spewing out, which provided a very impressive event for those who were exposed to it.
- The development of weapons that use BP is not a subject of this report, but can be found elsewhere (McNab 2004; Willis 2017).

## **2. Development of BP**

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The BP formulation still in use today is largely accredited to the Catholic monk Roger Bacon, c. 1250. He was the first European to publish the composition now known as BP (Davis 1943). Some credit the German Franciscan monk Berthold der

Schwarz with the invention of BP in 1313 AD. Even if Schwarz did not invent BP per se, he is largely accredited with the invention of firearms.

The technique used in manufacturing BP and its precursors was of foremost importance to successfully employ it. Merely mixing the three ingredients of BP—charcoal, sulfur (S), and potassium nitrate ( $\text{KNO}_3$ )—into a bowl, even in the correct ratio, will very likely not result in a product with the desired ignitability and explosive performance. Instead, a rather laborious mixing process, done with grinding stones and added water, was necessary to produce the intricate amalgamate needed for the first chemical explosive. Also, the BP had to be dried after the blending and stored away from heat and humidity, as it was both explosive and hygroscopic. Of note here is that the grinding process itself was very dangerous; if the mixture became too dry or too hot, it would explode, potentially resulting in loss of life and property. To reiterate for emphasis, the amalgamate that we call BP is not just a simple mixture of the components but a unique material that requires a long and precise process of mixing to achieve the proper blending and consistency.

BP became essential to civilization, primarily for its use in cannons, guns, and mining, though BP was not used in mining at first due to its high cost and fear of mine shafts collapsing due to the force of the explosion. Of note is that the storming of the Bastille (July 14, 1789) occurred in part because the French government had stored all the BP they had in that location. The angry and aggressive mob sought not only to free those imprisoned in the fortress but to gain access to BP for their revolution.

The formulation of BP has long been considered more of an art than a science. Even after hundreds of years of manufacturing the amalgamate, there are still significant variations in batches produced by industry, even at the same manufacturing plant. Through careful investigation of these variations and occasional failures of BP batches, the most commonly defined root cause has been the charcoal used. Whereas  $\text{KNO}_3$  and S can be produced at a very high level of purity, charcoal is dependent the kind of plant matter from which it originated, what elements the plant had absorbed from the soil in which it grew, and the pyrolysis conditions under which the charcoal was made.

Nobel and Abel (1875) investigated the transport phenomenon that occurs when BP is used in a gun to propel projectiles. Finding that the results were not consistent with known gas laws and noticing that there was a high amount of solid residue from the combustion of BP, they developed what would be called the Nobel–Abel equation of state, a gas equation of state that takes condensed combustion products into account. Blackwood and Bowden (1952) performed a study on the initiation of

the burning of BP, and how it develops and grows to explosion. They discovered that the initiation is due to local hot spots at temperatures of approximately 130 °C. This temperature is sufficient to melt S (whose melting point is 115 °C).

White and Horst (1980) noted significant lot-to-lot variation for BP. They studied how these variations could be minimized using physical igniter design concepts.

Sasse (1981) showed definitively that BP made from the same charcoal lot could produce significantly different burning rates. Applied pressure during the manufacture of BP was observed to induce considerable plastic flow, resulting in a fused conglomerate with little internal free volume. The internal pore structure inherent in charcoal gets “packed” with the S and KNO<sub>3</sub> crystals as the amalgamate is formed. Correlations between burning rate and internal surface area, pore volume, and density were found to exist. Sasse suggested that the adverse effect of water on BP performance results from the occupation of the internal free volume of the amalgamate with water. The subsequent, attendant retardation of the burning rate is due to the necessity to evaporate that water from the internal free volume (or what is left of the pore structure). He hypothesized that an increase in the degree of openness of the BP grains increases the burning rate. The openness Sasse is discussing is the internal free volume. Sasse (1983a) confirms his hypothesis on the openness of BP grains and measured burn rates up to 100 atm.

Consistent with Sasse’s concept of pore volume, McMahon and Dillehay (1984) investigated the equilibrium moisture uptake for BP. They found that the moisture level of BP could be controlled by exposing the BP to a fixed temperature and humidity condition for a set period of time.

White et al. (1981) investigated BP in high- and low-pressure igniter systems. Two different lots of BP were used with igniter action time varying considerably depending on the lot. They found slow-burning lots form large amounts of liquid droplets at the BP surface, which then move into the flame above it, whereas fast lots burn with very little droplet formation. White et al. also found that slow-burning lots are poorly mixed, while the fast lots are well mixed. Particle size and degree of mixing appear to play into the combustion properties. White et al. tried their best to control all the variables in making BP, but were unable to pinpoint a cause of the fast and slow lots. They believed a hidden variable existed that was of first importance in combustion of BP. They suggested the hidden variable might be the “degree of openness” discussed by Sasse (1983a).

Looking deeper, White and Sasse (1982) studied flamespread in primer tubes and showed that grain break-up or deconsolidation may take place during vigorous ignition, thereby indicating the mechanical properties of BP will affect performance. Burning rates for BP were measured and published at several



pressures. They believed the mechanical properties of the BP are essential to understanding variation in BP performance. They pointed out that there is no evidence for combustion-induced deconsolidation under constant pressure burning. However, when BP ignition is subjected to strong mechanical loading and pressure transients, there is evidence of grain break-up.

Sasse (1983b) then determined the chemical composition and physical properties of maple charcoal used in the production of BP, noting different samples of charcoal from the same “lot” differed in composition. These variations were attributed to the individual properties of the wood employed and the temperature histories in different locations in the kiln. He suggested requiring pre-blending of charcoal lots before mixing as a way to improve the uniformity of BP performance.

Smetana and Gultz (1983) looked at the manufacturing of fertilizer-grade  $\text{KNO}_3$ , specifically, “VERTAC produces fertilizer-grade potassium nitrate by a chemical reaction between potassium chloride and nitric acid. The reaction yields potassium nitrate, which is contaminated with acidic byproducts and residual nitric acid. VERTAC sprays their product with a solution of potassium hydroxide to neutralize it. The resulting product has been found to vary in pH from acidic pH 4.4 to basic pH 10.4. The effects of this variation in pH in terms of reliability, safety, and storability are not known.”

Subsequently, Smetana and Gultz claim the pH of the  $\text{KNO}_3$  affects the hygroscopicity and corrosiveness of BP. They also indicated that this may affect the burning rate of BP. They specifically recommended neutral pH (specification-grade)  $\text{KNO}_3$  be used in BP production.

Sasse et al. (1984) later used closed-bomb techniques to determine quickness and burning rate for two commercial BPs and a BP made at Indiana Army Ammunition Plant (IAAP); they concluded the IAAP BP was the most extensively classified US BP lot in history. (Thereby implying that the conclusions they drew were accurate.) IAAP BP was made with non-neutral salts of  $\text{KNO}_3$ . They concluded because the results were in line with one of the two commercial BPs, the new BP was suitable for use in ammunition. Note this is contrary to Smetana and Gultz’s conclusion that neutral-grade  $\text{KNO}_3$  be used in BP. Sasse et al. were aware of this conclusion, but still claimed non-neutral salts of  $\text{KNO}_3$  were acceptable. (The authors of this report believe it prudent to ensure storage compatibility and corrosion effects before implementing non-neutral salts.)

Wise et al. (1984) studied the substitution of the charcoal in BP with a number of crystalline organic compounds, including polynuclear aromatics and polyphenols and some phthalic acid salts. The results were mixed, but they were most impressed

with mixtures containing phenolphthalein (PP), as it produced combustion rates parallel to that of BP. Other compounds that showed promise were noted.

Sasse (1985) reviewed BP and scrutinized the charcoal it contains based on modern techniques such as scanning electron microscopy (SEM). He again showed that the physical properties of the BP (internal surface area, pore volume, internal free volume, and density) were related to the burn rate and provided burn rate data. Sasse (1988) published Chapter 2 in *Gun Propulsion Technology*, where he covered in detail what was known at that time about BP.

Sasse and Rose (1988) also examined the particle-size distribution (PSD) of BP as spheres and then as ellipsoids. They then reevaluated closed-bomb data by including the actual PSD in numerical models that consider the grains first as spheres and then as ellipsoids. This yielded small but significant improvements to the closed-bomb data. However, when compared to strand burn rate measurements, the new model still varied by a large amount. Sasse and Rose concluded grain fracture (or grain break-up) dominated the closed-bomb evaluation technique, but noted that fracture did not occur in strand burner experiments.

McBratney et al. (1991) studied replacing the charcoal in BP used in handheld signal flares with four of the compounds suggested by Wise et al. They suggested that PP and anthraflavic acid (AfA) showed promise as a replacement for charcoal in BP used in handheld flares.

### **3. Attempts at BP Replacements**

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As noted previously, the components of BP are charcoal, S, and  $\text{KNO}_3$ . Charcoal is a fuel and should contain about 20%–50% pyrolyzed volatiles in addition to carbon (Rose 1975; Rose and Hardt 1979; Sasse 1983a, 1983b). Volatiles refers not to moisture, residual solvent, or other readily desorbable materials, but to pyrolysis products evolved when a material is heated to 955 °C in an inert atmosphere (Johnson 1928).

Pyrolyzed volatiles are crucial to the ignition of BP (Sasse 1983a). Unfortunately, wide variations are found in different lots of charcoal. As a consequence, the performance of BP varies excessively from lot to lot. Since the US government does not buy a large quantity of charcoal, it has no leverage in changing the way it is manufactured. Extensive work has been done in the past to try to characterize charcoal, but the definition of what makes “good charcoal” is not fully understood (Kirshenbaum 1977; Rose 1980; Sasse 1988). The attempts to codify the essential characteristics of charcoal were never fully settled and, therefore, could never be implemented.

Trying to improve BP without improving charcoal seemed futile. Efforts to improve BP moved from making a better “specification” for the purchase of charcoal to finding a replacement for charcoal with a chemical compound with reproducible characteristics that would work in the igniter mixtures. Extensive work was done on BP by Vest (1955), Sasse (1981), Rose (1975), Kirshenbaum (1977), and so on, using chemistry principles to suggest compounds that worked. Of the compounds examined, the most promising were PP from Wise et al. (1984); AfA from Wise et al. (1984) and McBratney et al. (1991); and carbon black from Kirshenbaum (1977).

The US Army Combat Capabilities Development Army Research Laboratory (and its predecessor, the US Army Ballistic Research Laboratory [BRL]) did extensive development work on black powder substitute (BPS) formulations containing substituted organic ring compounds in place of charcoal. The DEVCOM Armaments Center (previously known as the US Army Armament Research, Development, and Engineering Center) continued the development effort of BPS formulations, focusing on the most promising organic compound, PP. Red Powder, a BPS, was developed by Anton Weber (1995) at Picatinny Arsenal, New Jersey. The chemical composition is a dispersion of PP,  $\text{KNO}_3$ , potassium hydroxide, and S. Gary Chen and others (Chen and Sun 2004; Blau et al. 2008) developed and optimized a BP replacement, which they called the moisture-resistant black powder substitute (MRBPS) formula, in 2008. MRBPS does not use the problematic charcoal, replacing it with PP, and does not use S. MRBPS contains PP, potassium perchlorate, and  $\text{KNO}_3$  with other minor ingredients. It met or exceeded ballistic performance of BP in mortars, but has yet to be fielded.

#### **4. Development of Guncotton, or Nitrocellulose**

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In the early 19th century, a number of chemists tried to develop useful explosives (Davis 1943). Henri Braconnot discovered that nitric acid and wood fibers would yield a combustible explosive in 1832. Theophile-Jules Pelouze and, separately, Jean-Baptiste Dumas did the same using paper and cardboard instead of wood fibers in 1838.

Circa 1846, Christian Friedrich Schonbein serendipitously discovered guncotton, or NC. He was working in his home with a mixture of nitric and sulfuric acid, which he spilled on the kitchen table. He grabbed a nearby cotton apron and sponged up the mixture, after which he hung the apron up to dry on the stove door. However, once the mixture was dry, it burst into flames. Schonbein worked with Rudolf Christian Bottger who independently invented NC. Yet a third chemist, FJ Otto, also independently invented this process the same year. Otto was the first to publish.

The material was named guncotton because the finished product was white and fibrous, having an appearance of cotton fibers. Guncotton as a propellant was an improvement over BP as the impetus was significantly greater than BP; it produced less wear on the gun barrel due to a lower flame temperature; and it produced less smoke out of the barrel than BP. However, guncotton was less stable than BP. NC needs to be fully washed to remove any excess acid; otherwise, it could burst into flames. It was discovered that the addition of an alkaline material (usually calcium carbonate [CaCO<sub>3</sub>]) will help with stability (Davis 1943).

One issue with using BP as a propellant was smoke out of the barrel from the guns would often obscure the view of the person firing the rifle, or in the case of a battlefield, the entire view of the enemy after only a few rounds were fired. In 1884 Paul Vieille invented “smokeless” powder, the culmination of which was a double-based propellant that was a mixture of NC and nitroglycerin (NG). Note here the term smokeless powder is misleading, as there is smoke generated, but significantly less than that generated by BP. This is due to the fact that BP produces a significant amount of solid products after combustion, whereas smokeless powders produce mostly gaseous products. The use of double-based propellants soon dominated the ammunition industry. The double-based propellants were superior to BP in many ways (more impetus, less barrel wear, and less smoke), but they are not as easy to ignite; ergo, BP was often relegated to the ignition train of large-caliber ammunition.

## **5. Development of Benite**

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The igniters used in many of the Army’s large-caliber rounds (e.g., tank rounds), circa the 1950s, consisted of a metal primer body with a set of holes, which allowed for the burning igniter gases to be released into the propellant bed. These primer tubes were usually filled with packed beds of BP. The packed bed igniters were not conducive to the proper fluid flow and uniform ignition of the propellant bed. Douglas C Vest (1955) of BRL (now the DEVCOM Army Research Laboratory) first proposed the concept of an extrudable mixture of the ingredients of BP and NC. He suggested a 50/50 mixture of the ingredients, but admitted to having no time to optimize the mixture. Hassmann (1958a, 1958b), experimenting with a number of Vest’s igniter formulas, optimized the mixture and found that an extruded form of the unincorporated ingredients of BP (60%) with NC (40%) (plus added ethyl centralite [EC] stabilizer) produced the best composition of the extruded igniter and called the composition Benite. This composition provided a more uniform ignition (by using strands instead of a packed bed) and was first marketed as a “non-hydrophilic” form of BP. Benite is hydrophilic, but far less so than BP itself, hence the marketing.

As previously noted, the making of BP is more than just mixing the ingredients together. It requires intimate mixing of the ingredients and expert know-how. Some would categorize it as an art. Over the years, some have worried that the art of making BP is rapidly being lost (Vest 1955; Hassmann 1958b). Herein lies the big advantage of Benite over BP; the manufacture of Benite only requires the thorough mixing of the ingredients rather than expert know-how.

However, Benite has several known disadvantages, the major one being its stability. Benite is required to pass a short-term thermal stability test (MIL-STD-286C with Change 2, Method 404.1.2, test first suggested by Urbanski [1964]) before a new lot of Benite is accepted for use in military weapons. The specification states Benite “shall not change the color of normal methyl violet test paper to a salmon pink color in less than 35 minutes and shall not explode in less than 5 hours.” Benite tends to fail this test at an unacceptable rate. This is evidenced as early as 1961 in a paper by Huselton and Kaplowitz in which they attest, “Soon after Benite production was started, it was found that the 134.5 °C heat test requirement of 40 minutes...could not be consistently met...” This was a problem early on and continues to be a challenge to the present day.

Huselton and Kaplowitz (1961) showed that substituting diphenylamine (DPA) for EC improved the short-term heat (stability) test. Of singular note here is this 1961 paper touts this claim about DPA and its marked improvement over EC when used in Benite, and also claims that this switch would take place shortly as long as the tests continued to go favorably. However, in 2021, 60 years later, EC is still used, and Benite still has problems meeting the thermal stability test criteria. A search of published Benite papers from 1961 to the present provides no clue as to why DPA was not implemented. One can only speculate as to why DPA is not in use, perhaps cost or perhaps it failed another test. Of some minor interest is that Huselton and Kaplowitz also mention adding a boron powder to the surface improves the short-term stability test results.

## **6. Summary and Conclusion**

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An overview of the history of BP was given, going back to its origin in China. As true for many inventions, the invention of the very first chemical explosive appears to have been accidental. The explosive soon spread to Europe and the formulation now called BP was invented. BP became essential for both war and mining. Even in modern times, BP has lot-to-lot variations due mainly to variations in charcoal. Much research has centered around the idea of either being able to fully characterize charcoal to improve lot-to-lot variation or replacing the charcoal with another fuel. Unfortunately, researchers have never fully agreed on what in charcoal is necessary

to make a good BP. The study of alternate compounds tends to concentrate on PP, AfA, and carbon black.

The development of NC with NG (double-based propellants) was revolutionary in the development of propellants. The idea of combining NC with the components of BP (known as Benite) was also revolutionary, as it provided an extrudable form of an igniter. Unlike BP, the mixing of Benite ingredients is rather straightforward. However, the Benite does have stability problems, which require further research to find a solution.

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## List of Symbols, Abbreviations, and Acronyms

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AfA	anthraflavic acid
ARL	Army Research Laboratory
BP	black powder
BPL	black powder-like
BPS	black powder substitute
BRL	US Army Ballistic Research Laboratory
CaCO <sub>3</sub>	calcium carbonate
DEVCOM	US Army Combat Capabilities Development Command
DPA	diphenylamine
EC	ethyl centralite
IAAP	Indiana Army Ammunition Plant
KNO <sub>3</sub>	potassium nitrate
MRBPS	moisture resistant black powder substitute
NC	nitrocellulose
NG	nitroglycerin
PP	phenolphthalein
PSD	particle-size distribution
S	sulfur
SEM	scanning electron microscopy

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