Chapter 2: Organic Petrology

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* 1. INTRODUCTION TO ORGANIC PETROLOGY

Petrology is the science concerned with the origin, structure and composition of rocks, with petrography being the sub-branch which determines the description and classification of rocks by microscopic examination (http://www.dictionary.com/browse/petrography). Thus, coal and carbon petrography is the microscopic study of organic and incorganic constituents found in coal, carbon, and other rocks. It is also applied to study or assess origin and formation of coal, the thermal/burial histories of sedimentary basins and petroleum exploration, and the technical performance of coal in beneficiation, combustion, coke making for metallurgical processes, pyrolysis and char conversion, coal ash utilization, and related purposes.

Coal consists of organic and inorganic components, but no two coals are alike, even within distinct coal seams. Coal petrography provides the opportunity to assess the microscopic organic and inorganic constituents and the degree of metamorphism (or rank) to which the organic matter has been subjected subsequent to its time of burial. It is only under the petrographic microscope that the organic components of coal, termed macerals, can be quantified; each maceral makes an individual contribution to the overall chemistry and technological behaviour of a coal (ICCP, 2015). In addition, visual observation and quantification enables the assessment of the inter-relationship between coal macerals and the inorganic matter, via the assessment of the microlithotypes. Furthermore, the degree of weathering or abnormal features resulting in anomalous behaviour during beneficiation and or utilization can be quantified. In recent years, with the discovery of shale gas, organic petrology has been applied to determine thermal maturity of shale gas fields, with petrographic vitrinite reflectance determination playing a crucial role in shale gas assessments. A similar assessment is undertaken in petroleum exploration.

Thus, coal and carbon petrographic studies enable the following: (Francis, 1965; Schapiro and Gray, 1964; Taylor *et al.*, 1967; Brown *et al.*, 1971; Smith, 1972a, 1972b; Steyn and Smith, 1977; Zimmerman, 1979; Berry, 1981; Stach *et al.*, 1982; Bustin *et al.*, 1983; Falcon and Snyman, 1986; Scott, 1989; Bailey *et al.*, 1990; Gray, 1991; ICCP, 1993; Hower *et al.*, 1998; Taylor *et al.*, 1998; Wagner, 2007; Suárez-Ruiz and Crelling, 2008; Suárez-Ruiz *et al.*, 2012; O’Keefe *et al.*, 2013; ICCP, 2015; Hackley and Cardott, 2016)

1. the assessment of the physical, (inferred) chemical, and (predicted) technological properties of coal constituents which, combined, contribute to the properties of the seam as a whole;
2. diagenetic interpretations which may be derived from coal constituents (their types, forms, and associations) regarding the original conditions of coal formation and the subsequent history of the seams and basin;
3. the evaluation and assessment of coal as an integral part of analyses during the exploration, mining, beneficiation, marketing, and utilisation phases in the coal industry;
4. the consideration of weathering, oxidation, and other anomalous effects either ***in-*** or *ex-situ*;
5. the assessment of combustion, gasification, coking, and residue products to gain in-depth understanding of conversion processes; and
6. the thermal maturity assessment of sedimentary basins targeted for shale gas exploration via vitrinite reflectance analysis, in conjunction with other petrological techniques.

The following is an updated version of the original text produced by Falcon and Snyman (1986), reproduced with permission of the Geological Society of South Africa. The original figures have been redrawn and amended where required, and additional tables and figures have been added. The original text pertaining to applied petrography has been incorporated into Chapter 3 along with relevant figures.

* 1. HISTORICAL DEVELOPMENT OF COAL PETROGRAPHY
     1. Development of Techniques in Coal Petrology

The first and most significant introduction to coal petrology was published in 1913 by Thiessen and White who, in discussing the origin of coal, stressed the importance of microscopic coal petrology (Ward and Suarez-Ruiz, 2008). Transmitted light techniques were developed using thin sections and were applied successfully by many scientists both in Europe and the United States of America (USA) during the following decades; however the terminology and techniques developed are generally no longer utilised.

Investigations into the examination of coal by means of reflected light on the polished surfaces of specimens also began in 1913 in Germany, but this technique took a further 25 years to develop. The prime investigators in this field include Seyler, Stach and later, Mackowsky, M. Teichmüller, and Kűehlwein. Marked improvements in resolution and optical quality were achieved during this period, particularly after the use of oil immersion objectives and techniques had been introduced.

From 1940 onwards, quantitative rather than purely qualitative investigations began to develop. These took the form of the measurement of the percentage of light reflected from the surface of vitrinite particles, the results of which were correlated with the rank or maturity (coalification) of coal. Subsequently Teichmüller, Ottenjan and others developed the identification of rank even further by measuring fluorescence intensity using ultraviolet light reflections from the surfaces of liptinite particles (Taylor *et al.*, 1998). These methods have been correlated with the production of hydrocarbons from source rocks, and are extensively used in the exploration for oil and gas in many parts of the world (Taylor *et al.*, 1998; Hackley and Cardott, 2016).

Despite the fact that coal petrography had been used for decades in the assessment of coals for coke manufacture, it was not until the oil crisis in the early 1970’s that this technique began to come into far wider fields of use. At that time, many users of oil converted to coal-fired technology due to the high price of oil. In addition, the seaborne trade of coal expanded, and many industrialised countries began importing coal from other parts of the world in order to augment and extend their own reserves by obtaining cheaper coal. And, in the search for alternative fuels, more specialised uses of coal in the chemical and conversion fields were initiated.

Such trading patterns and international movement and utilisation of coal soon brought to light important quality variations and anomalous behaviour in coals from different regions, despite acceptable conventional chemical specifications. It was therefore established that specifications such as volatile matter and calorific value, which may be applicable to one coal, may not necessarily serve to characterise the technological performance of another coal from a different region or coalfield. These differences were attributed to variations in coalification age, vegetation and climate at time of origin, composition and rank; they are not always identifiable by means of the empirical results of chemical analyses alone. It was in these circumstances that coal petrography provided a general understanding of the problems involved, and the differences in coal types in particular, in areass where little or no understanding of these facts existed before (Plumstead, 1957, 1966; Kemezys and Taylor, 1964; Cook, 1975; Mackowsky, 1968, 1975; Chandra and Taylor, 1982,; Falcon and Snyman, 1986; Falcon, 1989; Taylor et al, 1998; Cairncross, 2001; Glasspool, 2003; Hower *et al.*, 2012), to name a few.

Table 2.1 provides a typical example where the chemical specifications of two coals from different hemispheres could be considered comparable, but the coal performance was not as anticipated. The petrographic composition clearly explains the reason for the different conversion behaviours. South African inertinite-rich coals can perform very well in technologies designed for their conversion profiles.

In 1975 “Stach’s Textbook of Coal Petrology” was produced, detailing all the approaches and techniques used in coal petrology at that time. This book was updated in 1982 to deal with the considerable advances in the science in the latter part of the 1970’s. In the 1980’s and 1990’s, the applications of coal petrology expanded to organic petrology, with an emphasis on carbonaceous rocks, especially those considered to be petroleum source rocks and oil shales, as discussed in detail by Taylor *et al.* (1998) in the book entitled “Organic Petrology”. The ICCP Handbooks, produced and regularly updated by the ICCP (ICCP 1957, 1963, 1975, 1993, 2015), also provided valuable guidance in the field of coal petrography, and one can track the historical development of organic petrology through these documents.

Table 2.1: Coal petrography can provide data not apparent from the bulk chemical analyses.

|  |  |  |
| --- | --- | --- |
|  | POLISH COAL | SOUTH AFRICAN COAL |
| Proximate Analyses (%) |  |  |
| Volatile matter | 31.3 | 25.1 |
| Ash | 14.3 | 14.8 |
| Fixed carbon | 54.4 | 60.1 |
| Calorific value (gross) (MJ/kg) | 28.77 | 28.02 |
| Performance in same 600MW pf boiler | Heat in lower furnace | Heat in upper furnace |
| Petrographic analyses (vol%, mmf) |  |  |
| Vitrinite | 65.7 | 30.3 |
| Liptinite | 6.7 | 6.0 |
| Inertinite | 27.6 | 64.0 |

The measurement of vitrinite reflectance to determine coal rank and the use of fluorescence microscopy to examine liptinite macerals further expanded the applications of coal petrography. The advances in computer programs, automated point-counting systems, automated stages, and more recently, digital cameras, have all aided in continuously moving the science forward. All new petrographic microscopes these days are controlled by advanced software for reflectance determination, digital imaging, and even point counting for maceral quantification. Microscope manufacturers are able to produce high quality objective lenses suitable for air and oil immersion examination of coal mounts, up to a magnification of 1000 times.

Falcon and Synman[Snyman?] (1986) commented that further development in the field of automatic analysis of coal rank (vitrinite reflectance) would be necessary before fully automated analyses would become accepted on a world-wide basis. However, thirty years later, whilst automatic vitrinite reflectance determination is certainly possible, it is still not a globally accepted technique; the ICCP do not include automated systems in their vitrinite reflectance determination accreditation program, for example. Despite this fact, many companies do rely on automated vitrinite reflectance determination where quality control and large throughput of samples is required. In this instance, the software is calibrated to notify the operator of any sample not fitting into the expected range. In order to gain further acceptability of the automated technique, the ICCP had a working group assessing the automation of vitrinite reflectance (<http://www.iccop.org/workinggroup/automation/>), and determined that the technique was viable and comparable to results generated by non-automated techniques.

The latest developments in advanced software and digital cameras have re-energised the science and enhanced organic petrology. Today, coal and organic petrography remains a popular science globally, with most organic petrologists / coal petrographers associated with the ICCP or The Society for Organic Petrology (TSOP). The ICCP accreditation program, open to participation by members and non-members, has accredited over 500 petrographers globally since inception; to retain the accreditation, a petrographer requires re-accreditation bi-annually. Petrography is no longer confined to coals, but is used to assess sediments for thermal maturity, pyrolysis, char and gasification products, as well as residual carbon in ash, and environmental samples, amongst others. Professional societies such as the ICCP and TSOP ensure that the technique remains a well-recognised means of assessing solid fossil fuels and products globally, by way of regular round-robin exercises, working group participation, accreditation programmes, and annual society meetings.

* + 1. Development of Petrographic Nomenclature and Classification

Various forms of nomenclature for the constituents of coal began to evolve during the 1920’s when research workers studied the microscopic constituents of coal from extremely thin sections viewed in transmitted light. Organic components were recognised by their colours and shapes. The research, conducted by Thiessen and others, was thus able to determine the basic entities of coal and assisted in establishing, with greater confidence than had been possible before, the botanical origin of the components of coal (Stopes, 1919 as discussed by Taylor *et al.*, 1998; Ward and Suárez-Ruiz, 2008).

In 1935, Marie Stopes and Clarence Seyler developed a technique by which the bright gelified constituents of coal were chemically dissolved. The more chemically resistant microscopic constituents of coal which remained and were isolated or separated out in this manner, were recognised to be inert woody components and highly resistant spores, pollens, cuticles, and algae. This process, known as maceration (from the Latin word macerare = to soften), thus gave rise to the term maceral which, by analogy with the minerals in rocks, refers to the microscopically recognisable and discrete organic constituents in coal. Four types of constituents were recognised at this stage, namely: vitrinite, exinite (liptinite), micrinite, and fusinite.

In conjunction with the microscopic investigations a number of macroscopic coal rock types, or lithotypes, were recognised. These were termed vitrain, clarain, durain, and fusain.

Vitrain: bright coal; vitreous to sub-vitreous lustre with conchoidal fracture

Clarain: banded bright coal; thinly bedded bright coal with dull bands and even fracture

Durain: dull coal; matt lustre and uneven fracture

Fusain: dull coal with satin lustre; relatively friable.

With the growing importance of coal petrography, specifically in the steel industry, new challenges were encountered with the systems of nomenclature and the interpretations of the performance of coal constituents, and many systems of petrographic nomenclature evolved geographically. As a result, the need for a uniform international coal petrographic nomenclature was recognised, and in 1953 the newly instituted ICCP accepted this mandate as its first task (ICCP, 1957).

With a few notable exceptions, little attention had been paid at that stage to the highly variable bituminous coals and anthracites of Permian age in the continents of the Southern Hemisphere. The International Handbook of Coal Petrology (1963, 1975) was compiled predominantly based on the banded bituminous coals of the Carboniferous and the brown and subbituminous coals of the Mesozoic and Tertiary age found in Europe and North America

Thus, although many of the definitions and descriptions were found to be generally acceptable, it was established that the southern hemisphere Gondwana coals possessed a number of intermediate forms of organic constituents which did not fall easily into the accepted forms of terminology devised for the coals typical of the northern hemisphere (Snyman and Botha, 1993; Taylor *et al.*, 1998). Snyman (1961) coined the term semivitrinite to designate a maceral resembling semifusinite in terms of structure but behaving more like vitrinite on conversion; this maceral is now referred to as reactive semifusinite. Taylor *et al.* (1998) provides further discussion about the differences between Permian Gondwana coals and northern hemisphere coals.

Owing to the large number of different terms and synonyms based on a variety of methods of investigation, and the restricted and localised use of many of them, the ICCP committee adopted a restricted number of terms which were recognised by the majority of experienced petrographers globally. This became known as the Stopes-Heerlen System of Nomenclature (ICCP, 1963). The technical terms and maceral names used for brown coals (lignites) were separated from those used for hard black coals (bituminous coals and anthracites). The results were published in an extensive “International Handbook of Coal Petrology”, which has been expanded by supplementary editions (1963, 1975, 1993). More detail is given in Stach’s Textbook on Coal Petrology (1982 Edition), Bustin *et al.* (1983), Taylor *et al.* (1998), and Suárez-Ruiz and Crelling (2008). The ICCP are currently updating the Handbook, and sections have been published in reputable journals (ICCP 1998, 2001; Sýkorová *et al.*, 2005; Pickel *et al.*, 2017).

A major challenge lies in establishing the purpose for which the classifications should be used, viz. whether the classifications should be based upon the genetic origins and morphological forms of the components as used for scientific and geological purposes, or on the technological properties of the components for utilisation purposes. Inevitably. coal petrographers began amending the original descriptions and nomenclature to suit their respective applications or research needs, thus causing further proliferation in terminology and considerable difficulty in the standardisation of results between different laboratories (Snyman *et al.*, 1984).

For example:

* In the United States of America, the “Thiessen Bureau of Mines System” was based upon the thin-section transmitted light microscopic study of coal, whilst the “Spackman System” was based upon the property of reflectance of light from the polished surface of coal (Spackman, 2000). Using this approach, Spackman (1958), Spackman and Thompson (1964), and others, based their concept of a maceral on the idea that each constituent was a substance which possessed different properties at different ranks. For example, vitrinitic precursors in lignite (huminite) are known to have quite different physical and chemical properties from vitrinite occurring in bituminous coals and anthracites. The above authors, therefore, proposed that such differences should be reflected by means of the petrographic terminology.
* As a variation on the above theme, other researchers such as Schapiro and Gray (1960a, 1960b) and Schapiro *et al.* (1961), developed a classification for macerals based upon reflectance values (in oil) in relation to the coking characteristics of each maceral. Such methods differentiated between those macerals which reacted and changed on heating in the carbonisation process and those that remained inert. The term “entity” was proposed (synonymous with “maceral”). “Macerals”, which end in -inite (e.g. vitrinite) according to the ICCP nomenclature, were spelt with an –oid (e.g. vitrinoid) and maceral suites were spelt with the suffix –ite.
* In contrast to the concepts proposed by the USA, Timofeev *et al.* (1962) in the USSR devised the IGM classification system (taken from the Institute of Geology of the Academy of Sciences of the USSR, Moscow), based upon the diagenetic origin of the macerals (phyterals) and the composition of the plants which gave rise to coal formation. The micro-components are subdivided on the basis of two primary developmental trends, i.e.:
  + classes and sub-classes – these are characterised by differences in the substance of the micro-components. This refers to the process of increasing gelification mainly of cellulose, lignin, and similar vegetation as a result of prolonged decomposition under conditions which were initially oxidising and later reducing. The end points are vitrinitic (telinite and collinite); and
  + type and species – these are distinguished by the degree of structural preservation and by the amount of degradation of the plant material during its transformation. This sequence of decomposition progresses under weakly to strongly oxidising conditions and is characterised by the preservation of cell structure and increased reflectance due to high carbon-enrichment. The process is known as fusinitisation and has as its end members semifusinite and fusinite, i.e. inertinite.

In general, these two processes can indicate the genesis (origin and formation) of coal – providing drifting and re-deposition of the original components did not occur. The latter may be recognised by the presence of high mineral matter content and mechanical erosion. In cases where macerals with mixed properties arise, for example “fusinized” telinite, outside influences or secondary processes are thought to have superimposed their effects on the primary genetic trends.

Apart from these specialised systems of classification of the constituents of coal, several other countries have produced their own independent standards formalising the methodology, definitions and nomenclature to be adopted in petrographic procedure.

In the 1990’s (and subsequently updated in the 2000’s) a series of international standards were prepared by the International Standards Organisation (ISO), largely based upon the recommendations of the ICCP. These standards have formalised the international acceptance of certain petrographic techniques and nomenclature. The new system of subdivision, referred to as the ICCP System 1994 (ICCP, 1998) differs from that of the Stopes-Heerlen System (and replaces it), with the introduction of sub-groups and changes in nomenclature. The South African Bureau of Standards (SABS) has adopted these ISO standards in their entirety (as South African National Standards (SANS), as have many other countries. The ICCP classification can be explicitly used with any classification, be it Chinese, Australian, Indian, or South African (pers.comm Walter Pickel, 2017).

1. ISO 11760 (2005). Classification of Coals. International Organisation for Standardisation – ISO, Geneva, P.A., [www.iso.org](http://www.iso.org/).
2. ISO 7404-1 (2016). **Methods for the Petrographic Analysis of Coal - Part 1: Vocabulary.** ISO, Geneva, P.A., [www.iso.org](http://www.iso.org/).
3. ISO 7404-2 (2009). **Methods for the Petrographic Analysis of Coal - Part 2: Preparation of Coal Samples.** ISO, Geneva, P.A., [www.iso.org](http://www.iso.org/).
4. ISO 7404-4 (2009). **Methods for the Petrographic Analysis of Coal - Part 3: Method of Determining Maceral Group Composition.** ISO, Geneva, P.A., [www.iso.org](http://www.iso.org/).
5. ISO 7404-3 (2017). **Methods for the Petrographic Analysis of Coal - Part 4: Method of Determining Microlithotype, Carbominerite and Minerite composition.** ISO, Geneva, P.A., [www.iso.org](http://www.iso.org/).
6. ISO 7404-5 (2009). **Methods for the Petrographic Analysis of Coal - Part 5: Method of Determining Microscopically the Reflectance of Vitrinite.** ISO, Geneva, P.A., [www.iso.org](http://www.iso.org/).

The ASTM International (American Society for Testing and Materials) ([www.astm.org](http://www.astm.org/)) also produced a set of standards pertaining specifically to aspects of organic petrography, namely:

1. ASTM D121-15 (2015). Standard Terminology of Coal and Coke, ASTM International, West Conshohocken, PA.
2. ASTM D2797 / D2797M-11a (2011). Standard Practice for Preparing Coal Samples for Microscopical Analysis by Reflected Light, ASTM International, West Conshohocken, PA.
3. ASTM D2798-11a (2011). Standard Test Method for Microscopical Determination of the Vitrinite Reflectance of Coal, ASTM International, West Conshohocken, PA.
4. ASTM D2799-13 (2013). Standard Test Method for Microscopical Determination of the Maceral Composition of Coal, ASTM International, West Conshohocken, PA.
5. ASTM D388-17 (2017). Standard Classification of Coals by Rank, ASTM International, West Conshohocken, PA.

The current reference source (this Atlas[which Atlas?]) follows the ICCP nomenclature, which reflects the ISO standards for coal petrography, and may differ slightly from the ASTM and terminology used by TSOP (The Society of Organic Petrology). As the SABS has adopted the ISO terminology, standards SANS ISO 7404 parts 1 – 3, and 5 apply; part 4 is currently being updated by ISO. The reader is also referred to the ICCP publications of 1998, 2001, and 2017 dealing specifically with vitrinite, inertinite, and liptinite classification respectively (ICCP 1998, 2001, 2017).

In summary, the term maceral was originally introduced to distinguish organic from inorganic constituents in coal (Stopes, 1935; Stach *et al.*, 1982). Subsequently, however, following the ICCP, it has been used to define a unit of specific morphology (as well as size, shape, botanical affinity, and occurrence) irrespective of the rank of the coal (vitrinite, therefore, can be recognised in bituminous coal and in anthracite). For this reason, the nomenclature published by the ICCP and adopted by ISO has been adhered to in this document. However, because the southern African constituents display a wider range, for example, of reflectance (but similar morphological forms and structure), slightly broader definitions have been introduced herein in order to accommodate these macerals into the presently accepted standard terms and descriptions in southern Africa. The terms 'reactive semifusinite' and 'reactive inertodetrinite' are applicable, as these macerals have been shown to behave in a significantly different manner to the more inert semifusinite and inertodetrinite particles (Cook, 1975, Falcon and Snyman, 1986; Hagelskamp and Snyman, 1988; Diessel, 1992). Hower *et al.* (2012) concluded that not all Gondwana maceral types necessarily fit the ICCP definitions, which may result in a requirement for wider definitions of existing maceral types.

It must be emphasised, as coal is a complex rock, that organic petrology is a broader subject than coal petrography, with the former considering the organic components, inorganic and organic geochemistry, as well as coal rank (degree of maturity) (Ward and Suárez-Ruiz, 2008). All these characteristics are vital to obtain a holistic understanding of coal origin and formation, as well as to predict behaviour during utilisation.

* 1. PETROGRAPHIC CONSTITUENTS OF COAL

Prior to the detailed discussions on the petrographic aspects of coal, it is necessary to put into context the basic concepts regarding the composition and formation of coal. Coal is fundamentally composed of the fossilised remains of plant debris (phyterals) which have undergone progressive physical and chemical alteration through geological time. Peat (partially decomposed plant material) accumulated in mires, and is classified as limnic or paralic. Limnic coal deposits are inferred to have resulted from mires accumulating in slowly subsiding basins or where the mire was built up, isolated from marine conditions (i.e. more fresh water environments); paralic coal deposits formed in environments where there existed a hydrological connection with the sea at the time of peat formation (Hower *et al.*, 1998). Any form of organic matter may be preserved in some way in the peat, such as trees, shrubs, grasses, ferns, spores, pollens, aquatic plants, microorganisms, and the like, as long as suitable preservation conditions prevailed. Gondwana Permian coals are dominated by glossopterids, stunted deciduous broad-leaved trees or shrubs (Plumstead 1957, 1966; Falcon 1986b). These differed from the vegetation prevailing at the time of peat formation in the Carboniferous period of the northern hemisphere. The final coal composition was dependent on conditions in the peat mire which included plant type, nutrient availability, climatic conditions, and level of the water table, pH and Eh conditions (Falcon, 1986b). Taylor *et al.* (1998) concluded that the inertinite-rich nature of Gondwana coals is most likely a result of climate, where freeze-drying caused more oxygen to become bound to the organic structure, pre-conditioning mires to be inertinitic; in milder conditions, vitrinite may have formed.

Coals may be described or characterised at various levels of resolution, commencing on a macroscopic scale (lithotypes), progressing to the microscopic scale (Fig. 2.1).

Figure 2.1: The relationship between the macro- and micro-composition of coal (modified from Falcon and Snyman, 1986).

xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx

* + 1. Macroscopic Constituents of Coal

In hand specimen, successions of macroscopically identifiable layers of 5 mm or more are called lithotypes, which represent changes in organic input and conditions in mires. These layers, or bands, may be distinguished from one another on the basis of lustre (bright versus dull) and fracture pattern (irregular versus smooth), colour of the coal and colours of the streak produced by the coal, and finally texture and type of stratification. Lithotypes may be divided into non-banded / sapropelic coals, and banded / humic coals (O’Keefe *et al.*, 2013; Thomas, 2013).

* + - 1. Non-banded / Sapropelic Coals

Sapropelic coal lithotypes are non-banded, dull and compact, with an even granular surface and conchoidal fracture. They are considered to have their origins in open or deeper water and are formed from the accumulation of non-woody materials, such as algae and fine wind- or water-borne detrital organic matter (spores, pollen), and usually contain finely distributed mineral matter. The term allochthonous applies here, meaning redeposited, resistant plant fragments. Sapropelic coals, typically rich in hydrogen, include such categories such as cannel, boghead, torbanite, and various forms of hydro-carbon-yielding oil shales.

1. Cannel coals are characteristically black in colour and dull, sometimes with a greasy lustre. They are rich in spores and pollen and fine detrital organic matter (inertinite) and possess a uniform texture with fine microstratification. Vitrinite bands are commonly present. Cannel coals are typically formed in broad, shallow lakes surrounded by vegetation. Thus, with changing water level, these coals may grade laterally and vertically into humic coals.
2. Boghead coals are similar to cannel coals in outward appearance, but they possess a slightly browner colour and streak and their major constituent is algae. Torbanite is a frequently used synonym derived from a boghead coal found at Torbane Hill in Scotland. Boghead coals are typically formed at the centres of large basins into which transportation of detrital organic and inorganic matter was restricted, or in small basins in the swamps where algae flourished as surface colonies. Contact between boghead and humic coals is usually sharp.
3. Oil Shales are an extension of cannel and boghead coals. They possess a much higher proportion of mineral matter finely dispersed in the organic matter, thereby imparting to this coal lithotype a much higher density. Oil shale deposits form in areas marginal to the peat swamps, viz. on the flow of estuaries and lagoons, in shallow, open water with stagnant bottom conditions.

In terms of occurrence in southern Africa, torbanites are present and are of economic significance in certain seams.

* + - 1. Banded / Humic Coals

Humic coal lithotypes are banded with alternating fine layers of varying brightness, and typically form from wood and/or reed/sedge remains (O’Keefe *et al.*, 2013). They represent the accumulation of abundant humic matter within the original peat swamp or in very close proximity, thus being autochthonous. The lithotypes, or visually observed macrolayers, within this category formed in response to different conditions of plant growth and biochemical environment which existed for restricted periods during the development of the peat swamp. Figure 2.2 summarises the conditions prevailing in the major facies typical of coal-forming swamps. Figure 2.3 illustrates the development of a peat swamp with changing facies conditions.

Figure. 2.2: The major coal-forming facies and their characteristics in a typical Gondwana peat swamp (after Diessel, 1965, modified from Falcon and Snyman, 1986)

Stopes (1935) originally proposed four humic coal lithotypes: vitrain, clarain, durain, and fusain. These categories have been amended to encompass two more forms, namely duroclarain and clarodurain (Diessel, 1965, 1992) in order to characterise more efficiently the thinly bedded laminated coals typical of the Gondwana continents. These may be summarised as follows:

1. Bright Coal (Vitrain) – vitreous to sub-vitreous lustre and even to conchoidal fracture. May contain up to 10% dull coal, and is fairly brittle. This lithotype forms the brilliantly shining bands in humic coals, which break either with smooth surfaces or in small cubic blocks. Microscopically this band represents a layer of gelified woody material in which the cell contents and cell walls have reached a high degree of optical homogeneity.
2. Banded Bright Coal (Clarain) – thinly bedded bright coal containing thin (all less than 5mm thick) dull bands ranging in proportion between 10% and 40%.; the fracture is even. This lithotype has the lustre of shiny black satin with small hair-like bright streaks parallel to the bedding. Microscopically this band is dominated by narrow lenses and layers of gelified woody fragments with numerous leaf cuticles, spores, pollen, and droplets of resin distributed between them.
3. Banded Coal (Duroclarain) – dull and bright bands (all less than 5 mm thick) in approximately equal proportions (i.e. dull and bright bands representing 40-60% each). This lithoband is transitional in its characteristics and composition between clarain and durain, with the clarain characteristically slightly more dominant.
4. Banded Dull Coal (Clarodurain) – mainly dull coal with less than 40% bands of bright coal (all bands less than 5 mm thick); the fracture is uneven. This lithoband is transitional between clarain and durain, but in this case the durainous characteristics are marginally dominant.
5. Dull Coal (Durain) – matte lustre and tough with uneven fracture. Bright coal in thin bands up to 5 mm thick and comprising not more than 5%. This lithotype forms the bands of dull, sometimes granular, lustreless coal. It is the product of abundant oxidised particles or organic matter including partly decomposed roots, twigs and branches, leaves, and organic detritus with smaller proportions of leaf cuticles, spores, and pollen. Microscopically these bands appear as highly heterogeneous layers.
6. Fibrous Coal (Fusain) – dull coal with a satin lustre and it is relatively friable. Other bands comprising up to 5% are permitted. This lithotype is characterised by layers of highly oxidised, charcoal-like woody material which, when exposed on bedding planes, breaks with flat fibrous rectangular patches. The surface is soft and friable and soils the fingers easily. This represents trunks of wood which have been subjected to fire or to surface exposure and oxidation for extended period of time prior to submergence.

When more than 20% inorganic constituents (minerals) are present, the following lithotypes are used:

1. Shaly Coal – an intimate mixture of coal and between 20 to 60% clay minerals, resulting in a dull appearance with a brown streak. Alternating laminae (all >5mm thick) of coal and 20-60% shale are included.
2. Coaly Shale – a shale containing less than 40% thin coal bands or more than 60% shale.
3. Carbonaceous Shale – a shale containing less than 40% disseminated carbonaceous matter and no discrete coal bands.
4. Shale, mudstone, and so on – any sediment containing less than 10% carbonaceous matter.

Figure 2.3: The development pf a peat swamp with changing facies conditions (for example, rise in water levels) (modified from Falcon and Snyman, 1986)

In terms of global distribution most coals formed under terrestrial conditions, and are banded. These are therefore termed humic, including the majority of coals from southern Africa.

As the petrographic assessment of coal is based upon a fundamental understanding of the basic microscopic unit, i.e. the maceral composition of coal, this then becomes the foundation for the microscopic evaluation of coal. Macerals are therefore the most important target for the standardisation of petrographic terminology, nomenclature, and interpretation in order to adequately address the coal value chain (origin and formation to coal utilisation).

* + 1. Microscopic Constituents of Coal

Microscopically, coal is composed of a number of discrete organic constituents termed macerals. These are analogous to their inorganic counterparts, minerals, but differ in that macerals consist of mixtures of organic compounds without an orderly internal structure. Both the organic and inorganic components can range in size from 1 to over 200 µm (or more, if they occur in thicker homogenous bands). Maceral and mineral associations are termed microlithotypes. Coal type (maceral composition), rank (degree of coalification, and grade (degree of mineral inclusions) are all assessed microscopically.

* + - 1. Maceral formation (type)

Macerals are entities which evolved from different organs or tissues of the original plant material during primary accumulation to the early stages of biochemical degradation and early coalification (maturation). However, due to variable and often severe alteration, it is not always possible to recognise the plant material from which the macerals were originally derived.

The formation of the macerals during the early stage of peat accumulation is dependent on a number of factors including type of plant community, tectonic and climatic controls, ecological conditions, acidity (pH), and redox of Eh value. The latter plays one of the most important roles in the degradation of plant matter. For example, in aerobic conditions with unlimited access of oxygen, virtually complete disintegration of plant matter takes place and very little residue remains. In limited air (anaerobic conditions), however, such as under cover of leaf mould, mouldering occurs. Here the aerobic bacteria and fungi produce hydrogen-poor humic substances, the cellular contents and sap in the plant tissues dissipate, the cell walls dry out and darken and the vegetable matter is reduced to detrital grains and charcoal-like skeletal bands of wood. This process, if taken to its ultimate conclusion, is known as fusinitization, and results in the formation of semifusinite and / or fusinite (Fig. 2.4).

If a peat is partially submerged, oxygen supply is restricted and peatification proceeds with the production of humic acids and partial or full gelification of the woody plant tissue. This is the most important phase in biochemical degradation for coal formation and includes the progressive biochemical degradation by fungi and bacteria of, initially, the protoplasm and enzyme substances, then the storage of foods (such as sugars and starches),and ultimately lignin and cellulose which comprise the cell walls. The result of progressive peatification is the:

(i) gradual enrichment of the woody cell wall matter,

(ii) the preservation of the more resistant parts of plants, such as cuticles, spore or pollen walls and bark, and

(iii) the production of a gel, which is a precipitate of the humic acids in solution.

The successive processes of humification, gelification, and vitrinization converts the vegetable tissues into huminite and later vitrinite (Stach *et al.*, 1982).

Figure 2.4: Multiple and convoluted pathways to form coal macerals. (Modified after Hower *et al.*, 2013)

In cases where peats are subjected to reducing conditions, putrefaction or fermentation occurs. Here anaerobic bacteria consume the oxygen of organic substances, transforming them into hydrogen-rich products. For example, the fermentation of cellulose leads to the production of hydrogen, methane, acetic acid, carbon dioxide, and butyric acid. The coals which form from these processes are generally rich in extractable oils and nitrogen. Thus, macerals can form from a number of different pathways, as explained by Hower *et al.* (2013) (Fig. 2.4) and O’Keefe *et al.*, (2013).

Kerogen forms after thousands of years from the decaying organic matter, which, with further burial and temperature increases, breaks down into shorter chain carbon molecules (Arnold, 2013). Once the accumulating plant matter is buried at depths below 1 m, processes of “biochemical degradation” cease to function and the chemico-physical changes of “geochemical coalification” or maturation take over. As per ISO (2005), once the inherent moisture content is >75%, the compressed, buried peat is generally referred to as coal.

* + - 1. Coalification (rank)

Coalification is the process of metamorphosis which takes place with time and under conditions of increasing temperature and pressure, and results in the transformation of the original peat swamp through the progressive stages of brown coal (lignite), subbituminous, and bituminous coals to anthracites and meta-anthracites. The level to which a coal has reached in this coalification series is termed its rank. The proportions of the organic constituents which are formed during biochemical degradation at the peat stage (and which impart to coal its organic matter composition or type) and the process of maturation or metamorphosis (or rank of coal) are therefore independent of one another (Fig. 2.5). With increasing rank, the differences in the chemical and physical properties of the macerals diminishes, reflected by a convergence of reflectance values.

Figure 2.5: The formation of coal in terms of rank (maturity), type (organic composition) and grade (mineral matter composition) (modified from Falcon and Snyman, 1986)

Most macerals, once formed, undergo progressive physical, chemical,l and optical changes with increasing rank, but their proportions and shape, with minor exceptions, remain the same. In general, therefore, macerals may be distinguished from one another on the basis of morphology, relief, size, shape, colour, reflectance, and, sometimes, origin. Their names are descriptive and conventionally end in –inite (i.e.: vitrinite, inertinite, liptinite). Macerals may be grouped together into maceral groups or subgroups which are detailed and descriptive in nature. Maceral subgroups are distinguished on the basis of physical structure and refer to differences (such as preservation) in their materials of origin, e.g. charcoal or degraded wood. Maceral varieties refer particularly to the plant organs or tissues from which the coal has been derived, as well as the preservation conditions.

The distinction of the maceral group is based primarily on reflectance, but the names are derived from morphologic and genetic considerations and, to some extent, on their technological characteristics. Each maceral has a distinct set of physical, chemical, and technological properties for a given rank. The reader is recommended to consult the following publications for more detail pertaining to coal macerals: Stach *et al.*, 1982; ISO 7404-1, 1994; Taylor *et al.*, 1998; ICCP, 2001 (inertinite classification); ICCP, 1998 (vitrinite classification); O’Keefe *et al.* 2013; Pickel *et al.*, 2017 (liptinite classification). Descriptions of the macerals are also located opposite each plate in this Atlas, illustrating the maceral in question.

* + - 1. Maceral Groups

The three maceral groups, vitrinite, liptinite, and inertinite, form the basis of coal petrography. Each group includes a series of macerals which can be regarded as belonging together, either because of similar origin (such as liptinite) or because of the mode of degradation and preservation (vitrinite and inertinite). In Figure 2.6, the maceral groups have been conventionally separated into “reactive” and “unreactive” categories on the basis of their change in form and structure, relative to one another when subject to heat. These, and other major characteristics of the three maceral groups, are summarised in Tables 2.2 to 2.4.

Figure 2.6: A summary of the microscopic organic components of coal – maceral groups and macerals. (modified from Falcon and Snyman, 1986)

As the majority of coals mined, traded, and converted globally are medium-rank C bituminous coals, and with more than 98% of South African coals falling into this rank category (Hancox and Götz, 2014), the main thrust of the next section deals with macerals of bituminous coals. Low-rank C and B coals do not occur in southern Africa, and subbituminous coals are rare.

Huminite is the term given to the (equivalent) vitrinite maceral group in low rank coals; liptinite and inertinite macerals present in the low rank coals have the same terminology as bituminous coals. A suitable reference source for enhanced discussion on huminite and low rank coals are Stach *et al.* (1982); Taylor *et al.* (1998); Sýkorová *et al.* (2005). By definition, huminite is the maceral group present in lignite and immature sedimentary rocks with reflectance values intermediate to those of darker liptinite and brighter inertinite (ASTM, 2014).

Vitrinite Maceral Group

Vitrinite is a group of microscopically recognisable constituents which formed from the cellulose and lignin of cell wall material, the cell fillings of the woody tissue of plants (trunks, branches, twigs, roots, and leaf tissue), and colloidal humic gels. The structure of vitrinite may be difficult to determine due to the extreme processes of gelification and vitrinization. For preservation as vitrinite, vegetation must fall into the peat swamp, or be rapidly covered by water or sediments to prevent severe biochemical alteration by oxidation, or be parts of roots penetrating peat. Huminites are identified in low rank coals, and are considered to be the precursors of vitrinite macerals. Gelification, resulting in a “glassy” appearance (vitrum), is believed to take place in anaerobic or relatively reducing conditions. Vitrinite occurs more frequently in Carboniferous seams of the northern hemisphere (60-80 vol. %) than in the Gondwana coals (sometimes <20 vol. %) or in Cretaceous coals of North America (ICCP, 1998). In sediments, vitrinite is the main component of kerogen Type III.

Depending on coal rank, vitrinite appears dark to light grey under the microscope, with reflectance values falling between that of liptinite and inertinite; the values change with progressive rank and chemical alteration due to maturation (Plate 11). Vitrinite does not display anisotrophy when viewed under cross-polarized light, enabling easy differentiation from dark semifusinite (reactive semifusinite), but may show slight fluorescence. The fluorescence may be a result of bituminous substances formed during coalification. In cases where the vitrinite is clearly fluorescing, the reflectance of the particles may be suppressed, leading to inaccurate rank determination. This is discussed further in Bustin *et al.* (1983).

The maceral group vitrinite is defined by the level of reflectance; the three subgroups are divided based on: 1) relatively pure layers or lenses; 2) continuous phase of groundmass binding other coal components; 3) amorphous filling of cells, pores, and fissures (ICCP, 1998) (Table 2.2). In terms of polishing hardness, vitrinite is soft and shows no relief (except possibly corpogelinite).

The chemical structure of vitrinite is characterised by a relatively high oxygen content compared to the other maceral groups, with oxygen levels steadily decreasing with an increase in rank as the degree of aromaticity and condensation increase. Relatively rich in aromatic structures, vitrinite does also contain aliphatic compounds. Its properties affect most industrial processes in which coal is utilised.

For practical purposes, knowledge of the undifferentiated vitrinite content of coal is usually sufficient. Specialised studies, however, may require knowledge of the various individual macerals in the vitrinite group, a list of which is given in Table 2.2. The ICCP (1998) publication applies, as well as Taylor *et al.* (1998), Suárez-Ruiz and Crelling (2008), O’Keefe *et al.*, 2013; and the reader is advised to follow the ICCP website ([www.iccop.org](http://www.iccop.org/)) for up to date changes in terminology and classification.

TABLE 2.2: Macerals in the Vitrinite Maceral Group (following the ICCP System 1994)

|  |  |  |  |
| --- | --- | --- | --- |
| MACERAL SUBGROUP | MACERAL | FORM | PLATE |
| Telovitrinite (preserved botanical cell structures may or may not be visible)  Detrovitrinite (finely fragmented vitrinized plant remains <20 µm, isolated or cemented by amorphous vitrinitic matter)  Gelovitrinite (colloidal infillings of vitrinitic material in former voids; relatively rare sub-maceral group) | Telinite  Collotelinite    Vitrodetrinite  Collodetrinite  Gelinite  Corpogelinite | Coalified cell walls of recognisable plant tissues, more or less intact.  More–or-less homogeneous structure less layers; may show mottling in low rank coals; widely used as rank indicator.  Discrete, small vitrinitic fragments of varying shape; a secondary maceral.  Mottled, compact vitrinitic groundmass binding other coal components.  Pure colloidal gel, homogenous infilling of cracks and other voids; a secondary maceral.  Structure-less homogenous and discrete bodies of humic cell infillings. | 2  3-4  5  5-6  7  8 |

Pseudovitrinite is not included in Table 2.2, but is an important form of vitrinite due to its negative impact on coking properties. Pseudovitrinite, a term coined in 1966 by Benedict *et al.* (as stated by Kaegi, 1985), is believed to be derived from telovitrinite, more specifically collotelinite. It exhibits a somewhat higher reflectance than collotelinite, at least 0.025% higher in the same sample. Typified by small slits, typically perpendicular to bedding planes, and serrated fragment margins (O’Keefe *et al.*, 2013), pseudovitrinite is fairly common in southern African Permian-aged coals (Kruszewska, 2003). This maceral is considered to be the product of in situ dessication (drying out), possibly accompanied by a degree of oxidation in the seam after gelification had consolidated the vitrinite-rich material. The reader should consult Kaegi (1985) and Mastalerz and Drobniak (2005) for a more detailed discussion on the identification and origin of pseudovitrinite; also refer to Plate 9 for photographic images.

Inertinite Maceral Group

Inertinite represents a group of macerals derived from plant material that has been strongly altered and degraded in oxidising conditions before or in the peat stage of coal formation. Redox, biochemical, and chemical processes also impacted on inertinite formation. Scott and Glasspool (2006) believe that most, if not all, inertinite macerals formed through exposure to elevated temperatures. However, whilst some inertinite maceral forms almost certainly were derived from peat fires, aerobic or –sub-aerobic decomposition during the process of oxidation and ultimate carbon-enrichment termed fusinization played a key role in the formation of inertinite macerals (O’Keefe *et al.*, 2013) (Fig. 2.4). This results in maceral forms with reflectance values higher than vitrinite and liptinite maceral groups at the same rank. In many cases the original parent material is the same woody and cellular constituents from which the vitrinite maceral group is derived. Depending upon the extent of tissue degradation, these constituents may retain their original strong, well-defined plant tissue structures, or exhibit varying degrees of reduced structures, ending finally in fairly homogeneous structure less forms (Table 2.3). Some of the structure less inertinite occurs as broken pieces of the structured inertinite; the remainder of the structure-less forms may represent the oxidised residue of non-woody plants.

Subdivision of the inertinite maceral group is based upon reflectance, the original parent material, and the effects of sedimentation on detrital forms. The grey level observed petrographically may be more closely related to the depositional environment rather than the degree of coalification, with the reflectance dependent primarily on the chemical composition of the inertinite macerals (ICCP, 2001). Inertinite macerals do not typically exhibit fluorescence, and may exhibit anisotrophy.

Relative to the other maceral groups, the chemical structure of inertinite is characterised by high carbon and low oxygen and hydrogen content (ICCP, 2001), with a high degree of aromatization. Gondwana coals tend to have a higher inertinite content than northern hemisphere coals, largely related to the climate at time of peatification. But some Cretaceous and Jurassic coals of North America, China, and Europe are inertinite-rich. Due to the dominance of inertinite in South African coals, semifusinite is subdivided into reactive and inert semifusinite, with the reactive semifusinite exhibiting a shade of grey close to the corresponding vitrinite in the same unheated sample, with comparable chemical and technological properties (Hagelskamp and Snyman, 1988).

A list of inertinite macerals is given in Table 2.3. The ICCP (2001) publication applies, as well as Taylor et al. (1998) and O’Keefe et al. (2013); and the reader is advised to follow the ICCP website ([www.iccop.org](http://www.iccop.org/)) for up to date changes in terminology and classification. The term sclerotinite was replaced by the terms funginite and secretinite, thus differentiating between two macerals with different origin and formation pathways.

Liptinite Maceral Group

Liptinite is derived from non-humifiable plant matter (Taylor et al., 1998; Pickel et al., 2017). The term liptinite has replaced the term exinite as it was determined that liptinite is a more accurate description representing the chemically distinct parts of plants rather than just the chemically-resistant exines of spores. Thus, the term includes: spores; cuticles; suberine cell walls (suberinite); resins (resinite); algae; and polymerised waxes, fats and oils of vegetable origin. The subdivision of the liptinite maceral group is based upon specific plants (algae), parts of plants (spores and cuticles), or products of plants (waxes and resins). This maceral group is far less common than vitrinite and inertinite, except in certain cases where high concentrations of liptinite material occur in sapropelic bands of coal, forming cannel coals (sporinite- and cutinite-rich) and boghead (algal-rich) coals. In some places, such liptinite-rich coal bands are commonly and collectively termed “torbanite”, an informal name arising from a town in Scotland where such material was first found; rare torbanite deposits do occur in South Africa. Alginite, typically occurring in deep water coals, and bituminite may be associated with sedimentary rocks assessed for shale gas potential.

TABLE 2.3: Macerals in the Inertinite Maceral Group (following the ICCP System 1994 (ICCP, 2001), with some modifications)

|  |  |  |  |
| --- | --- | --- | --- |
| MACERAL SUBGROUP | MACERAL | FORM | PLATE |
| With plant cell structures  Macerals lacking plant cell structures  Fragmented | Fusinite  Semifusinite – inert (ISF)  Semifusinite – reactive (RSF)  Funginite  Secretinite  Micrinite  Macrinite  Inertodetrinite | Highly reflecting, well preserved cellular structure often intact or fragmented (bogen structure); no fluorescence; hard; exact origin debated between incomplete combustion in peat fires, and / or advanced oxidation.  Intermediate structure and reflectance between vitrinite and fusinite in the same coal; derived from wood (more Northern hemisphere coals) or leaves (especially Gondwana coals); shows irregular anisotropy; common component of southern African coals.  RSF reflectance values close to vitrinite at same rank, but the maceral exhibits structure and possibly partial anisotropy.  Fungal remains; highly reflecting single or multi-celled fungal spores, sclerotia, hyphae, mycelia. Almost completely absent in southern Africa coals, more common in Northern Hemisphere coals, and low rank coals of Asia.  Round to oblong, non-cellular, vesicled to non-vesicled forms with high reflectance; may exhibit low reflectance; previously referred to as sclerotinite; origin uncertain – maybe oxidised resins; common globally.  Very small, rounded organic grains occurring as aggregates, of inertinitic reflectance with upper size limit of 2 µm, distinguishing micrinite from inertodetrinite; generally considered a secondary maceral; occurs in most coals globally, but not that common. Origin uncertain, possibly derived from liptinite, or inertinitic fragments.  Large compact, amorphous, structure-less inertinite of variable shape; variable origin, including metabolic products of fungi/bacteria, invertebrate excrement, or flocculated humic matrix substances partially oxidised; occurs globally, but generally rare.  Discrete inertinitic fragments 2-10 µm of varying shape without microscopically recognisable structure; excludes fusinite fragments; secondary maceral derived from oxidised/fusinised organic precursors that have undergone desiccation, mechanical crushing, attrition before / during decomposition and compaction; common in Gondwana coals. | 12-14  15-16  17-18  19-21  22  23  24-26 |

Liptinite exhibits lower reflectance than other organic components in the same sample of sedimentary rock and coal in low and medium rank coal, appearing black to dark grey or rusty brown in reflected white light. Under short wavelength radiation, such as ultraviolet or blue light fluorescence, liptinite is observed to autofluoresce, possibly with different intensities related to rank, maceral type, chemical composition, and degree of oxidation / alteration. A more orange fluorescence occurs at higher rank, with a more yellow (blue-light excitation) or greenish-yellow (UV excitation) at lower rank (Bustin et al., 1983; Pickel et al., 2017). Compared to the other maceral groups, the chemical structure of liptinite is more hydrogen-rich and aliphatic. At ranks above medium volatile bituminous, liptinite begins to fade out, becoming extinguished in more mature coals (typically above 1.5 RoVmr%) as chemical changes occurring during maturation render their optical distinction from vitrinite and inertinite macerals difficult. Fluorescence microscopy is important to accurately detect and classify liptinite macerals, especially where liptinite is closely associated with clays (Kruszewska, 2003).

A list of macerals in the liptinite group is given in Table 2.4. The ICCP recently updated the classification of liptinite (Pickel et al., 2017). Taylor et al. (1998) and O’Keefe et al. (2013) are useful references, and the reader is advised to follow the ICCP website ([www.iccop.org](http://www.iccop.org/)) for up to date changes in terminology and classification. All liptinite forms excluding suberinite and bituminite are observable in southern African coals, typically occurring as less than 10 vol %, although there are exceptions.

TABLE 2.4: Macerals in the Liptinite Maceral Group

|  |  |  |
| --- | --- | --- |
| MACERAL SUBGROUP | FORM | PLATE |
| Sporinite  Cutinite  Exudatinite  Resinite  Alginite  Suberinite  Liptodetrinite  Bituminite | Outer membranes (exines) of micro-, macro and megaspores; usually compressed at higher rank and aligned with sediments, well-preserved, variable shape and wall thickness, distinct botanical forms. Most common form of liptinite.  From leaf and stem cuticles. Elongated, usually serrated, well preserved, distinct botanical forms, parallel to stratification. May exhibit serrations on one edge.  A secondary maceral, infilling voids, cavities, empty cells; strong fluorescence. Derived from hydrogen-rich sources (e.g. liptinite).  Pod-shaped, globular or irregular bodies occurring as discrete forms; may fill cellular cavities or amorphous shapes in vitrinitic lenses; origin is cell excretions; can show red internal reflections. Where the reflectance is comparable to vitrinite, is termed corpogelinite, or secretinite where the reflectance is higher.  Algal colonies or solitary, of planktonic and benthic origin; rounded, elongated, semi-compressed, distinct and well-preserved in botanical form. Rare in humic coals; subcategories: telalginite being larger, more structurally distinct than lamalginite.  A rare form in lignites, from suberous tissues, well defined cell structure; intense fluorescence. Not encountered in southern Africa due to rank.  Small detrital fragments of the above macerals. Rare.  An amorphous degradation product, distinguished from vitrinite by lower reflectance and higher fluorescent intensity. Occurs as fine-granular groundmass, or laminae, streaks, whisps, bands, threads. Extremely rare. | 28-31  32-33  34  35  36 |

* + - 1. Minerals in coal (grade)

Minerals occur in several forms in coal, and are highly variable in physical appearance and chemical composition. Mineral matter may be deposited during peat formation either arising from dust particles blown in by wind or from precipitation deposited during the passage of ground water solutions. It may also arise from inorganic compounds or organo-metallic complexes found in the original plant material (Fig. 2.7). Minerals which are observable microscopically can be included in the petrographic analyses; but mineral matter finer than the optical resolution of the microscopic system in use can be overlooked. Thus, ash determination via proximate analysis and ash oxide analysis are vital to complement the petrographic determined mineral matter.

Figure 2.7: The origins and types of mineral matter in coal (modified from Falcon and Snyman, 1986)

The forms in which minerals may occur in coal fall into two major categories (Fig. 2.7). The first category includes the intrinsic (syngenetic) inorganic matter which was present in the original living plant tissue (also referred to as inherent mineral matter, including trace elements). This mineral matter is ultimately trapped in coal in the form of sub-microscopic mineral grains and as organo-metallic complexes (including pyrite, siderite, and colloidal clays, for example). Included intrinsic mineral matter may occur as discrete minerals grains within the coally matrix (e.g. quartz, clays, and to a lesser extent feldspar, apatite, muscovite), introduced by means of wind and water or precipitation in situ. Cell lumens, commonly preserved in macerals, may contain minerals, where the cell lumens were infilled with clays prior to compaction, thus largely retaining the original cellular structure. Intrinsic mineral matter is typically difficult to beneficiate. However, excluded intrinsic mineral matter are grains external to the coal seam, derived from the roof or the floor (Falcon, 2013), and are readily beneficiated, if included during mining.

The second category includes the extrinsic (epigenetic) or introduced forms of mineral matter, typically deposited by percolating waters into fractures, cavities and pores within the coal seam long after the initial accumulation of the peat. These minerals forms may occur as nodules, replace, transform, or encapsulate syngenetic minerals, and cut across bedding planes, occasionally disrupting the original layers. Trace elements may become incorporated into these secondary deposits. Various epigenetic carbonate minerals (calcite, dolomite, ankerite), pyrite, marcasite, and some clays are more easily removed during beneficiation than syngenetic minerals.

Mineral alterations due to oxidation and / or weathering can be observed petrographically, including mineral salts and jarosite (oxidation of pyrite).

Table 2.5: Minerals found in coal (Hower et al., 1998); other minerals are possible.

(dom – dominant; ab-abundant; c – common; r – rare; v r – very rare)

|  |  |  |  |
| --- | --- | --- | --- |
| Mineral Group | Minerals | Composition | Occurrence |
| Clay | illite-sericite | (K, H3O)(Al, Mg, Fe)2(Si,Al)4O10(OH)2βnH2O | dom - ab |
| montmorillonite | Al4Si4O10(OH)2βnH2O | r - c |
| kaolinite | Al4Si4O10(OH)8 | c - v c |
| halloysite | Al2Si2O5(OH)4 | rare |
| Iron disulfides | pyrite | FeS2 | r - c |
| marcasite | FeS2 | rare |
| melnikovite | FeS2 | rare |
| Carbonates | siderite | FeCO3 | c - v c |
| ankerite | CaFe(CO3)2 | c - v c |
| calcite | CaCO3 | c - v c |
| dolomite | CaMg(CO3)2 | r - c |
| Oxides | hematite | Fe2O3 | rare |
| magnetite | FeFe2O4 | v r |
| rutile | TiO2 | v r |
| Hydroxides | limonite | HFeO2 | r - c |
| goethite | HFeO2 | rare |
| diaspore | HAIO2 | rare |
| Sulfides | sphalerite | ZnS | rare |
| galena | PbS | rare |
| chalcopyrite | CuFeS2 | v r |
| pyrrhotite | FeS2 | v r |
| Phosphates | apatite | Ca5(PO4)3(F, Cl, OH) | rare |
| crandallite group | (Ba, Ca, Ce, Nd, Pb, Sr, Th)(Al, Fe)3[(P, As)O4]2(OH)5 with or without H2O | rare |
| Sulfates | barite | BaSO4 | rare |
| gypsum | CaSO4β 2H2O | rare |
| Silicates | quartz | SiO2 | r - c |
| zircon | ZrSiO4 | rare |
| biotite | K(Mg, Fe)3(AlSi3O10)(OH)2 | v r |
| staurolite | Fe2Al9O6(SiO4)4(O, OH)2 | v r |
| tourmaline | Na(Mg, Fe)3Al6(BO3)3(Si6O18)(OH)4 | v r |
| garnet | (Fe, Mg, Ca, Mn)3(Al. Fe, Cr)2(SiO4)3 | v r |
| epidote | Ca2(Al, Fe)Al2O(SiO4)(Si2O7)(OH) | v r |
| sanidine | KAlSi3O8 | rare |
| orthoclase | KAlSi3O8 | v r |
| augite | Ca(Mg, Fe, Al)(Al, Si)2O6 | v r |
| amphibole | (Na, Ca)2(Mg, Fe, Al)5(Si, Al)8O22(OH)2 | v r |
| kyanite | Al2SiO5 | v r |
| chlorite | (Mg, Fe, Al)6(Al, Si)4O10(OH)8 | rare |
| Salts | syvite | KCl | v r - c |
| halite | NaCl | v r - c |
| kieserite | MgSO4BH2O | v r - rare |
| mirabilite | Na2SO4B10H2O | v r - rare |
| melanterite | FeSO4B7H2O | v r |

Table 2.5 lists the key minerals observed in coals globally (also refer to Ward, 2002; Thomas, 2013, Ward, 2016). The most abundant minerals in southern African coals are clays (kaolinite and illite), with a variety of common carbonates (calcite and dolomite), sulphides (pyrite), and quartz, plus rarer apatite, rutile, and zircon. As stated by Pinetown et al. (2007), mineral matter typically makes up between 8 – 35 weight % in coals from the Mpumalanga coalfields, dominated by clays. Photographic illustrations of the most common southern African mineral forms may be found in Plates 37 – 43, and associated with carbominerites in Plate 47. Table 2.6 highlights the optical properties of certain minerals as observed petrographically.

Clay

Clay is overall the most abundant and widely-spread mineral occurring in coal globally and in southern Africa, often comprising up to 80% of the minerals present (Falcon, 2013). It occurs as minute grains (1-2 µm), as small lenses, microscopically visible bands, infilling cell cavities (flocculated clays), replacing cell structures to form clay petrifications, and as veins. Kaolinite is a common syngenetic clay mineral, with illite and montmorillonite also being determined in many coalfields. In some coals, the clay occurs in vermicular form, or flocculated form infilling cell lumens, or infilling worm burrows. In reflected light, under oil immersion, clay has a very variable appearance. It can appear from almost white (due to internal reflections) to orange-brown with an uneven granular texture (most common form), to almost black with an even, matt surface. However, it is most commonly brown and granular and occurs in small lenses.

Quartz

Quartz occurs in most coals globally, but generally only in small quantities. It may be found as small (5 - 20µm) isolated syngenetic detrital grains, or as sharp, angular, sub-rounded, or well-rounded larger forms. The shape is largely determined by the distance travelled before inclusion into the peat mire. Locally, where a seam has been invaded by vitreous silica, chalcedony veins, petrifications, and cell fillings are abundant. In reflected light, under oil immersion, quartz appears glassy, as its refractive index is very close to that of oil. A polished surface of coal should be viewed first in air to determine whether quartz is present or not. In air, quartz is darker grey than the coal and has a much higher relief because of its greater hardness. Internal reflectance can also be determined, and the inclusion of colourful rutile rods occur in some particles.

Carbonates

Carbonate minerals occurs as nodules, usually of siderite, and as veins and cell fillings of calcite, dolomite, and ankerite. They can be syngenetic or epigenetic, with significant occurrences occurring in southern African coals associated with hydrothermal fluid precipitation. In reflected light, under oil immersion, siderites are typically brownish grey and very anisotropic, occasionally showing crystalline textures. Calcite may be lighter, and may exhibit twinning.

TABLE 2.6: Optical properties of common minerals observed petrographically

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| MINERALS | INTENSITY  OF LIGHT  REFLECTION | COLOUR | ANISOTROPISM | PHYSICAL DISPOSITION AND HARDNESS | Plate  No. |
| Kaolinite  Illite  Montmorilonite | No Reflection | Light to dark brown to black, granular | Isotropic | Scaly, soft; can swell in water | 37 |
| Quartz | Low | Glassy | Weakly anisotropic, internal reflection | Granular, hard | 38 |
| Pyrite | Very high | Yellow to bronze | Isotropic | Granular or massive, hard | 39-40 |
| Marcasite | Very high | Pale bronze-yellow | Highly anisotropic | Tabular, often repeatedly twinned, radiating aggregates, hard |  |
| Siderite | High | Yellow-brown | Highly anisotropic, white-brown reflections | Rhombohedral cleavage, compact or nodular, medium hardness | 41 |
| Calcite  Dolomite  Ankerite | Medium | Colourless to white-yellow-brownish | Highly anisotropic, white internal reflections | Rhombohedral cleavage, twinning, medium hardness | 42 |
| Haematite (secondary mineral) | High | White to blue-red without immersion, steel-grey in immersion | Strong anisotropism, red internal reflections | Tabular, granular, fibrous, hard |  |
| Limonite-Goethite | Medium | Grey | Weakly anisotropic, especially in oil immersion, brown internal reflections | Massive, compact, granular, hardness medium, often as a secondary overgrowth on pyrite marcasite |  |

Iron sulphides

Gondwana coals are generally low in iron sulphide minerals compared to Laurasian coals (Falcon, 2013). The iron sulphides, being pyrite and to a lesser extent marcasite, are ubiquitous in southern African coals, but usually in very low quantities, except for certain areas and one or two seams. Pyrite can occur in a wide variety of modes, from tiny micron sized nodules included in the macerals (syngenetic), to large nodules or replacement concretions (epigenetic), and infilling cell lumen and cleats (syngenetic / epigenetic). It has also been observed to replace or transform other minerals. In reflected light, under oil immersion, pyrite is extremely bright yellow, being about 50 times as bright as the surrounding coal, and generally exhibit relief.

Other minerals

Apatite, barite, gypsum, and sphalerite are found rarely in coal, but can be locally significant. Apatite occurs as very fine grains, generally micron to sub-micron in size, distributed within a maceral, frequently being vitrinite. It is of significance in terms of coking coal, as the phosphorous content is deleterious to metallurgical materials.

* + - 1. Microlithotypes

The organic and inorganic constituents of coal combine in various associations to form microscopic associations, termed microlithotypes. By definition, microlithotypes have band widths greater than 50 µm and contain at least 5 % of a maceral group (ISO, 1994; Taylor et al., 1998). Lithotypes are the megascopic associations of microlithotypes, as discussed above. Microlithotypes may be composed of pure macerals (i.e. monomaceral forms of microlithotypes) or varying proportions of different macerals (bimaceral forms if two macerals comprise the association), or trimaceral if three are present). If minerals are present in proportions greater than 20% by volume (or greater than 5% by volume for sulphide minerals specifically) then the association is termed carbominerite, irrespective of however many types of macerals are present (Table 2.7). If minerals are present in proportions greater than 60%, the microlithotype is termed minerite (or rock).

All microlithotypes bear the suffix “-ite” in order to distinguish them from macerals. The physical properties of microlithotypes are related to the maceral assemblage. The chemical composition will reflect the predominating macerals.

Tables 2.7 and 2.8 present a summary of the major microlithotypes in bituminous coals and anthracite and their characteristic constituents, subdivided based on organic and carbominerite composition. Figure 2.8 presents the associations diagrammatically.

The arrangement of microlithotypes, including and excluding mineral contaminants, is important technologically. Microlithotypes provide information about the genesis of coal seams, and can assist in solving problems of seam correlation, as well as issues pertaining to crushing, washing, and utilisation. Figure 2.9 illustrates the behaviour of vitrite when constrained by inertite (bimaceral particle) rather than as a pure monomaceral. The technological importance of microlithotypes are discussed further in Chapter 3.

TABLE 2.7: Summary of Microlithotypes

|  |  |  |  |
| --- | --- | --- | --- |
| MICROLITHOTYPE | MACERAL-GROUP COMPOSITION (MINERAL-FREE) | MICROLITHOTYPE GROUP | PLATE REF |
| Monomaceral  Collite  Telite | V > 95% | Vitrite | 44 |
| Sporite  Cutite  Resite  Algite | L > 95% | Liptite |
| Semifusite  Fusite  Secretite  Inertodetri[ni?[te  Micrite  Macrite | I > 95% | Inertite |
| Bimaceral  Sporoclarite  Cuticloclarite  Resinoclarite  Algoclarite | V + L > 95% | Clarite  V,L | 45  46 |
|  | V + I > 95% | Vitrinertite  V, I |
|  | I + L > 95% | Durite |
| Trimaceral  Duroclarite  Vitrinertoliptite  Clarodurite | V > I, L  L > I, V  I > V, L | Trimacerite  V, I, L |

TABLE 2.8: Summary of Carbominerite

|  |  |  |
| --- | --- | --- |
| Association of coal with a specific mineral or mineral group | Composition | Plate  Reference |
| Carbargilite | Coal + 20-60 Vol %  clay minerals | 47 |
| Carbosilicate | Coal + 20-60 Vol %  Quartz |
| Carbopyrite | Coal + 5-20 Vol %  Sulphides |
| Carbankerite | Coal + 20-60 Vol %  Carbonates |
| Carbopolyminerite | Coal + 20-60 Vol %  Mineral matter |
| Minerite (rock) | >60% mineral matter |

Figure 2.8: The Association of Macerals into Microlithotypes (modified from Falcon and Snyman, 1986)

Figure 2.9: The arrangement of macerals in terms of microlithotypes is important technologically: A) here a vitrite band is constrained between two layers of inertinite reducing its ability to swell. B) vitrite, occurring as a monomaceral, has not been constrained during pyrolysis and, therefore, is seen to have swollen and expanded significantly, with thin walls and large pores.

* 1. SAMPLING, PREPARATION AND ANALYTICAL TECHNIQUES FOR PETROGRAPHIC ANALYSIS
     1. Sampling

For quantitative petrographic analysis, it is most important that the coal samples are representative of the parent source. Zhu (2014) provides an excellent document on coal sampling standards globally, and SANS 18283:2007, 14180:1998, and 139029-1:2001 and associated documents apply to coal sampling in the South African context. As stated by Zhu (2014), about 80% of total variances in sample analysis come from errors during collection. All samples must be representatively selected and stored appropriately before analysis (airtight storage to prevent oxidation and/or unrequired physical changes). Sampling can occur in seam (box cut, mine face, or borehole cores); or from moving streams (conveyor belts) (Taylor et al., 1998); or sampled from trucks, railway wagons, ships, or stockpiles. Such sampling may be done mechanically or manually.

1. In seam – i.e. selected bands, or blocks, channels, or the entire vertical face of a coal seam. In this case samples may be taken in one locality (sampled from boreholes or as hand-cut vertical channel samples from an exposed seam face) or repeated at specific lateral distances apart. Unweathered samples must be selected (unless the study involves the weathering of the coal). Care must be taken to ensure sample integrity is maintained and the correct orientation with respect to the sediments is recorded and marked on the sample / sample bag. This type of sampling is necessary when investigating the range of variations in coal quality within a seam, both vertically and laterally and in response to specific geological features (for example in the proximity of faults and igneous intrusions). Such information is used in reserve evaluations, quality distribution and in mine planning operations when the selecting or blending of different qualities for specific purpose may be necessary.
2. Moving streams or static storage sites. Once removed from the seam, coals may need to be sampled at various points: for example, as a run-of-mine product (usually taken from conveyor belts), during or after crushing, milling or beneficiation operations, during transportation (by road, rail or sea), in storage (in dumps or stockpiles), and finally prior to utilisation (e.g. pulverised fuel to a boiler). In these cases, the size of the coal pieces and the quantity to be sampled are of prime importance in determining the number and weight of increments (sub-samples) to be taken in order to make a composite sample which will be statistically representative of the original product. It is necessary to consult and apply suitable standard procedures.
   * 1. Preparation

The most widespread method of microscopic examination of coal is the use of reflected incident light on polished surfaces of grain mounts, under oil immersion, to enhance reflectivity differences. SABS ISO 7404 part 2 applies here, as well as Taylor et al. (1998). Thin sections are rarely used these days, but are discussed by Taylor et al. (1998).

To prepare the coal for statistical analysis, the sample is crushed to pass a 1 mm sieve (with a minimum of fines generated) and then riffled to obtain a 15 to 100 g sample as representative as that used in conventional proximate analyses. In some instances, it may be necessary to remove excess fine particles; i.e. particles passing a -20 or -30 µm sieve. This should not be done for chemical analyses or instances where the sample cannot be biased in any way. The 1 mm sample is mixed with epoxy resin and hardener, and moulded as a block mount (typically 20 or 30 mm in diameter, depending on the polishing equipment sample holder). This is left to cure (up to 24 hours, preferably under vacuum to remove all air bubbles). Subsequently, the surface of each block is ground and polished to the required level for petrographic analysis (Fig. 2.10). The objective is to achieve a clean, uniformly flat, relief , -pit-, and scratch-free surface of the embedded coal particles. A series of grinding papers and polishing clothes, with water and / or polishing compounds of decreasing particle size are used on an automatic polisher; there are a variety of different suppliers of equipment and polishing consumables globally. SABS ISO 7404 part 2 and Taylor et al. (1998) provide typical sequence of events, typically finishing on a 0.05 µ alumina cloth.

Figure 2.10: A summary of the major sampling and preparation steps necessary for the statistical petrographic analyses (modified from Falcon and Snyman, 1986)

For more detailed scientific studies of a seam, for example its genetic origin by means of the in-situ sequence of bandings, a series of small, whole pieces of coal cut perpendicular to the direction of the bedding may be embedded in the epoxy resin. In this case, orientation relative to the original seam must be noted and then the surface of the coal and resin block polished (usually by hand). This approach is, however, more academic in nature and is not typically used in commercial practice (Hower and Wagner, 2012). However, the macroscopic scale texture of the coal can be observed and the juxtaposition of lithotype deposition is preserved.

It is of prime importance that the polished surface of the coal block be of very high quality (refer to Plate 1 and Fig. 2.11). The criteria used to judge a polished surface of coal may be summarised as follows:

1. The finished polished surface should be as free from relief as possible; certain macerals and minerals show enhanced relief.
2. The finished polished surface should be free from pits and scratches.
3. All inclusions of mineral matter should be retained naturally in the finished polished surface.
4. The macerals of the coal should be free from burring, burning, or smearing caused by “over-polishing”.
5. The final surface prepared for study should be free of grinding and polishing chemical solutions.

Figure 2.11: Example of poor polishing: surface scratches and residual polishing solution are visible.

Chemical etching of the polished surface can be used to reveal botanical features of macerals, specifically vitrinite, not always evident on unetched block mounts. Etched particles provide useful insight into the identification of plant tissue types and coal facies, revealing the internal make-up of macerals. Etching makes use of potassium permanganate (Stach et al., 1982; Taylor et al., 1998; Hower et al. 1998.; ASTM D5671-95 (2011)). The notes accompanying Plate 10 provides a summary of an etching technique used to prepare the samples photographed in Plate 10.

* + 1. Analytical Techniques

Conventional petrographic investigations are carried out in reflected incident/white light at a total magnification of between 250 and 500 times. The equipment required for use in petrographic analysis is a petrological polarising microscope, with oil-immersion objectives (typically x40, x50, x60), eye pieces with magnification between x8 and x12 and a cross hair (or graticule), mechanical or automated stage, stable light source (LED is now favoured over halogen lamps), an ultra-violet light source or blue light filter cube for fluorescence, and a system to measure reflectance (there are a variety of different systems on the market currently). The immersion oil must be non-drying, non-corrosive, with a refractive index of 1.518 at 23oC and a wavelength of 546 nm (ISO, 1994). A rotatable stage is good to have and lambda plates along with an analyser to assess anisotrophy.

The techniques used for quantification and qualification of coal composition are those recommended by the ICCP and the standards mentioned above in Section 2.2; Stach et al. (1982) and Taylor et al. (1998) are also excellent reference sources. Under reflected light, the different components making up coal are differentiated by their various colours, or shades of grey, due to reflectivity or adsorption of light, as well as structure. Some components, both organic and inorganic, may fluoresce when illuminated using light of short wavelength light, such as ultra-violet or blue light.

The type of coal is determined petrographically by group maceral analysis and/or microlithotype analysis, where the organic components are assessed based on structure and the shade of grey.

The rank is measured by means of vitrinite reflectance, typically on collotelinite.

Coal grade, or the mineral matter component, can be assessed in the type analysis, or can be assessed independently of the organic matter.

Figure 2.12 demonstrates the key differences optically between the maceral, microlithotype, and reflectance analytical techniques. It must be clearly understood that coal petrography, whilst providing a significant amount of information pertaining to the understanding of coal origin and utilisation predictions, should not stand alone and requires evaluation with geochemical and physical data to provide a holistic understanding of the coal sample.

Figure 2.12: A summary of the major analytical techniques used in coal petrography (modified from Falcon and Snyman, 1986)

The standardised petrographic analytical techniques are: i) maceral analysis, ii) microlithotype analysis, iii) reflectance analysis, all largely based on ISO standards 7404 parts 3, 4, and 5, respectively, and are discussed below. Non-standardised petrographic analyses are also outlined below and discussed in more detail in Chapter 3.

* + - 1. Maceral Analyses

The composition of coal (coal type) is determined by classifying and counting macerals microscopically. Maceral analyses are carried out on well polished grain mounts / block mounts under oil-immersion using a suitable oil-immersion objective (typically to enable a total magnification of x500) and an automatic point countering stage at traverse spacing of 0.4mm and intervals between the traverses of 0.5 mm; alternatively, according to ISO 7404-3, the step length is equal to half the maximum particle diameter. The traverse spacing’s can be amended depending on the point count system, block mount size, and particle size and distribution, as long as a systematic scheme of transection to cover the entire surface of the block mount is conducted. At least 500 points (excluding binding resin) are counted under the cross hair (Fig. 2.12, and ISO 7404-3), and, depending on the purpose of the analysis, visible minerals may be ignored, counted collectively or counted individually. Macerals may also be counted as groups only, or as individual macerals. It is generally accepted in southern Africa that apart from vitrinite, liptinite, and inertinite macerals, reactive inertinite macerals should also be distinguished for technological purposes.

The results are presented as volume %. According to the standard ISO 7404 part 3, an accurate assessment of a well prepared surface will statistically represent the entirety of the coal block and thus the coal sample under assessment. Repeatability and reproducibility limits are provided in ISO 7404-3, and vary depending on the percentage of the component. However, a good rule of thumb is that the repeatability limit should be 2.5% and the reproducibility limit 5%. As geochemical data is expressed in weight %, caution is advised when comparing petrographic and geochemical data, unless the data has been converted to the same unit. Macerals are discussed in more detail in Section 4.1. The ICCP offer accreditation in the determination of maceral groups (SCAP – single coal accreditation program, [www.iccop.org](http://www.iccop.org/)).

* + - 1. Microlithotype Analyses

Microlithotype analyses are carried out in a similar manner to maceral analysis (i.e. under reflected white light with immersion oil), but in this case a suitable 20 point graticule in an ocular of the microscope is used. The effective distance of the graticule is 50x50 microns, and each intersection of the graticule, therefore, represents 5% of the total number of intersections (Fig. 12). For a reading, at least ten graticule intersections must cover the coal particle/s. For a complete microlithotype analysis, 500 areas have to be measured in a systematic scheme of transection, and the results are expressed at volume percentage (vol %).

Where a graticule is not present in the ocular, it is possible to conduct the analysis selon-la-ligne, or according to the line, using a 100 division micrometer scale (Taylor et al., 1998). The same principle applies, with the micrometer divided into 5% intersections.

The position of the graticule sections, or proportion of the 50x50 micron area, with respect to maceral composition determines the microlithotype is as follows:

Monomaceral: all intersections cover a single maceral (i.e. vitrinite, inertinite, liptinite).

Bimaceral: at least one intersection on each of the two macerals of which the microlithotype is composed (i.e. clarite, durite, vitrinite).

Trimaceral: at least one intersection on each of the three macerals of which the microlithotype is composed.

Further detail is provided in Table 2.7.

Where intersections occur on minerals, it must be determined whether the carbominerite categories apply, or if greater than 60%, the category ‘rock’ (or Minerite) is given (Table 2.8). Carbargillite applies when more than 20% of the graticule points fall on clays, carbankerite for carbonates, and carbosilicate for quartz. Where more than 5% of the graticule points fall on pyrite or other sulphides, carbopyrite is recorded. This is because the density of pyrite is so much higher than the other components.

The 20 point graticule may also be used to obtain a combined maceral-microlithotype analysis, i.e. maceral and microlithotype analyses are carried out at the same time (Hower and Wagner, 2012). Twenty intersections indicate the microlithotype composition and one fixed intersection defines a maceral. The advantage of this method is that not only are the measurements of microlithotypes and macerals done simultaneously, but also the estimation of the maceral proportions in bi- and trimaceral microlithotypes. This technique is especially useful when assessing whole-coal blocks rather than crushed particles, and should be conducted perpendicular to the bedding plane.

* + - 1. Reflectance Measurements

Coal rank is commonly expressed in terms of vitrinite reflectance. Vitrinite exhibits a constant and linear loss of volatile matter and increase in carbon content with rank, not evident in the other macerals. The linear change is observed petrographically by an increase in the whiteness of the maceral. Reflectance measurements are taken on the smooth, scratch-free, relief-free, polished surface of vitrinite particles (homogenous bands of collotelinite in particular) and are carried out in monochromatic green light (ʎ = 546 nm) by means of a photomultiplier or alternative detectors such as spectrometers with fibre optic cables, or digital imaging. Figure 2.13 shows a range of reflectance readings taken on different vitrinite macerals within a single coal particle.

Random reflectance (%RoVmr), typically used by the power and trade industries, is measured with the polarizer removed from the optical path of the microscope and without rotating the microscope stage. The operator moves across the block mount surface, typically following a raster pattern, and readings are taken on the grain in the orientation in which it is encountered. This differs from maximum vitrinite reflectance. The ICCP offers accreditation in the determination of vitrinite reflectance (SCAP – single coal accreditation program, [www.iccop.org](http://www.iccop.org/)).

For the measurement of maximum reflectance (%RoVmax), typically used in the coking industry, the polarizer should be in the 45ᵒ position if using a Berek prism, or zero if using a glass plate or Smith illuminator. The microscope stage is rotated through 360ᵒ and the maximum reading is taken. For this purpose it is essential that the objective with a high magnification (50 or 60X) is used and that the objective is perfectly centred. With new automated microscopic systems, the computerised system may rotate the light path, rather than having to rotate the stage. The method of determining maximum reflectance by calculation is not recommended where a high degree of accuracy is required, but can be used for assessment purposes. As low rank coals are optically isotropic, random and maximum reflectance readings are typically the same (Suárez-Ruiz and Ward, 2008).

Bireflectance is a useful technique to assess tectonic stress and structural changes in vitrinite, cokes, graphites, and chars. The true maximum and minimum reflectance values are measured in polarised light and by rotating the stage or the analyser or polarizer (Crelling et al, 2005), ideally on orientated samples. The anisotropy is calculated from the difference (Rmax – Rmin) (Suárez-Ruiz and Ward, 2008, and references therein). Bireflectance with a mean reflectance of >1.64% is normally pronounced (Crelling et al., 2005).

All electrical components should be switched on at least half an hour before the instrument is calibrated to allow for stabilisation of the light source or pathway. Certified mono-crystalline prisms used as calibration standards include yttrium-aluminium-garnet (YAG), typically certified at 0.9%; strontium-titanat, typically certified at 5.37%. Silicon carbide (typically certified at 7.50%), gadolinium-gallium-garnet (3G) (typically certified at 1.73%), and spinel (typically certified at 0.42%) are other materials used as vitrinite reflectance standards. Following calibration, it is recommended that an in-house standard is checked to confirm accurate calibration prior to analysis. A minimum of 100 readings are taken on vitrinite bands or particles which fall within the graticule area of measurement (Fig. 2.12), avoiding poorly polished or pitted vitrinite; high quality sample preparation is vital for reliable reflectance measurements. Typically, areas of measurement are encountered along traverses 0.5 mm apart and with steps of 0.5 mm along the horizontal cross lines, but this can vary depending on the block mount size as well as the particle size and distribution (refer to ISO 7404 part 3, 1994). The reflectance of any vitrinite grain should not be measured more than once. Checking of the instrument calibration should take place before and after the measurement of each sample.

Figure 2.13: Various reflectance measurements on different vitrinite macerals, ranging from 0.56% to 0.66%. Telinite reports lower and collodetrinite reports higher values (middle band) than collotelinite. Variable values are obtained for corpogelinite.Reflectance readings should be taken on collotelinite for consistency, where available. (Image taken with Diskus Fossil on a Ziess Axioimager; oil immersion, reflected light, polariser out, monochromatic digital camera, x500).

The recorded readings are reported to two or, less commonly, three decimal places, as the mean random reflectance, or mean maximum reflectance, and statistically represent the distribution of readings in the polished block. The standard deviation should always be included to indicate the distribution of the readings; the value should fall well under 0.10%, but this can vary depending on coal rank (anthracite may report higher standard deviation values, for example). Most software running the reflectance analysis calculates the final mean values, but ISO 7404 part 5 lists the applicable calculations. Repeatability values correspond to 0.06% (same operator, same sample); reproducibility values (different subsamples, different operator, and same number of measurements) is 0.08% (SABS ISO 7404 part 5).

Reflectance histograms showing the distribution of readings are particularly useful when assessing coals, and can rapidly provide information about contamination, blending, heat effect, and so on (Suárez-Ruiz and Ward, 2008, Fig 2.7). In fact, coal petrography is the most effective technique to identify coal blends, and where the geochemical analyses and performance data may show anomalies, as it is not a bulk analysis. Figure 2.14 provides two examples of histograms constructed from reflectance analysis data. The ICCP offer accreditation in the determination of coal blends (CBAP – coal blend accreditation program, [www.iccop.org](http://www.iccop.org/)).

Figure 2.14: Reflectance histograms. A) Single bell curve, single population, with no evidence of blending, heat affect, or contamination. B) Bi-modal distribution, in this instance indicative of heat affected particles due to a dolerite intrusion immediately below this sample.

Following the ECE-UN Coal Classification system (Table 2.9), terms low rank, medium rank, and high rank apply, corresponding to terms lignite, bituminous coal, and anthracite respectively (brown coal is a category of lignite). The limit between low and medium rank is 0.5 %RoVr (random vitrinite reflectance); vitrinite below 0.5 %RoVr is termed subbituminous, or low-rank, coal. Above 2 %RoVr coal is termed anthracite, or high-rank coal. Refer to Plate 11 for photographic images showing the change of grey associated with increasing rank as observed petrographically.

Table 2.9: International Classification of in-seam coals, based on rank (adapted from ECE-UN, 1998). Both designations (low-, medium-, high-rank, and lignite, subbituminous, bituminous, anthracite) apply to both humic and sapropelic coals. (%RoVr = percent random vitrinite reflectance).

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| % Bed moisture | | | Percentage mean random vitrinite reflectance %RoVr | | | | | | | |
| 75 | 35 | | 0.4 - | 0.5 - | 0.6 - | 1.0 - | 1.4 - | 2.0 - | 3.0 - | 4.0 - 6.0 |
| LOW-RANK | | | | MEDIUM-RANK | | | | HIGH-RANK | | |
| C | | B | A | D | C | B | A | C | B | A |
| Lignite | | | Subbituminous | BITUMINOUS | | | | ANTHRACITE | | |
|  | | |  | Para | Ortho | Meta | Per | Para | Ortho | Meta |
| Vitrinite is dark grey | | | Vitrinite is dark grey | Vitrinite is a slightly lighter grey | Vitrinite medium grey |  | Vitrinite appears light grey | Vitrinite appears white |  | Vitrinite is whiter than inertinite |
| steam | | | steam | steam | PCI/soft coking | Prime coking | Blend LV/PCI | Possible PCI | Graphitizable carbon |  |

Vitrinite reflectance V-classes are occasionally reported in the metallurgical assessment of coals. The V-classes correspond to 0.5 % reflectance value categories, commencing at V1. The number of counts occurring in each V-class are recorded. The V-class quantification system is comparable to the bars in reflectance histograms.

With the need for the development of advanced techniques in the exploration of the petroleum and shale gas industries internationally, vitrinite reflectance has been applied to assess the thermal maturity of sedimentary rocks on dispersed organic matter (Hackley et al., 2015; Hackley and Cardott, 2016). The data is then used to indicate likely areas of good hydrocarbon yield relative to other areas, or depths of limited or no yield. ASTM standard D7708-14 (ASTM, 2014) applies in this instance. Whole rock, grains, or concentrated organic matter are prepared in block mounts as for routine petrography. The light reflected from the particle surface (typically vitrinite) is recorded as % reflectance after calibration of the detection equipment (as described above). The major categories of thermal maturity in respect to the generation of liquid hydrocarbons are:

1. Immature - random reflectance less than or equal to 0.5%;
2. Mature - random reflectance between 0.5 – 1.35%;
3. Over mature - random reflectance greater than 1.35%.

Typically 20 – 30 measurements should be obtained on sediment samples containing organic matter; it can be difficult to locate and accurately identify the organic matter, and non-compliant analyses can be reported (<20 measurements). As with routine vitrinite reflectance analysis, the mean and standard deviation values are calculated. Samples may contain two vitrinite populations, generally with the lower one representing the true maturation level; the higher values may be representative of reworked vitrinite (Bustin et al., 1983). However, oxidation can also lower the reflectance readings where the samples have been obtained from an outcrop.

Vitrinite reflectance suppression can also occur where a reduction in expected reflectance values arises as a result of atypical hydrogen-rich vitrinite chemistry inherited from the precursor plant material or where hydrogen has been introduced into the vitrinite by the chemical microenvironments of deposition, diagenesis, and catagenesis (ASTM D7708-14, 2014; Suárez-Ruiz and Ward, 2008). As stated by Suárez-Ruiz and Ward (2008), under such conditions, it can be difficult to find an indicator of rank that is totally independent of the organic and inorganic composition of the sample source, or of the other influencing factors such as the depositional environment of the original peat deposit. This suppression can only be determined if there are non-supressed particles in the sample, or previous data is available for comparison. It must be recommended that the particles showing vitrinite suppression are eliminated from the mean value, but their occurrence must be noted.

The ICCP offers a training course in dispersed organic matter, as well as a training module with blocks available for purchase, as well as ICCP accreditation on the determination of vitrinite reflectance on dispersed organic matter. Rego Menezes et al. (2015) considered the standardization of reflectance measurements on dispersed organic matter between laboratories.

* + - 1. Non standardised petrographic techniques

In efforts to enhance the available information obtainable by coal petrography, petrographers globally have devised various analytical schemes. These include, but are not limited to:

1. Abnormal condition / oxidation analysis
2. Mineral group analysis
3. Coal Char petrography
4. Coke petrography
5. Fly ash and unburnt carbon petrography
6. Thermal maturity determination on dispersed organic matter petrography using vitrinite reflectance
7. Determination of facies and depositional environments

The above analyses are discussed further in Chapter 3. They all follow the maceral or microlithotype point count concept, assessing the component either directly under the cross-hair, or a 50x50 micron particle or area of a particle. Dispersed organic matter assessments make use of vitrinite reflectance, as discussed above.