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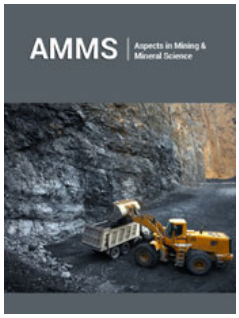
Review Article

Towards Soft Matter Physics of Coal Mesophases: Recognizing the Role of Mesophase/Nematic Liquid Crystals in the Conversion of Coal to Coke and Coal Co-Carbonization by H Marsh

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Abstract

A mesophase is an intermediate liquid-crystalline (often nematic) phase that forms in the plastic layer of coal during heating. It plays a crucial role in the formation of the anisotropic structure of coke, determining its mechanical and thermal properties. The genius Harry Marsh's contribution to the mesophase concept of the coal formation was establishing the relationship between coal quality and mesophase development. In joint works with G. Hermon and G. Cornford, Marsh demonstrated that the formation and growth of nematic mesophases are largely determined by eutectic effects during the combined carbonization of coal components. This means that mixtures of components can form mesophases at lower temperatures and under different conditions than the individual components. This happens because individual coal maceral groups interact, lower the system's melting or softening point, and change how coke and carbon structures form. Marsh identified how mesophase domains form in localized ROIs of coal with varying thermal activity and linked this to the plastic state. Localized thermal activity creates tiny reaction zones where aromatic molecules align and merge into larger mesophase spheres. The temporary melting or softening phase of coal allows planar molecules to move, stack, and build the mesophase/liquid crystal structures. Marsh also thoroughly studied the textures that arise during carbonization and established correlations between temperature, heating duration, and the expression of anisotropic mesophase domains. Marsh demonstrated how thermally induced flows and diffusion processes influence mosaic anisotropy in coke. A genially important applied area of Marsh's work was the co-carbonization of coals as a pathway for the mesophase regulation in coal formation, processing and technological usage.

Keywords: Harry marsh; Coal; Coke; Pitch; Mesophases; Nematic liquid crystals; Carbonization; Co-carbonization; Coal formation; Coke formation; Soft matter carbon physics; Soft matter carbon chemistry; Mesophase geochemistry

Formation and Behaviour of Mesophases During Carbonization and Coking as a Reaction-Diffusion Process with Regulatory Feedback

Since the early 1970s, the concept of the determining role of mesophases in morphogenesis (dissipative structuring) of cokes and graphites has become established [1,2]. Well before the formation of graphite with its sufficiently perfect three-dimensional crystalline structure, organic matter undergoes a stage of liquid (viscous-flowing)/liquid-crystalline mesophase state during carbonization [3]. Consequently, reaction-diffusion processes in a thermotropic medium during heating [4,5] and combustion leave their imprint upon the ultimate microstructure of the carbonizate. The application of the term "morphogenesis" to liquid crystals is not novel, as similar terminology has previously been applied to the spatial organization of multicomponent mixtures of liquid-crystalline substances [6] and to phase transitions therein [7]. Thus, reaction-diffusion/morphogenetic (in the Turing sense)

processes constitute an adequate model and form of description for the mechanisms of structural organization in thermotropic liquid crystals or mesophases. Moreover, in the case of heating or combustion, these processes must be considered as thermally induced self-organization within a temperature front, possessing common features with photoinduced self-organization [8,9], likewise observed in liquid-crystalline systems under the action of a light beam [10,11].

Since the characteristics of mesophase formation depend upon the carbonized structure (in the case of mixtures of combustible substances, upon their proportion and phase characteristics [12]), the morphometric characteristics of mesophases, as well as the granulometric composition of the ultimate carbonizate, correlate with the dimensions of microstructures or the dispersity of the carbonized substance, with adjustments for coalescence of liquid-crystalline structures/textures [13,14]. Whilst heterodispersity (and morphometric anisotropy of particles) is characteristic of powdered forms [15], this is not resolved so unambiguously for liquid-crystalline/mesophase forms in contact with the surrounding medium, yet may be calculated from the energetic prerequisites of interaction between the substrate phase and the mesophase [16]. Simultaneously, for the case of mesophase formation directly during the carbonization of a substance that serves as precursor to both the mesophase and the substrate phase with which it makes contact, each successive stage during combustion requires special calculation of energetic relationships, yielding in the computational output (particularly kinetic calculations) qualitatively distinct mesophase forms upon the substrate (corresponding to the classical conceptions of Marsh, formulated long before the advent of high-performance computing [17]).

Reaction-diffusion mechanisms in combustion processes, including the coalescence mechanism, should lead to the formation of phases of spherical geometry [18], which is indeed observed in electron-microscopic examination of these mesophases [19]. However, in reality, the physicochemical characteristics of the surfaces of phases with which mesophases make contact (particularly the manner in which a liquid droplet/partially ordered medium contacts a substrate- the hydrophobicity/hydrophilicity of the substrate, determining the contact angle [20,21])- as well as numerous additives and external fields-perturb the surface of "mesophase spherules" during coalescence and solidification [22,23], with the result that strict spherical geometry does not form. This indicates that the regulation of this process is not limited to free energy alone, and the process itself indeed possesses characteristics of "morphogenesis"- that is, a reaction-diffusion process on a mesophase, liquid-crystalline physical basis, involving the carbonized precursor and the product of its decomposition, which emergently influences the course of the process (regulating it, in control-theoretic terms) of mesophase sphere coalescence directly during its formation [24]. Simultaneously, since the placement of spherules upon the surface of a homogeneous phase satisfies the criteria of the spherical packing model [25,26], it is evident that the porosity contributing to the ultimate form of carbonized mesophases-both of the original substance and of mesophases at various stages of their formation/coalescence-are in principle not identical or correlating [27], since the interspherical gaps of such packings are not identical, and the conservation of phase volume after combustion does not constitute an approximation adequate to reality, owing to substance losses during gas evolution and the collapse of pore walls during heating or combustion [28,29]. This "independence effect" manifests itself particularly in the case of multicomponent combustible mixtures or substances in which impurities determinatively influence the mesophase structures produced during coking (the formation of coke-like but not fully identifiable structures, or "semi-coke" structures [30]). Therefore, in studying the processes of mesophase formation/unstable liquid-crystalline formation in combustible systems, emphasis must be placed upon both the physics (diffusion, contact and wetting, mesophase coalescence, front propagation) and the chemistry of combustion, leading to the manifold of mesomorphic structures and their derivatives observed in experimental investigations.

Let us consider the processes of mesophase formation in carbonized media in the broad sense, sensu lato (that is, considering the complex of effects/mechanisms felicitously united in the foreign literature by the formulation "development of the mesophase and coke-carbon forms" [31]). The conceptual and applied expediency of such consideration is conditioned by the activity of mesophases in coal-coke transformation processes [32], which determines the complex of functional properties of coke as a product of a reaction-diffusion process-from morphology (see above) to rheology, not excluding local thermoreological characteristics and micromechanical properties, including strength and hot strength (CSR) [33]. Any perturbations affecting these variables (as a phase space) during preparation, heating, and combustion exert decisive influence upon morphology and thus upon the reaction-diffusion dynamics in the system. As a positive example of formative regulation, one may cite the formation of needle-like structures through mesophase deformation [34]; as a negative example (though capable of driving systems to rapid structural reorganization from a metastable mesophase state), one may cite the destruction of long-range order as a result of disruption of aromatic ring interactions/stacking [35] (at the stage of preliminary treatment with chemical agents, which refers us to the theory of flame retardant action, or at the stage of burning out certain organic structures prior to the pyrolysis stage, which refers us to the chemical physics of combustion and explosion [36,37]).

The original carbonized substrate itself constitutes, in a certain sense, a link in the regulation and control of the morphology of mesophase combustion products, since if carbonization preserves the framework and plan of (chemically partially substituted) structure, then the forms of carbonized products will possess morphism (homeomorphism or isomorphism) relative to the original substrate (so-called hereditary morphism). For example, upon carbonization of helical polyacetylene, we inevitably obtain helical graphite, owing to the morphostabilizing carbonization of the product synthesized in a chiral nematic mesophase [38]. For less organized media, preservation of orientation/anisotropy is possible, as a rule not anticipated in earlier conceptions [39] (since

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even at present, redox stabilization of mesophase does not, strictly speaking, ensure its morphological stability during carbonization [40]).

Carbonization and the Development of Liquid-Crystalline Mesophases

Carbonization and the processes of liquid-crystalline mesophase development during it have been the subject of pronounced systematic interest since the 1970s, and strictly speaking, since 1972/1973, when the first article in a series was submitted and published in the journal Fuel by Harry Marsh (after the first part, published in 1973, subsequently published with co-authors) entitled "Carbonization and liquid-crystal (mesophase) development" [41]. In arguing the relevance of investigating liquid-crystalline effects in coal and coal blend carbonization and in coke production, H. Marsh (subsequently laureate of the Henry Storck Award, which recognized his role in establishing the functions of mesophase/nematic liquid crystals in the conversion of coal to coke, as summarized: "In the area of carbonization, Marsh has been responsible for recognizing the role of mesophase (nematic crystals) in the conversion of coal to coke"), the author of the first submitted article appeals to optical/polarization microscopy data demonstrating the presence of anisotropic structures resembling growing liquid crystals, limited by chemical heterogeneity within the plastic phase and dependent upon the presence of solid surfaces (inert particles) within the carbonizing system.

In accordance with structural conceptions, aromatic and heterocyclic compounds were initially studied [42,43], since the greatest quantity of substances existing in the liquid-crystalline state are aromatic compounds containing benzene rings with substituents (in para-positions), and derivatives of aromatic compounds containing alternating linear and cyclic (benzene ring) groupings, as well as substituents (typically likewise in para-position), according to the established definition, likewise form the basis of liquid-crystalline thermotropic media (strictly speaking, requiring differentiation from liquid-crystalline thermoplastics/thermoreactive plastics, investigated and developed subsequently [44-46]).

The processes of mesophase formation in coal tars and in series of samples belonging to various stages of the physicochemical evolution of coal were investigated [47]. It is well known that the calorific value of coal increases along the dynamic scale from lignite to bituminous coal with increasing fixed carbon content and decreasing moisture and volatile matter content. In connection with this, since the international system of coal classification of high, medium, and low rank has established a coding system employing the mean reflectance index of vitrinite or the vitrinite reflectance reflectogram, it is evident that one of the subsequent subjects of analysis in the series of works should have been vitrinites, which was indeed realized [48]. Such an approach may readily be correlated with results of investigating liquid-crystalline substances by polarization-microscopic methods, since, in determining coal rank by vitrinite, one need only employ a polarization petrographic microscope (equipped with a microspectrophotometric or microphotometric attachment on its trinocular head). Unlike other indices-such as total (organic) carbon, volatile matter composition, or calorific value-it does not depend upon the overall composition of coal (for example, the relative proportions of various substances); the sole requirement for determination is the presence of vitrinite in the coal.

However, this very simplicity constitutes a problem and a source of artefacts. Firstly, it is evident that for multicomponent coals, mesophase formation will differ from point to point. Secondly, vitrinites themselves, representing coal macerals with "gelified" remains of plant tissues (sometimes sensu lato insufficiently demonstrably assigned to liquid-crystalline formations, since the plant cell walls originally present in the strata/maceral possessed elements of liquid-crystalline structure [49,50]) without morphometric characteristics/descriptors of fusinization, predetermine the presence/absence of mesophases in particular coal regions, topically correlated with their maceral or tissue composition. Thus, the dependence of mesophase form upon coal rank in local regions, revealed in the initial works of Harry Marsh, may be collocalized, firstly, with regions of differing thermal activity, and, secondly, with vitrinization (more precisely, vitrinitization, as the term vitrinization has undergone semantic change through incorrect usage in the humanities and socioeconomic domains). Therefore, it is evident that mesophase as a product of particular carbonization stages does not exist in isolation from the remainder of the sample's phase assemblage-rather, one may speak of eutectic mixtures or differential phase formation. Indeed, in one of the works from the same cycle, investigating the development of anisotropy in coal as a result of nematic liquid crystal growth from the plastic phase during carbonization on samples modelling real coalification processes, Marsh, Hermon, and Cornford documented the determining role of eutectics in the formation and development of nematic mesophases in these systems [51]. If one speaks of the superadditive contribution to the thermophysical behaviour of macerals, it is quite evident that mixed nematic liquid crystals in coals form eutectic zones stable at lower temperatures than individual components, and in the co-carbonization of two (or more) coal rank gradations, this leads to qualitatively distinct reaction-diffusion and interfacial phenomena, morphologically distinguishing this case from cases of carbonization of any of these coals individually.

This demonstrated yet intuitively comprehensible conclusion altered the direction of Marsh's research group. Initially, subsequent works on optical textures of carbonization of model compounds (acenaphthylene, decacyclene, and others) were divided: a separate article on the carbonization of acenaphthylene and decacyclene appeared [52] and a separate article on co-carbonization of these compounds [53]. Subsequently, several further articles bore a distinctly multicomponent "co-carbonization" emphasis, investigating not only the co-carbonization of coal constituents as such, but also the co-carbonization of coal and liquid fuels/coal tars [54-56]. Mesophase formation during coal and certain available organic additives co-carbonization was investigated not only in terms of processing results but also dynamically [57,58].

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Phase kinetics in liquid-crystalline systems constitutes an extraordinarily productive domain of physicochemical investigation, encompassing: kinetic analysis of polymerization and depolymerization processes in multicomponent mixtures (particularly composite mesophase structures) of liquid crystals and phase segregation processes; the effects of solvents upon the morphology and physical properties of liquid-crystalline formations [59,60]; phase transitions proper in liquid crystals [61,62], both equilibrium and non-equilibrium; mesoscopic physicochemical gradient effects [63,64], including those inevitably leading to diffusion and complex rheological flows in the system [65]; and so forth. Thus, one may consider this ensemble of approaches applicable to investigating the formation and reactive rheological behaviour of multicomponent mesophase systems arising in carbonized substances, which are examined in Marsh's series of works with co-authors, as well as in the present article. The prerequisites for this may be supplemented by kinetic constraints upon the formation of ultrafine cellulose fractions [66], particularly during diffusion-limited decomposition of plant remains, which, in the absence of reaction-diffusion (kinetic) limitation, would not permit even discussion of the formation of highly organized coals-owing to earlier high-dispersity breakdown of wood and other remains, yielding qualitatively different mesophases (mesophases with nanocellulose composite filling will be discussed in greater detail in the section on liquid crystals of cellulose and lignin).

Retardation of Fundamental Work in Liquid-Crystalline/Mesophase Coal Chemistry from the 1980s Onwards

The cycle of investigations by Harry Marsh's collective, devoted to mesophase development during carbonization, the publication of which continued into the 1980s, proceeded along two directions: (a) investigation of general regularities of mesophase formation; (b) applied screening of coals and other fuels. According to the opinion of specialists in British coal science, the cessation of publication of the cycle in the early 1980s was connected not with exhaustion of the subject matter or methodological base, but with the fact that during the period of "Thatcherism," which preceded the closure of many British mines and the dismissal of tens of thousands of specialists, this subject became economically disadvantageous (private communication from Dr. H. Frankfeld, 2018), with the result that even sufficiently profitable applied results could not be realized and save the research programme, individual fragments of which were occasionally published until the mid-1990s (however, already with emphasis not upon coal fuel). Articles within this cycle retained for a considerable time the aforementioned phase character, accounting for co-carbonization phenomena [67-70]; However, in these works the emphasis already shifted to co-carbonization with fuel tars. As examples of general results concluding this publication cycle, one may enumerate: the concept of variations in solvation and solvolysis schemes of additives during co-carbonization; the possibility of employing microscopic optical texture analysis methods of cokes in comparing the characteristics of solvent effectiveness; demonstration of the effectiveness of donor-hydrogen carriers interacting with products of thermal depolymerization of coal; the general mesophase stage in mechanisms of coal liquefaction and coal blend mixtures for coke production; establishment of differences in mesophase dynamics of soluble and insoluble fractions; establishment of the fundamental dependence between the molecular weight of fuel fractions and mesophase texture dimensions (for example, low-molecular fractions of coal extract produce coke with optical textures of larger dimensions than coke from the original extract, whilst higher molecular weight fractions produce coke with textures of smaller dimensions); establishment of the influence of rheological parameters/fluidity of the medium upon mesophase behaviour and chemical stability; proof of the idea that "dominant partner effects" influence optical texture size (that is, establishment of feedback wherein a secondary component-a low-molecular organic or inorganic agent or additive-controls the development of liquid crystal morphology); identification and analysis of mechanisms of mosaic anisotropy development during co-carbonization in coke; establishment of positive correlation between coal processing temperature and the expressiveness of anisotropic mesophase domains in coke (with pronounced structure of thermoinduced flows) [71-74]. It is now almost impossible to imagine how, despite such a solid background with evident applied value, these works were discontinued.

In 1981, Marsh and Grint published their final article-an attempt to draw metallurgists' attention to the possibility of replacing coking coals with coal pitch: interaction between coal and tars at the interface of coal particles with coke formation displaying pronounced disperse optical texture-the result of co-carbonization interaction between coal and tar, leading to increased coke strength [75]. However, despite the application of methods and instrumental approaches employed in mesophase analysis, this work bore minimal relation to mesophases and contributed least to understanding pseudo-liquid-crystalline effects observed in coals and cokes during rational processing.

The cessation of this direction of work by its masters, with the absence of logical justification for the reasons for halting the publication cycle and an integrating review of the physics of the mesomorphic state of these substances, left successors and workers in adjacent fields in a state of uncertainty regarding the vector of research activity. Since the "masters" in their final works shifted towards mesophases in tars/pitches, many specialists reactively switched to analysing tars, bitumens, various liquid distillation products, and liquid fuels. Consequently, subsequently a whole series of authors incorrectly concluded that the formulation of mesophase phenomena in solid fuel chemistry constituted merely an extended sensu lato interpretation of fluidity and rheological properties of these fractions and processing products, which testifies to weak familiarity of those asserting this with the physics underlying the liquid-crystalline state and its distinctions from other branches of physics of partially ordered media (soft matter physics).

The mid-1980s produced a very small quantity of works in the field of mesophase tars and their derivatives, fragmentarily investigated under laboratory conditions limited in experimental

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scope, which provided, by several criteria, substantial advantages compared with large-scale reactor experiments (bulk approach), including the possibility of work in thermal vacuum and experimental gas environments [76]. Beginning in the 1990s, after a decade's hiatus, articles appeared on mesophases involving co-carbonization products, particularly coal tars and combustible composites based thereon [77-83]. Scepticism regarding viscous manifestations of mesophase activity (probably for intuitive reasons, substantially less than formerly towards its solid-phase realization in Marsh's works with co-authors) ceased to exert determining influence upon progress in this research area. Probably, the established historical priority in this sphere likewise played a role, since in the late 1960s works were conducted proving, by X-ray diffraction analysis methods, the existence of mesophase at early carbonization stages of tars [84].

The period from the early twenty-first century to the present has been characterized by an almost exclusive emphasis upon liquid-crystalline/mesophase manifestations in tars/pitches and viscous fuels [85-91]. Works investigating liquid-crystalline behaviour in coals are virtually not realized, particularly in the maximum programme-with linkage to horizons/geochronological reference points/evolutionary stages/biogeochemical and biomineralization descriptors of corresponding periods, and still less to bargeochemical/thermobargeochemical maps of sections for correlating natural coalification conditions and fusinization with any of the laboratory models of "mesophase" (or at least indirectly involving metastable mesophase formation) carbonization. The sole matter inspiring hope for possible correction of the situation is the equivalent applicability of methods developed for coals by Marsh with co-authors to other energy sector substances, which might be analysed, taking into account their physicochemical approaches-particularly, conceptions of eutectic functions and heterogeneous/multiphase flows in the morphogenesis of carbonization and co-carbonization products [92]. However, this approach is not so productive for (already) viscous-flowing systems, except for mesophase composites composed of such tars and coal fragments or other solid coal processing products [93] (and this represents not a novel approach, but rather a novel perspective on Marsh's approach, distinguished by emphasis upon the binding matrix: tar, pitch). Therefore: to return to Marsh's original idea, one must return not to the idea of eutectic as such, but to the physical principles of microheterogeneous organization of media-precursors of liquid-crystalline carbonizates. In other words, to trace hereditary chains of mesophase property changes from phytochemistry and natural compound chemistry to caustobioliths and other definitive forms of geological genesis.

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